ANALYSIS OF TASTE-AND-ODOR COMPOUNDS IN DRINKING WATER USING GAS CHROMATOGRAPHY AND FLAME IONIZATION DETECTION (GC-FID)

By

XUEWEN WANG

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Dalian Polytechnic University

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ANALYSIS OF TASTE-AND ODOR COMPOUNDS IN DRINKING WATER USING GAS CHROMATOGRAPHY AND FLAME IONIZATION DETECTION (GC-FID)

Thesis Approved:

Dr. Gregory G. Wilber

Thesis Adviser

Dr. John N. Veenstra

Dr. Dee Ann Sanders

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Title of Study: ANALYSIS OF TASTE-AND-ODOR COMPOUNDS IN DRINKING WATER USING GAS CHROMATOGRAPHY AND FLAME IONIZATION DETECTION (GC-FID)

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Abstract: Geosmin (GSM) and 2-methylisoborneol (2-MIB) are known to cause taste and odor problems in water. This study investigated the usefulness of a SPME-GC-FID method for analyzing aqueous geosmin and 2-MIB and better understanding the capacity of a copper-based algaecide for controlling these compounds in a drinking water treatment process.

Analysis of geosmin and 2-MIB are possible with a standard SPME-GC-FID method, although the method sensitivity is at or slightly above the odor threshold concentrations. This renders the method less useful for situations with very low concentration, but it was still confirmed to be a rapid and reliable method. The detection limit of GC-FID can reach the threshold (25 ng/l) of these compounds.

The copper-based algaecide did show a potential for removing 2-MIB, but the variables that control its reaction rate remain unknown. For the recommended dosage of 1.188×10^{-9} mg/l, no significant difference was found, even though the 2-MIB showed a slow decrease to about 18%. In another experiment which the algaecide dosage was doubled, both GSM and 2-MIB exhibited a slight decrease. These results indicate that an acidic, copper algaecide does result in reductions in 2-MIB, but not at the rate observed in the full-scale plant. It is concluded that one or more additional factors plays a role in the reaction, a factor not present in the bench-scale experiments.

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CHAPTER I

INTRODUCTION

Geosmin (GSM) and 2-methylisoborneol (2-MIB) are known to cause taste and odor problems in water. Geosmin and 2-MIB are produced by members of certain groups of benthic and pelagic aquatic microorganisms including algae, found in source waters such as lakes, reservoirs, and running waters. Currently, there are not many efficient and commercial methods to remove GSM and 2-MIB due to their stability with respect to chemical (Peter, 2007) and biological degradation. In addition, the odor threshold concentrations (OTCs) for GSM/2-MIB can range from 4-20 ng/l (Lloyd, 1998).

Numerous studies have focused on the removal of GSM and 2-MIB, and some treatment methods have been applied by drinking water treatment plants. These methods include activated carbon, ozone, biofiltration and application of algaecides. Adsorption by either granular activated carbon (GAC) or powdered activated carbon (PAC) is considered one of the most efficient ways to remove organic contaminants in water. However, the presence of natural organic matter (NOM) in water significantly reduces activated carbon adsorption capacity for GSM and 2-MIB. Ozone and UV are efficient but expensive ways for removal, but additional chemicals added into the water result in formation of disinfection by-products and potential health problems. Microbial treatment methods

applied by the water industry (activated filter beds) are few in number (Persson, 2007). Some indeed decrease the concentration of GSM and 2-MIB to very low levels, but usually microbial processes take much longer, up to several weeks or even months. Dissolved GSM is slowly degraded by microorganisms in aerobic freshwater, but little is known about the fate of this compound under anoxic conditions. Similarly, few research has been carried out on natural 2-MIB degradation. Most taste-and-odor outbreaks are unanticipated, and thus there is a heavy reliance on water treatment plants to control their impairment of drinking water. As such, a chemical process that can be applied on an as-needed basis is desirable. Schweitzer and Ekstrom (2006) found that EarthTec®, a copper-based algaecide, application resulted in effective removal of these compounds, especially with geosmin. In Tulsa Oklahoma, Mohawk Park Water Treatment Plant, EarthTec® has shown excellent abilities to remove geosmin and 2-MIB in lake water. Therefore, the assumption that EarthTec® and other similar copper-based algaecides have abilities to remove taste and odor compounds can be made based on these results.

A rapid method employing solid phase micro extraction (SPME) with gas chromatography (GC) and flame ionization detection (FID) has been developed for the analysis of GSM and MIB in water in some studies. Routine quantification at µg/l concentrations can be accomplished using SPME-GC-FID.

Then, the specific objectives of the research described here include:

- To determine the usefulness of the GC/FID method for GSM and 2-MIB analysis
- To better understand the use of a copper-based algaecide for control of geosmin and 2-MIB in drinking water treatment.

CHAPTER II

LITERATURE REVIEW

2.1 Source of GSM/2-MIB

Geosmin and 2-methylisoborneol (2-MIB) are low molecular weight volatile alcohols produced by actinomycetes (gram positive bacteria) and cyanobacteria (blue-green algae). 2-MIB is produced during those organisms' life cycle and geosmin is trapped in the cell bodies and released after the end of life cycle. High concentration of geosmin and 2-MIB in water sources is not always an evidence of algae presence. Factors that trigger proliferation of actinomycetes and cyanobacteria are nutrient input in water sources, warm temperature, and stagnation and reduced water quality associated with drought conditions (Ho et al., 2012).

As the major source of the taste-and-odor compounds, cyanobacteria have been present on earth for around 2.5 billion years and keep evolving and adapting to our environmental conditions, especially in water sources, such as ocean, lakes, and reservoirs (Paerl et al., 2001). Actinomycetes have been associated with earthy-musty odors in water and fish since the early 1900s (Adams, 1929; Thaysen, 1936) but their actual contribution to odor in freshwater was unknown. In the late 1960s, the taste and odor (T&O) compounds, geosmin and 2-MIB, were identified from actinomycete cultures

(Gerber and Lechevalier, 1965; Gerber, 1979, 1983). Since then, actinomycetes have attracted considerable attention in the water industry as a major T&O source of drinking water.

Cyanobacteria synthesize geosmin and 2-MIB throughout growth, which relates to photosynthesis and pigment synthesis. These algal cells store or release these T&O compounds depending on growth phase and environmental factors that affect these processes (Naes et al., 1988; Rashash et al., 1995, 1996; Srinivasan and Sorial, 2011). Cell damages due to death, senescence, and biodegradation release geosmin and 2-MIB into water (Srinivasan and Sorial, 2011).

2.2 Methods to remove GSM/2-MIB

Taste and odors caused by metabolic by-products formed by algae and other microorganisms in water supply are often seasonal in occurrence. Thus the treatment selected requires a versatile technology that can operate under prominent composition fluctuations (Ferreira, 2013). Conventional water treatment processes such as coagulation, sedimentation and filtration are not efficient methods for removal. Effective and accepted treatment options to control taste and odor compounds include advanced oxidation processes (AOP) like ozone oxidation and adsorption using granular or powdered activated carbon (GAC/PAC) and microbial treatment.

2.2.1 Powdered Activated Carbon (PAC) Adsorption

Activated carbon is an effective method of control for these two compounds. For seasonal occurrences, powdered activated carbon is more commonly utilized (Summers, 2013), so

most applications focus on the PAC treatment. Among those options mentioned above, PAC treatment is the simplest method and perhaps the most widely applied (e.g., Srinivasan and Sorial, 2011), but is rather expensive compared to conventional treatment processes such as coagulation treatment, in particular when it is used on a continuous basis (Yoshihiko, 2013). The PAC dosages to remove organic compounds in water treatment plants are determined by several factors: contact time with the liquid phase; raw water quality; presence of natural organic matter; and adsorbent characteristics (Newcombe et al., 1997; Newcombe and Cook, 2002).

Ferreira and coworkers (2013) investigated the impact of PAC adsorption, combined with coagulation process by using ferric salt as coagulant on the removal of organic compounds that produce tastes and odors in a water supply. The highest 2-MIB removal efficiency (about 70%) was achieved within 15 minutes' contact time without the coagulant addition and with the highest PAC dosage. Lower removal efficiencies were observed when adding PAC after the coagulant. This maybe because the coagulant coats the surface of the carbon and interferes with the 2-MIB coming in contact with the carbon's surface and pores.

Even though relatively high removal efficiency can be achieved by PAC treatment, it is uneconomical at a higher concentration of GSM and 2-MIB (Herzing et al., 1997) and the treatment is easily hindered by the present of other natural organic matter (NOM) in the source water (Cook et al. 2001). Yoshihoko et al. performed their study on assessing the removal capacity of superfine powered activated carbon (SPAC, partical size < 1μm) on GSM and 2-MIB in the presence of natural organic matter (NOM). The result showed the capacity of SPAC was 27% greater than that of PAC. The presence of NOM reduced the

MIB adsorption capacity on SPAC by 85% while it reduced the capacity on PAC by 84% indicating that the adsorption capacities of SPAC and PAC were reduced to a similar extent by competitive adsorption of NOM.

2.2.2 Advanced Oxidation Processes (AOP)

In the past few years, research on the removal of taste and odor compounds has been greatly focused on oxidative techniques and advanced oxidation processes (AOPs) (Jung et al., 2004; Park et al., 2007; Song and Shea, 2007). In advanced drinking water treatment process, ozonation is one of the most commonly applied technique to remove these taste and odor compounds. However, the oxidation reaction is known to produce disinfection by-products (DBP), which might produce a significant risk. At this point, the optimization of ozone combined with other technologies commonly UV radiation, is studied for optimizing ozone dosage. Ho et al. (2002) concluded the highest removal efficiency (98%) of GSM and 2-MIB with ozone was observed at the present of NOM with higher specific UV absorbance (SUVA) for the shortest contact time.

Liang et al. (2007) found that pH is a significant factor affecting oxidation of 2-MIB by ozonation, increasing the pH increased the removal rate of taste-and-odor compounds (Yuan et al., 2013), while the presence of natural organic matters did not have a great effect on ozonation of GSM and 2-MIB. It is known that decomposition rate of ozone in water, resulting in more high reactivity of ·OH groups increases which increase the removal rate of odor compounds with the increase of pH. Nerenberg et al. (2000) found that removal efficiencies of these compounds increased with increase in temperature, ozone dosage, pH, and H_2O_2 concentration. Westerhoff et al. (2006) found that geosmin showed better removal than 2-MIB because of better second order reaction kinetics.

AOPs can effectively eliminate geosmin and 2-MIB in water; however, high cost and fouling problems are considered to be a huge problem for water treatment plants.

2.2.3 Microbial Treatment

Geosmin and 2-MIB can be biodegraded by gram-positive bacteria because their structure is similar to biodegradable alicyclic alcohols and ketones (Rittmann et al. 1995). Some recent studies showed that biological sand filtration was shown to be an effective process for the complete removal of geosmin and 2-MIB, with removal shown to be predominantly through biodegradation. In addition, geosmin and 2-MIB were also effectively degraded in batch bioreactor experiments using biofilm sourced from one of the sand filters as the microbial inoculum. The biodegradation of 2-MIB and geosmin was determined to be a pseudo-first-order reaction with rate constants ranging between 0.10 and 0.58 d^{-1} in the bioreactor experiments. Rate constants were shown to be dependent upon the initial concentration of the microbial inoculum but not the initial concentration of geosmin and 2-MIB when target concentrations of 200 and 50 ng/l were used. Furthermore, rate constants were shown to increase upon re-exposure of the biofilm to both taste and odor compounds. The control culture with no added carbon source did not reveal any significant increase in bacterial abundance during this time. From this it may be concluded that these bacteria may be responsible for the biodegradation of the geosmin in the enrichment culture within the sand filter and also in the bioreactors.

2.2.4 Organic Acid Treatment

Lowering pH was proved to be an effective method to remove GSM and 2-MIB by Park's (2013) study on this program. Another study was conducted on reducing GSM and 2-MIB using different organic acids. (Pahlia et al., 2013). The standard solution of

GSM/2-MIB was diluted to 1µg/kg and a total mass of 30g. Pure (99.8%) glacial acetic (CH_3COOH) and pure (99.7%) citric acid ($[HOOC(OH)C(CH_2COOH)_2]$) were used in the acidification treatment. In the acetic acid treated samples, a significant drop ($p \leq 0.05$) of GSM was observed at 1.0% and 4.0% acid concentration, while in the citric samples, a significant drop of GSM was observed at 0.1% and 1.0%. It indicated that pH lower than 1.9 gave the greatest reduction of GSM. For 2-MIB, either acetic or citric acid at a minimum concentration of 0.1% could significantly reduce 2-MIB to its minimum detectable level, which means 2-MIB could be effectively removed at pH less or equal to 2.61.

2.2.5 Copper-based Algaecide

Copper is an essential micronutrient for growth of algae and cyanobacteria. It has been using in various metabolic and enzyme processes (Cid et al., 1995). However, higher concentrations of copper may serve as a cellular toxicant. The mechanism of copper toxicity is found by Kenefick et al. (1993) in a study where membrane damage was seen within 24 hours in cultured cells of the cyanobacterium microcystis aeruginosa following treatment with copper (0.64 mg L-1 Cu as CuSO4). At lower concentration, copper ions need to be transported into cells by a process of facilitated diffusion through the membrane (Florence and Stauber, 1986). Figure 2-1 shows the transport of copper complexes into the cell membrane by diffusion.

Copper-based algaecides, including chelated copper and copper sulfate two forms, have been used for an effective removal and growth inhabitation of algae (Wagner, 2004). Copper-based algaecides, particularly chelated forms treatments tend to inhibit rapid repopulation of algae, since chelated copper form contains less copper than copper sulfate.

Moreover, copper-based algaecides can be less effective in alkaline waters or at lower temperatures, although chelated forms perform better (Wagner, 2004; García Villada et al., 2004). The application of these algaecides can kill certain algae species if they are applied properly.

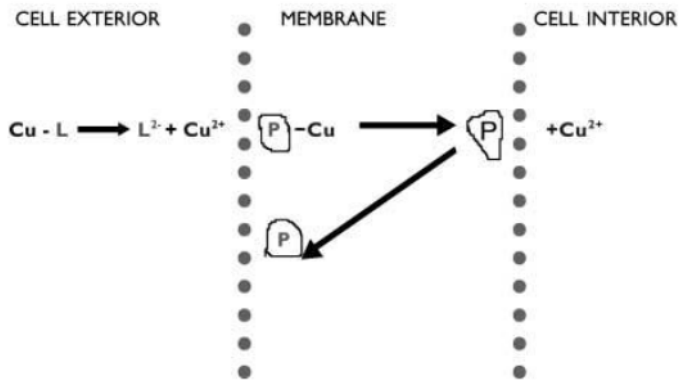


Figure 2- 1 transport of copper complexes into the cell membrane by diffusion (P is carrier protein) (Florence and Stauber, 1986)

2.3 Methods to analyze GSM/2-MIB

2.3.1 SPME-GC-MS

SPME-GC-MS is a simple and sensitive method to measure very small amounts of GSM/2-MIB in environmental water samples. Saito and coworkers (2008) investigated determination of geosmin and 2-MIB by headspace solid-phase microextraction (HS-SPME) coupled with gas chromatography–mass spectrometry. To optimize the extraction of geosmin and 2-MIB, six different commercially available fibers used in this study, the PDMS/DVB fiber gave superior extraction efficiency in comparison with PDMS and DVB/CAR/PDMS fibers. The detection limits of these compounds by HS-SPME/GC–MS under optimized conditions were 0.9 and 0.6 pg/ml, respectively. Rebecca (1998)

also proved that SPME-GC-MS detection limits of around 1 ng/l for both geosmin and 2-MIB meant that the method can be used to detect these compounds at concentrations below their odor threshold.

Some research employed different extraction method accomplishing with GC-MS. Sadao and coworkers (2001) discovered that the sensitivities of stir bar sorptive extraction (SBSE) method was 54 times higher for 2-MIB and 10 times higher for geosmin than those of the SPME method. The detection limits of the SBSE method evaluated in river water were 0.33 and 0.15 ng/l respectively for 2-MIB and geosmin.

2.3.2 SPME-GC-FID

GC/MS is the most accepted method for analysis of these compounds. It is one of the goals of the current study to determine if GC-FID can also be used. A rapid method employing solid phase micro extraction (SPME) has been developed for the analysis of GSM and 2-MIB in water. Routine quantification at µg/l concentrations can be accomplished using gas chromatography (GC) and flame ionization detection (FID).

Lior et. (2012) investigated isolating GSM and 2-MIB from a digestion basin in an aquaculture unit used SPME/GC/FID to measure the GSM and 2-MIB in the liquid samples. Concentrations of the compounds (1 ng/l detection limit) were determined in the study. Steven et al. (1998) investigated the method employing SPME-GC-FID for analysis for GSM and 2-MIB in water. The concentration levels of geosmin and 2-MIB reached 10 ng/l, the result was confirmed by using a GC-MS detention.

SPME-GC-FID is also a simple solid method for analyzing other organic compounds. Headspace (HS) HS-SPME-GC-FID revealed to be a clean, simple, fast and reliable

methodology for the determination of the methanol and acetic acid by Nunes et al. (2005). The standards in the concentration range between 40 and 100 mg/l for methanol and between 25 and 105 mg/l for acetic acid were performed. The results demonstrated that HS-SPME-GC-FID was a simple rapid useful way for methanol and acetic acid estimation. Quantification limits, determined by the concentration of each standard required to give a peak height ten times higher than the noise, was estimated to be 32 mg/l for methanol and 26 mg/l for acetic acid. Also for low concentration of benzene and substituted benzenes in water samples, SPME-GC-FID could reach a 15 ng/l detection limit for benzene (Cristina et al., 2003). In their study, different types of fiber of SPME were discussed, using PDMS/DVB/CAR fiber was considered the best analytical condition.

CHAPTER III

METHODOLOGY

Geosmin (GSM) and 2-methylisoborneol (2-MIB) are microbial metabolites with muddy/musty odors which contaminate water supplies and can be absorbed by aquatic organisms. Humans can detect them in water at ng/l concentration. One main goal of this project is to prove SPME/GC/FID is a solid and accurate method to analyze the concentration of GSM and 2-MIB at low ng/l levels. The result will be verified by parallel analysis of the same samples detected by gas chromatograph coupled to a mass spectrometer detector (Shimadzu GC/MS-QP5050A). The second primary goal is to investigate the capacity of EarthTec®, a copper-based algaecide to remove GSM and 2-MIB. The reaction lasts for four days, because the lake water travelled in the water treatment pipeline for about two days, so the experiment is designed within doubled travel time in pipeline (4 days) to see how the reaction performed.

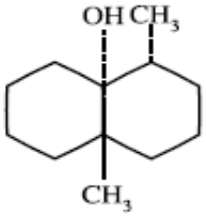
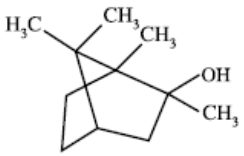
3.1 Experimental chemicals

3.1.1 Geosmin and 2-MIB

The standard of geosmin (CAS # 23333-91-7) and 2-MIB (CAS # 2371-42-8) was purchased from Supelco (Sigma-Aldrich, St. Louis, MO, USA) as a 100 μg/ml in 2 ml solution in methanol. Basic information about these compounds is shown in Table 3-1.

The solutions were stored at 4 °C in refrigerator and used after dilution with deionized water. EarthTec® was obtained from Earth Science Laboratories, Inc. (Bentonville, AR, USA) as an example of algaecides used in this study.

Table 3- 1 Chemical Information (Supelco Analytical, Bellefonte, PA)

Compound Name	Compound Structure	Molecular Formula	Molecular Weight	CAS registry number
Geosmin		$C_{12}H_{22}O$	182.3	CAS # 23333-91-7
2-methylisoborneol (2-MIB)		$C_{11}H_{20}O$	168.3	CAS # 2371-42-8

3.1.2 EarthTec®

EarthTec® is a copper solution containing 99.99% cupric ions (Cu++), which are toxic to microscopic organisms at low doses. It is used in lakes, ponds, municipal drinking reservoirs, or other water systems to control algae. It has the ability to remain in suspension over long periods with mathematically predictable copper levels which allow precise control of algae and bacteria without over-treatment (Earth Science Laboratories, Inc. 2009). Based on observation from Mohawk Park Water Treatment Plant's personnel, EarthTec® was assumed as the primary algaecide to remove geosmin and 2-MIB. EarthTec® used in this study was obtained from Earth Science Laboratories, Inc.

(Bentonville, AR, USA) as an example of algaecides. Recommended dosage of EarthTec® (1 gal/1,000,000 gal raw water) and twice of the recommended dosage (2 gal/1,000,000 gal raw water) will be applied to 100 ng/l geosmin/2-MIB solution.

The mechanism of the reaction remains unknown. These experiments were performed to add to the available data exhibiting the ability of EarthTec® to remove geosmin and 2-MIB.

3.2 Solid phase micro-extraction (SPME)

Solid phase micro-extraction is a simple and inexpensive method for the analysis of volatile and semi-volatile compounds occurring in a wide variety of food, water and environmental matrices (Eisert and Levsen, 1996). It does not require a large volume of sample (25 ml), expensive equipment, or extremely time consuming efforts (30 minutes to 1 hour). SPME relies on the partitioning of organic compounds from a matrix directly into a solid phase. A fused silica fiber is coated with a suitable absorbent phase and bound to the tip of a syringe plunger. The plunger is plugged into the needle which serves to protect the delicate fiber. The needle is used to pierce the septum of a sealed vial containing the sample and the SPME fiber is then extended (see Fig.3-1). The fiber can be directly immersed into a liquid sample or placed in the headspace above the sample for qualitative analysis. Analyte molecules are absorbed onto the coating. After equilibration, the fiber is retracted into the needle and inserted into the heated injection port of a gas chromatograph (Lloyd, 1998). In this study, manual assemblies of SPME including a 2cm-50/30μm DVB/Carboxen™/PDMS StableFlex™ SPME coated fiber

(Supelco part number 57348–U) and SPME fiber holder (Supelco part number 57330–U) (Fig. 3-1) were purchased from Supelco (Bellefonte, PA).

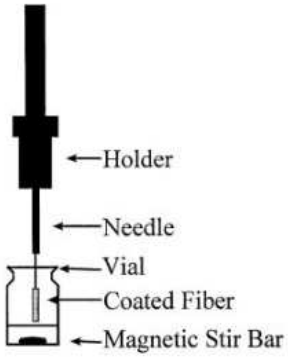


Figure 3- 1 Solid Phase Micro-extraction (Lloyd, 1998)

3.3 Gas Chromatography (GC) and Flame Ionization Detection (FID)

Gas chromatography is a common method for separating the components of a solution and measuring their relative quantities. In gas chromatography, a sample is rapidly heated and vaporized at the injection port. The sample is transported through the column by a mobile phase consisting of an inert gas. The components are then detected and represented as peaks on a chromatogram. A FID typically uses a hydrogen/air flame into which the sample is passed to oxidize organic molecules and produces electrically charged particles (ions). The ions are collected and produce an electrical signal which is then measured. A GC-FID configuration is shown in Fig. 3-2. While generally less sensitive than mass spectrometry, flame ionization is readily available in water quality labs. This study will examine the usefulness of GC-FID analysis and compare it to the sensitivity achieved by mass spectrometry observed in related studies (Zhao, 2012; Park, 2013).

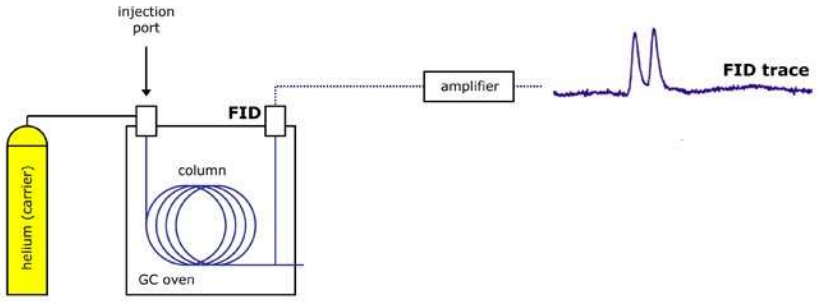


Figure 3- 2 GC-FID configuration

(http://www.uochb.cz/web/structure/1133.html)

Extraction and GC Procedure

I. Prepare needed concentration of geosmin and 2-MIB samples by diluting with deionized water.

II. Transfer 25 mL of each sample into a screw-cap sample vial with a PTFE septum. Add 3.5 g sodium chloride and a magnetic stir bar into each sample.

III. Incubate the vial in a heating block at 65°C. After 30 minutes of incubation, SPME fiber is injected through the airtight vial for head space extraction of GSM and 2-MIB for 20 minutes.

IV. Remove the fiber from the vial. GSM and 2-MIB concentrations are analyzed by injecting the fiber into the splitless operated injector of an Agilent 7890B GC with a FID. GC and FID conditions are shown in Table 3-2.

Table 3- 2 The parameters of GC and FID

Parameter	Condition
GC Model	Agilent 7890B
Column type	Equity™-5 fused silica capillary column (30m×0.25mm×0.25μm), Supelco, Bellefonte, PA
Injector	Split-less
Injector temperature	250°C
Oven temperature	Hold at 60 °C for 2 min from injection, increase to 100 °C at 20 °C /min, followed by an increase to 200 °C at 10 °C /min and to 250 °C at 20 °C /min and hold at this maximum temperature for 3 min
Carrier gas	Helium
Flow rate	1 mL/min
FID temperature	280 °C

3.4 GSM and 2-MIB chromatogram

Previous published research, using a closed loop stripping analysis (CLSA)-GC-FID methodology for the analysis of geosmin and 2-MIB (Romero, 2007), has proved that GC-FID is an easy methodology, very reliable, robust, and with low error in routine analyses of GSM and 2-MIB from drinking and natural water samples. The chromatogram of 200 ng/L GSM and 2-MIB is shown in Fig. 3-3. Comparing the detection time to the previous experiments (Zhao,2012; Park, 2013) which used GC-MS, the peaks appearing at 9 min and 12 min are known to be the peaks of 2-MIB and GSM.

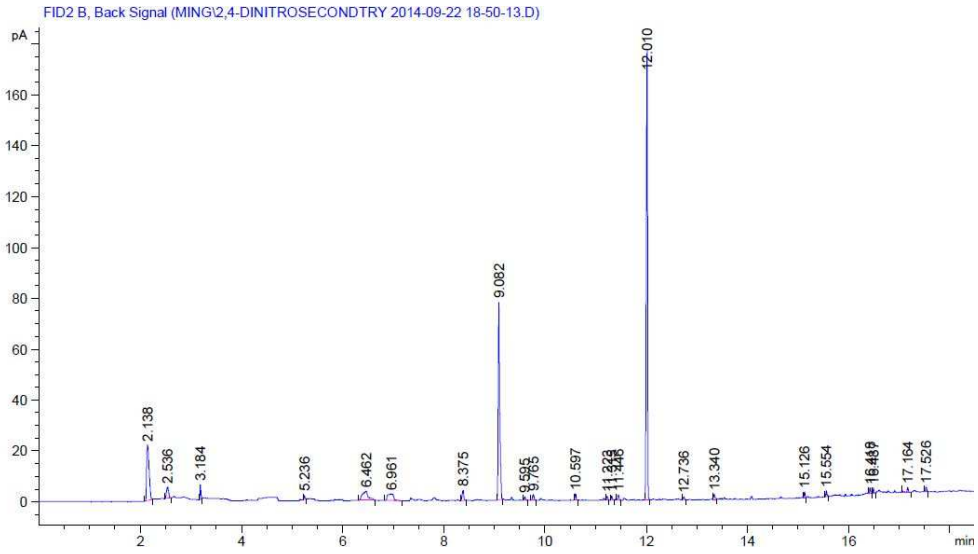


Figure 3- 3 GSM and 2-MIB chromatogram

(screenshot of a GC-FID analysis result)

CHAPTER IV

FINDING AND DISCUSSION

Samples containing geosmin and 2-MIB were analyzed by the SPME extraction technique and GC-FID analysis method. Results and discussions of each three experiments are described below. Duplicate samples under the equivalent condition were conducted at each data point. In order to minimize experimental errors, the averages of duplicate samples` results were used as final results of these experiments.

4.1 Calibration Curve

Two calibration curves of GSM and 2-MIB from 0 to 200 ng/l (25 ng/l, 50 ng/l, 100 ng/l, 200 ng/l) are shown below. Great linearity and R^2 value were obtained. The slopes were 1.0565 and 1.2408 for 2-MIB and GSM, respectively, while R^2 value were 0.998 and 0.994, respectively. From the chromatogram in Figure 3-3, we can see that there are no problems with overlapping peaks or peak resolution that can affect the chromatographic detection. Since the average population odor threshold is around 15-20 ng/l, and lowest standard tested (25 ng/l) was detected readily, it appears that we can expect that GC-FID analysis can detect GSM and 2-MIB at least down to thresholds. The retention time for an analyte as the time it takes after sample injection for the analyte peak to reach the

detector (Romero, 2007) is stable and consistent comparing to the results from previous research (Zhao, 2012). Above all, it is observed that GC-FID is a repeatable and potentially accurate method to analyze GSM and 2-MIB at their odor threshold (25 ng/l). Using concentration versus peak area instead of mass to express the calibration curve is because that SPME extracted the sample, not absorb exact amount of solution, and the sample extracted was proportional to the mass.

Table 4- 1 Peak Area of 2-MIB

Concentration (ng/l)	Set 1	Set 2	Average Area
0	0	0	0
25	20.77411	22.16271	21.46841
50	44.64479	41.50256	43.073675
100	112.7062	84.9668	98.8365
200	213.89001	203.58347	208.73674

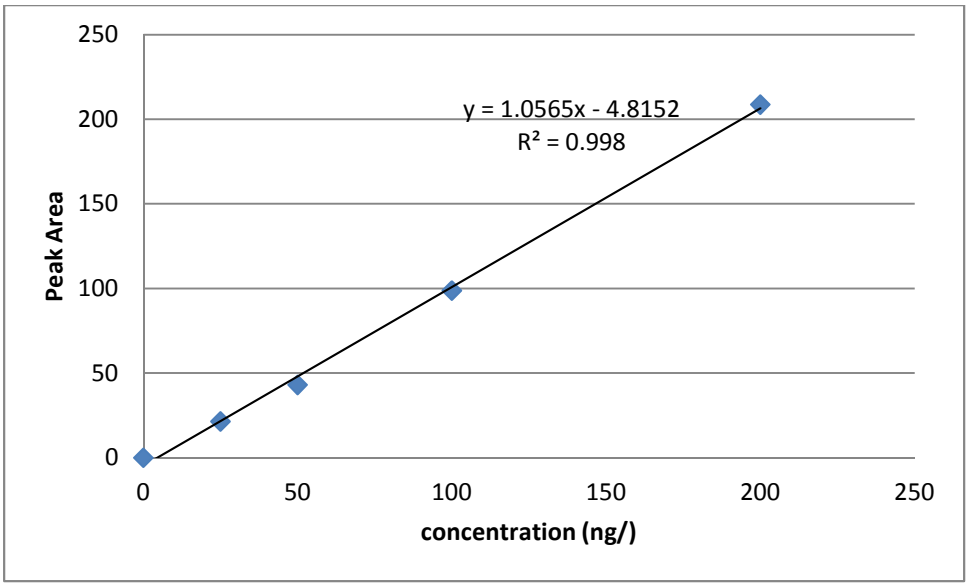


Figure 4- 1 Calibration Curve of 2-MIB

Table 4- 2 Peak Area of GSM

Concentration (ng/l)	Set 1	Set 2	Average Area
0	0	0	0
25	25.18407	21.55905	23.37156
50	59.41027	61.39054	60.400405
100	133.82628	137.89635	135.861315
200	254.1654	230.6503	242.40785

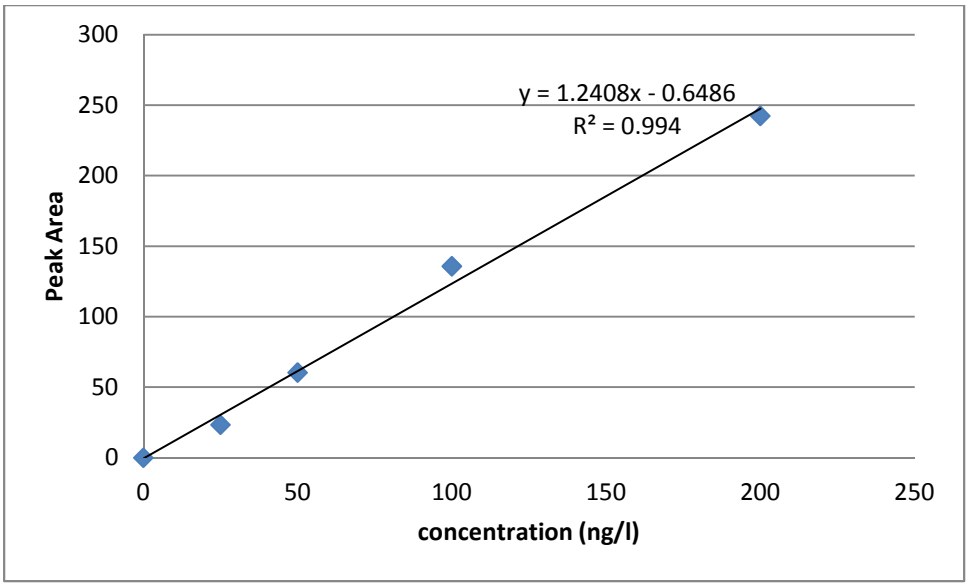


Figure 4- 2 Calibration Curve of GSM

A third set of experiments was done to confirm the consistency of the calibration curve. The peak area and concentration were listed below.

Table 4- 3 The Third Set of Peak Area of 2-MIB and geosmin

Concentration (ng/l)	Peak Area	
	2-MIB	GSM
0	0	0
25	19.85652	25.43144
50	46.96103	49.61527
100	95.70641	110.06602
200	205.07344	232.90698

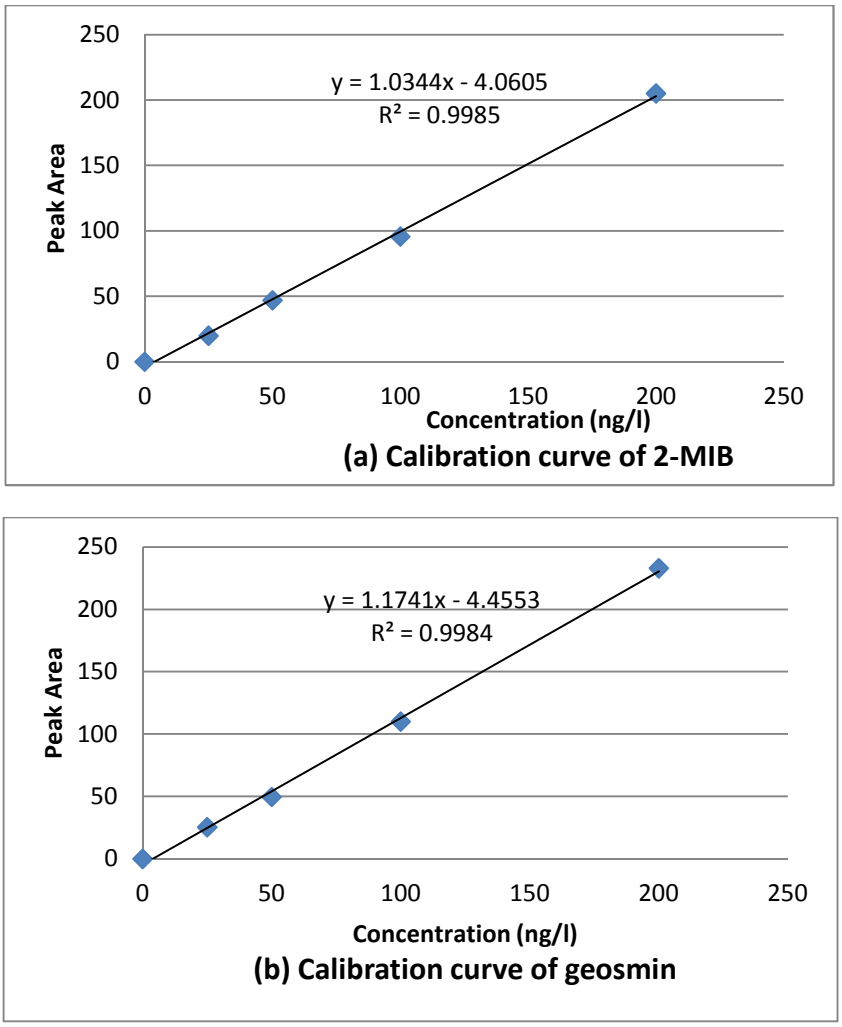


Figure 4- 3 The Third Set of Peak Area of (a) 2-MIB and (b) geosmin

Comparing the slope and y-intercept for each compound, slopes for 2-MIB were 1.0565 and 1.0344, y-intercepts were - 4.8152 and - 4.0605. Both of the parameters were very close. For geosmin, the slopes were 1.2408 and 1.1741, y-intercepts were -0.6486 and -4.4553, which also showed a steady and consistent calibration curve. At this point, SPME-GC-FID was verified that it's a consistent and reliable method to measure the concentration of 2-MIB and geosmin down to at least 25 ng/l.

4.2 Standard solution of GSM and 2-MIB with EarthTec®

In Tulsa water treatment plant, they use discovered that EarthTec® has excellent ability to remove geosmin and 2-MIB. However, no detail in mechanism is revealed to back up the EarthTec® process. So in this experiment, reaction between geosmin/ 2-MIB and EarthTec® is recreated in lab scale, different dosage of EarthTec® are applied in standard (100ng/l) geosmin/MIB solution, to see if EarthTec® is the main factor for removing the taste-odor compounds. Due to the previous lab work, we know that there is slight differences between the deionized water and lake water, so deionized water is used to dilute geosmin and 2-MIB solution in the duplicates samples below.

4.2.1 Standard geosmin/MIB with Recommended dosage of EarthTec®

Below is the experiment investigating the removal ability of recommended dosage of EarthTec® with standard concentration of geosmin and 2-MIB.

Samples: 100ng/l geosmin/2-MIB solution

EarthTec® 1gal/1,000,000 gal raw water, equivalent to 1.188×10^{-9} mg/l

Table 4- 4 Peak Area of 2-MIB with 1.188×10^{-9} mg/l EarthTec®

Day	Set 1	Set 2	Average Area	Concentration (ng/l)
0	113.0827	118.5963	115.8395	117.5692
1	86.10667	91.97424	89.04046	89.25604
2	179.3914	221.2978	200.3446	206.8489
3	115.707	89.56506	102.636	103.6198
4	197.7925	234.3652	216.0789	223.4721

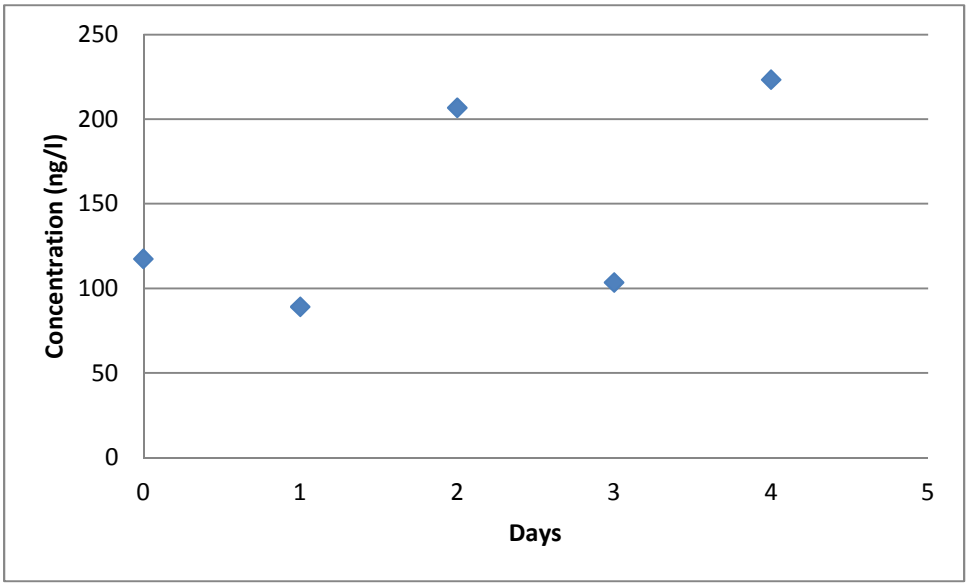


Figure 4- 4 Plot of 2-MIB with 1.188×10^{-9} mg/l EarthTec®

Table 4- 5 Peak Area of geosmin with 1.188×10^{-9} mg/l EarthTec®

Days	Set 1	Set 2	Average Area	Concentration (ng/l)
0	169.3116	197.675	183.4933	227.0299
1	128.3826	144.3649	136.3737	168.5639
2	336.3737	434.4305	385.4021	477.5583
3	224.1021	107.4461	165.7741	205.0439
4	320.0738	452.2976	386.1857	478.5306

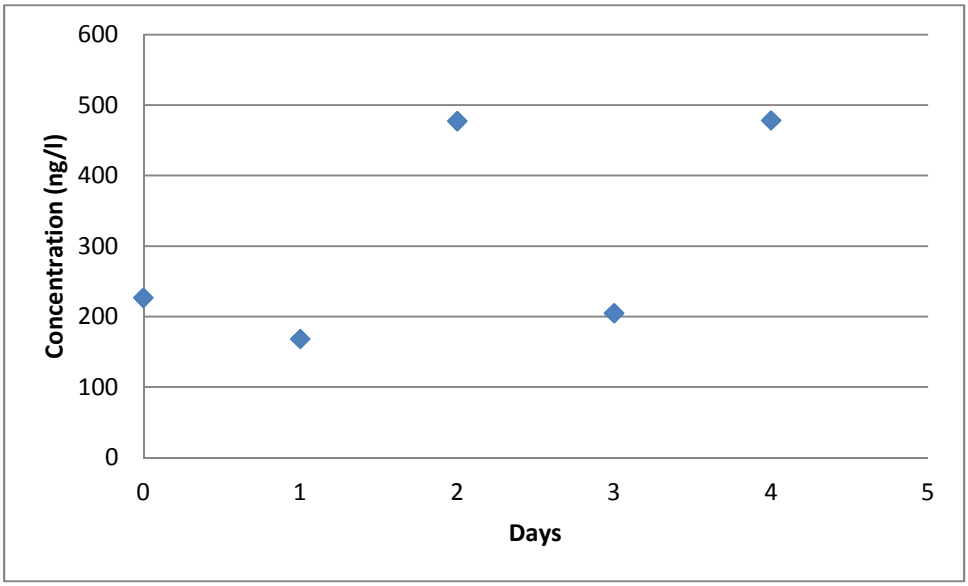


Figure 4- 5 Plot of geosmin with 1.188×10^{-9} mg/l EarthTec®

Above was the first set of experiments investigating the removal rate of EarthTec®. It's obvious to see that the concentrations on the second and fourth days were extremely high, almost twice as high as on the other days, which can't be used to analyze. This

experiment was re-run to check if this methodology can be used for analyzing 2-MIB and geosmin.

Table 4- 6 Peak Area of 2-MIB with 1.188×10^{-9} mg/l EarthTec® (redo)

Day	Set 1	Set 2	Average Area	Concentration (ng/l)
0	103.80070	102.5875	103.1941	104.2094
1	97.90993	95.70879	96.80936	97.46389
2	91.84613	94.73284	93.28948	93.74514
3	88.27743	86.10667	87.19205	87.3032
4	82.51533	86.96916	84.74225	84.71498

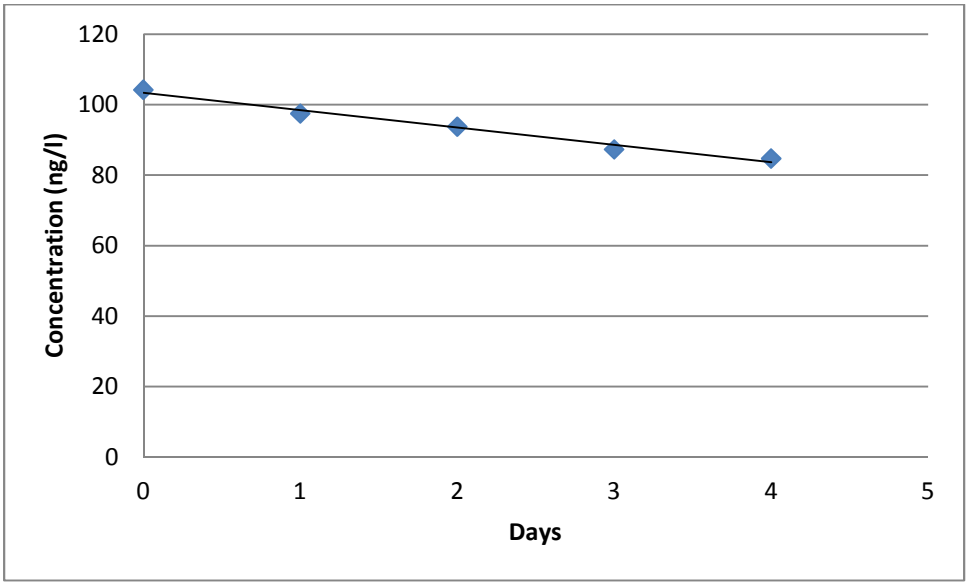


Figure 4- 6 Plot of 2-MIB with 1.188×10^{-9} mg/l EarthTec® (redo)

Table 4- 7 Peak Area of geosmin with 1.188×10-9 mg/l EarthTec® (Redo)

Day	Set 1	Set 2	Average Area	Concentration (ng/l)
0	112.3688	133.63589	123.0023	151.9727
1	111.4466	140.437	125.9418	155.62
2	126.64516	138.3113	132.4782	163.7304
3	138.664	141.58585	140.1249	173.2184
4	166.4891	166.81346	166.6513	206.1323

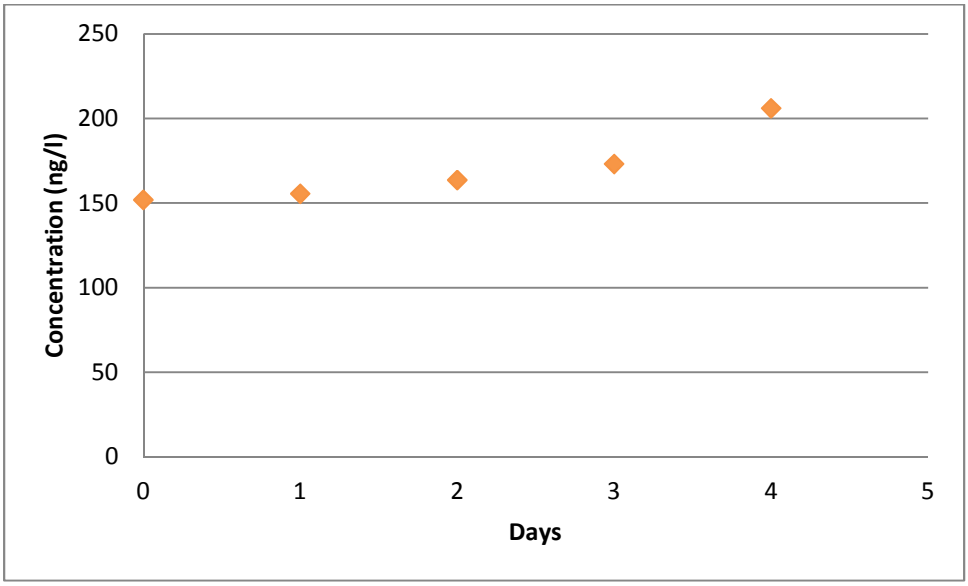


Figure 4- 7 Plot of geosmin with 1.188×10^{-9} mg/l EarthTec® (redo)

It's clear to see that MIB has a slight decrease (17.88%) with the 4-day reaction. Comparing the results with Zhao(2012) and Park's (2013), 2-MIB performed a consistent slight decrease (less than 20%) trend, but geosmin didn't. The low percentage removals

could be caused by EarthTec®, or volatilization, photolysis, sorption, and biodegradation. The degradation processes of these compounds by volatilization are too slow to be considered significant. Photolysis of geosmin and 2-MIB could not contribute significantly to removal of geosmin and 2-MIB, since the vials containing the solution are brown. Otherwise, GSM showed a slight increase during the experiment. This could be caused by instrument error, but also could occur because of SPME or during sample preparation. DVB/Carboxen/PDMS is reported as the most sensitive for small compounds and organic acids due to its increase retention capacity resultant from the mutually potentiating effect of adsorption and absorption to the stationary phase. The small pores (10 Å on average) of Carboxen make this coating fiber particularly effective for extracting small molecules (Kataoka et al., 2000). SPME is, however, sensitive to experimental conditions. Any change in experiment will affect the sorption rate, including the time required for extraction of the analytes.

4.2.2 Standard geosmin/MIB with Twice Recommended dosage of EarthTec®

Below is the experiment investigating the removal ability of twice recommended dosage of EarthTec® with standard concentration of geosmin and 2-MIB.

Samples: 100ng/l geosmin/2-MIB solution

EarthTec® 2gal/1,000,000 gal raw water, equivalent to 2.376×10^{-9} mg/l

Table 4- 8 100ng/l 2-MIB with 2.376×10^{-9} mg/l of EarthTec®

Day	Set 1	Set 2	Average Area	Concentration (ng/l)
0	104.0644	125.7488	114.9066	116.5836
1	120.1366	91.41241	105.7745	106.9356
2	29.26741	122.6274	75.94741	75.42323
3	27.8781	79.78844	53.83327	52.05965
4	80.74574	95.54005	88.1429	88.30777

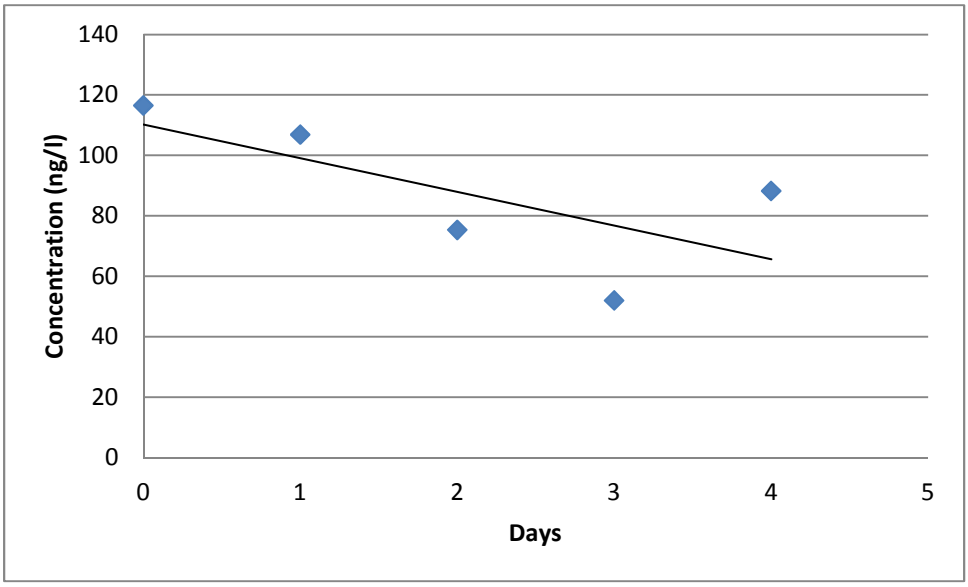


Figure 4- 8 Plot of 2-MIB with 2.376×10^{-9} mg/l EarthTec®

Table 4- 9 100ng/l GSM with 2.376×10^{-9} mg/l of EarthTec®

Day	Set 1	Set 2	Average Area	Concentration (ng/l)
0	189.4258	179.39140	184.4086	228.1656
1	197.67497	179.8839	188.7794	233.5889
2	189.5053	185.9595	187.7324	232.2898
3	144.7809	164.13663	154.4588	191.0038
4	196.8135	226.4461	211.6298	261.9417

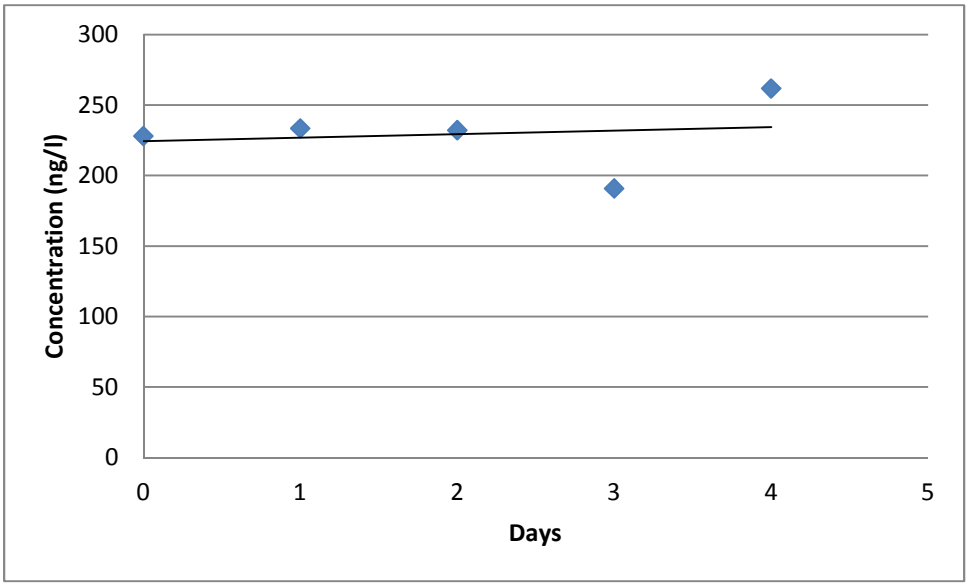


Figure 4- 9 Plot of geosmin with 2.376×10^{-9} mg/l EarthTec®

During the second experiment, a significant decrease of both compounds occurred on the third day. MIB still showed a slow decrease during the experiment, when the concentration continuously dropped down by 53.15% with two days (second and third

day), and return to a higher concentration on the fourth day. Meanwhile an obvious decrease (16.84%) also found on GSM, but not as significant as that of MIB. Since the concentration of both compounds return to a higher concentration on the fourth day (last day of experiment), this decrease was probably caused by operator error, like transferring the solution to each reaction vial. But 2-MIB did perform a decrease (23.41%) within the reaction time.

With 2.376×10^{-9} mg/l of EarthTec®, Park (2013) didn't find any significant decrease of either geosmin or 2-MIB, while Zhao (2012) observed a significant decrease (78.64%) of both compounds by 200ng/l geosmin/2-MIB with twice recommended dosage of EarthTec®. The results in this experiment was inconsistent with either of the earlier works. EarthTec® did show a potential to remove geosmin and 2-MIB, but the results indicated that there might be some other variables control the reaction rate which remains unknown.

The calibration curve indicated that SPME/GE/FID was a consistent and potentially accurate method to analyze geosmin and 2-MIB, and the detection limit was down to 25 ng/l. The reaction results from different dosage of EarthTec® with standard geosmin/ 2-MIB solution were not inconsistent with the earlier work. For the recommended dosage of EarthTec® (1.188×10^{-9} mg/l), no significant difference was found, even though the 2-MIB showed a slow decrease to about 18%. In another experiment which the EarthTec® dosage was doubled, both GSM and 2-MIB exhibited a slight decrease, in which both. From these results, EarthTec® did show a potential for removing geosmin and 2-MIB, while the variables that controlling its reaction rate remained unknown.

CHAPTER V

CONCLUSION

Taste-and-odor problems in drinking water have caused many customers' complaints for decades. Water treatment plants aim to remove these taste-and-odor compounds by numerous methods, such as granular/powdered activated carbon (GAC/PAC), advanced oxidation processes (AOP), algaecides, and biofitration etc. Based on a Tulsa water treatment plant's observation, EarthTec® was tested as the primary algaecide to remove geosmin and 2-MIB. In addition, an alternate measuring method (GC-FID) for these compounds was investigated. This study investigated the usefulness of a SPME-GC-FID method for analyzing aqueous geosmin and 2-MIB and better understanding the capacity of a copper-based algaecide for controlling these compounds in a drinking water treatment process. From the results of those experiments, the following conclusions can be made.

- Analysis of geosmin and 2-MIB are possible with a standard SPME/GC/FID method, although the method sensitivity is at or slightly above the odor threshold concentrations. This renders the method less useful for situation with very low concentrations, but it was still confirmed to be a rapid and reliable method. The detection limit of GC-FID can reach the threshold (25 ng/l) of these compounds.

During the whole experiment, there were numerous complications encountered with the GC-FID system. Adjusting the proper setpoint for the GC-FID program to make the output peaks to be sharp, clear without tail or baseline took a long time. Sometimes the results didn't come out as we expected, and attempts to determine the source of the problem were unsuccessful. As such, the experiment was repeated many more times than planned.

The capacity of EarthTec® to remove GSM/MIB was analyzed. As seen in earlier work, the rate of EarthTec®'s reaction with gesomin and 2-MIB was inconsistent. While showing potential for removing 2-MIB, the variables that control its reaction rate remain unknown. For the recommended dosage of EarthTec® (1.188×10^{-9} mg/l), no significant difference was found, even though the 2-MIB showed a slow decrease to about 18%. In another experiment which the EarthTec® dosage was doubled, both GSM and 2-MIB exhibited a slight decrease, in which both.

- Relative to controls, these results indicate that EarthTec® does result in reductions in 2-MIB level, but not at the rate observed in the full-scale plant. It is concluded that one or more additional factors plays a role in the reaction, a factor not present in the bench-scale experiments.

SPME/GC/FID is recommended for analyzing geosmin and 2-MIB at or above the concentration of 25 ng/l. Since GC/FID costs much less than a GC/MS, and due to its repeatable calibration curve, it's a useful, economic and potentially accurate method. Even though the detection limit of GC/FID is not as low as GC/MS, it is still a recommended and useful method for analyzing in most situations.

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APPENDICES

Here listed all the raw data of calibration curve and experiment results from Agilent 7890B GC with FID.

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Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
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                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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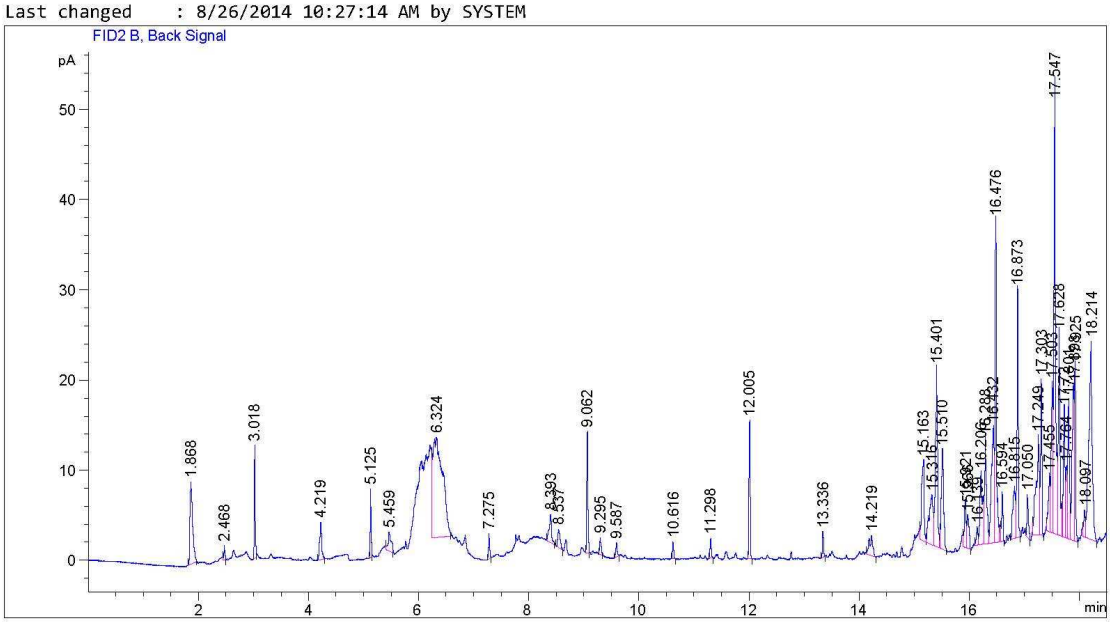


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	1.868	BB	0.0495	30.36997	9.06518	2.44739
2	2.468	BB	0.0203	2.07492	1.55844	0.16721
3	3.018	BB	0.0181	14.24314	12.46939	1.14780
4	4.219	BB	0.0350	10.44023	4.13865	0.84134
5	5.125	BB	0.0189	9.12459	7.54019	0.73531
6	5.459	BB	0.0513	8.05365	2.05582	0.64901
7	6.324	BB	0.1529	138.72794	11.10589	11.17953
8	7.275	BB	0.0213	3.89159	2.74773	0.31361
9	8.393	BB	0.0428	9.13170	3.06156	0.73589
10	8.537	BB	0.0408	5.64146	1.95747	0.45462

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	9.062	BB	0.0244	20.77411	13.49007	1.67410
12	9.295	BB	0.0270	3.45364	1.81739	0.27832
13	9.587	BB	0.0316	3.30400	1.57688	0.26626
14	10.616	BB	0.0242	3.05675	1.92089	0.24633
15	11.298	BB	0.0254	3.57627	2.19150	0.28820
16	12.005	BB	0.0263	25.18407	15.35430	2.02948
17	13.336	BB	0.0245	4.36327	2.81819	0.35162
18	14.219	BB	0.0576	9.39302	2.17382	0.75695
19	15.163	BV	0.0415	28.23026	8.97288	2.27496
20	15.316	VV	0.0641	27.66745	5.44626	2.22961
21	15.401	VB	0.0362	51.97479	19.83771	4.18844
22	15.510	BB	0.0395	31.04655	10.95636	2.50192
23	15.921	BV	0.0312	11.29298	5.14296	0.91006
24	15.966	VB	0.0325	9.48572	3.67574	0.76442
25	16.139	BV	0.0301	3.89628	1.98731	0.31399
26	16.206	VB	0.0469	28.41418	8.03537	2.28979
27	16.288	BV	0.0341	30.14314	11.67254	2.42912
28	16.432	VV	0.0442	44.21532	12.83897	3.56314
29	16.476	VB	0.0272	66.17463	35.72140	5.33275
30	16.594	BB	0.0239	8.56107	5.44782	0.68990
31	16.815	BV	0.0415	18.75837	5.71521	1.51166
32	16.873	VB	0.0246	44.80176	27.44043	3.61040
33	17.050	BB	0.0281	8.67930	4.67398	0.69943
34	17.249	BV	0.0502	42.09154	11.00452	3.39199
35	17.303	VB	0.0259	28.71427	17.18091	2.31397
36	17.455	BV	0.0344	15.18891	6.33488	1.22401
37	17.503	VV	0.0278	36.25924	16.68675	2.92199
38	17.547	VV	0.0291	103.91116	49.90784	8.37379
39	17.628	VB	0.0313	47.87252	22.40164	3.85786
40	17.720	BV	0.0306	29.47882	14.20957	2.37558
41	17.764	VV	0.0224	13.12257	7.96265	1.05750
42	17.801	VV	0.0296	32.90203	15.45764	2.65144
43	17.898	VV	0.0335	43.48727	17.20278	3.50447
44	17.925	VB	0.0251	33.70361	19.40324	2.71604
45	18.097	BV	0.0303	6.11912	2.98943	0.49312
46	18.214	VB	0.0525	89.91296	21.97320	7.24573
Totals :				1240.91015	487.32334	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/2/2014 3:10:41 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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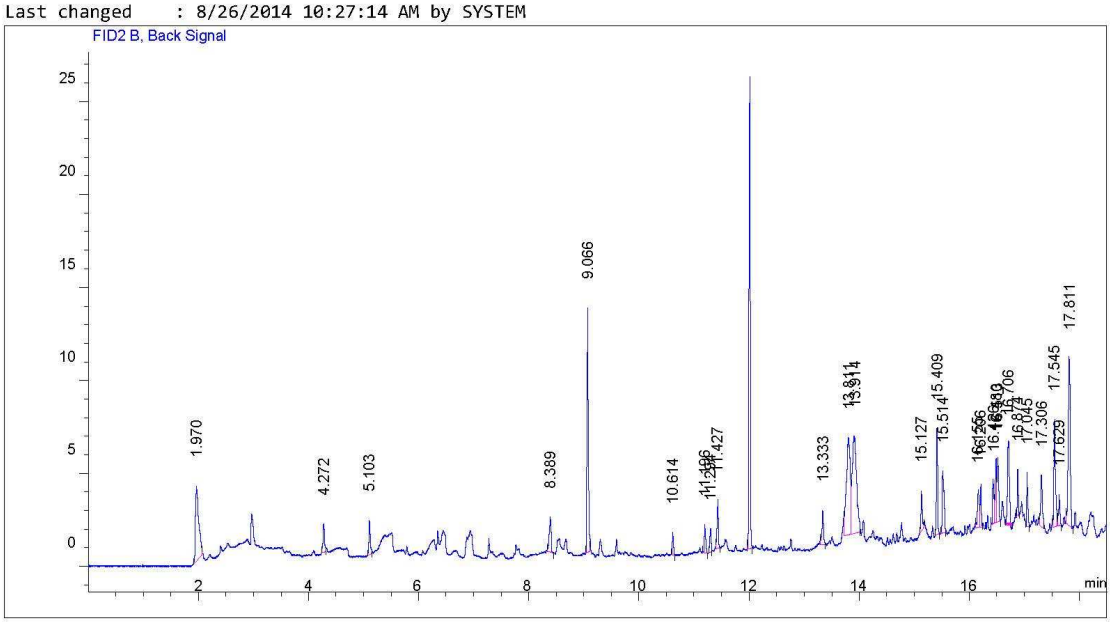


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	1.970	BB	0.0583	16.06396	3.98174	6.04529
2	4.272	BB	0.0311	3.06118	1.49406	1.15200
3	5.103	BB	0.0265	3.18359	1.84787	1.19807
4	8.389	BB	0.0358	4.34001	1.87270	1.63326
5	9.066	BB	0.0261	22.16271	13.15432	8.34041
6	10.614	BB	0.0234	1.82944	1.19683	0.68847
7	11.196	BB	0.0268	2.52271	1.50326	0.94936
8	11.294	BB	0.0244	2.02060	1.25072	0.76040
9	11.427	BB	0.0269	4.45487	2.53235	1.67648
10	12.007	BB	0.0271	21.55905	25.28353	16.22694

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	13.333	BB	0.0272	3.39225	1.82858	1.27659
12	13.811	BV	0.0597	25.43144	5.18013	9.57052
13	13.914	VB	0.0694	29.65500	5.22301	11.15996
14	15.127	BB	0.0248	3.66933	2.13694	1.38087
15	15.409	BB	0.0248	8.99295	5.68701	3.38428
16	15.514	BB	0.0316	7.63617	3.32252	2.87369
17	16.155	BV	0.0299	4.52840	1.92058	1.70415
18	16.206	VB	0.0225	3.29990	2.27550	1.24184
19	16.426	BV	0.0276	4.43446	2.35331	1.66880
20	16.480	VV	0.0233	5.34346	3.36484	2.01088
21	16.513	VB	0.0310	6.89954	3.38188	2.59648
22	16.706	BB	0.0336	10.05875	4.31981	3.78537
23	16.874	BB	0.0191	2.81761	2.43863	1.06034
24	17.045	BB	0.0220	4.08860	2.90065	1.53865
25	17.306	BB	0.0322	6.03452	2.73290	2.27095
26	17.545	BB	0.0299	11.59782	5.75961	4.36457
27	17.629	BB	0.0252	2.77262	1.65056	1.04341
28	17.811	BB	0.0345	22.31562	8.99717	8.39796
Totals :				265.72685	119.59101	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/2/2014 3:40:58 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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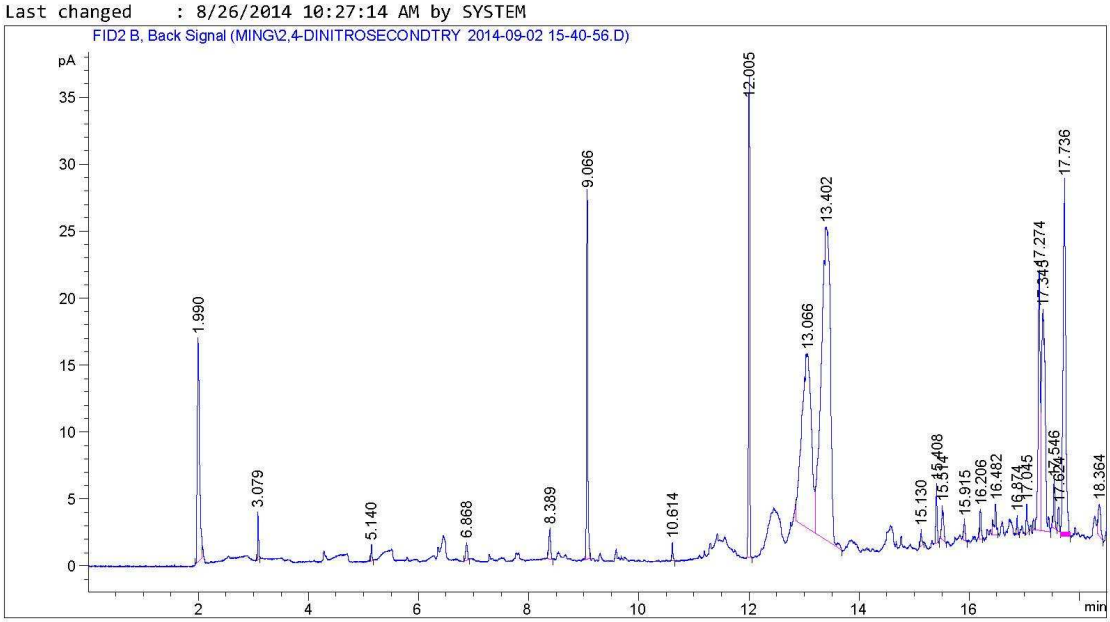


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	1.990	BB	0.0440	48.03417	16.67493	5.57789
2	3.079	BB	0.0176	4.16244	3.56796	0.48336
3	5.140	BB	0.0197	1.56029	1.22407	0.18119
4	6.868	BB	0.0345	2.87725	1.30722	0.33412
5	8.389	BB	0.0333	5.08077	2.27143	0.59000
6	9.066	BB	0.0254	44.64479	27.37809	5.18431
7	10.614	BB	0.0248	2.15197	1.36810	0.24989
8	12.005	BB	0.0258	59.41027	35.69278	6.89892
9	13.066	BV	0.1442	157.26823	13.01382	18.26253
10	13.402	VB	0.1404	269.57373	23.33843	31.30383

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	15.130	BB	0.0254	1.98631	1.17181	0.23066
12	15.408	BB	0.0233	6.59885	4.35815	0.76628
13	15.514	BB	0.0332	6.33383	2.52821	0.73551
14	15.915	BB	0.0288	2.79289	1.50539	0.32432
15	16.206	BB	0.0268	3.99933	2.20326	0.46442
16	16.482	BB	0.0344	5.67154	2.23513	0.65860
17	16.874	BB	0.0175	1.56606	1.35632	0.18186
18	17.045	BB	0.0221	2.97041	2.09867	0.34493
19	17.274	BV	0.0387	50.90965	19.33833	5.91180
20	17.345	VB	0.0571	77.16342	16.46769	8.96048
21	17.546	BB	0.0282	6.32874	3.52427	0.73492
22	17.624	BV	0.0263	3.24324	1.76297	0.37662
23	17.736	VB	0.0418	89.32533	26.42923	10.37276
24	18.364	BB	0.0430	7.49911	2.34127	0.87082
Totals :				861.15261	213.15752	

*** End of Report ***

```
==========================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/2/2014 4:12:26 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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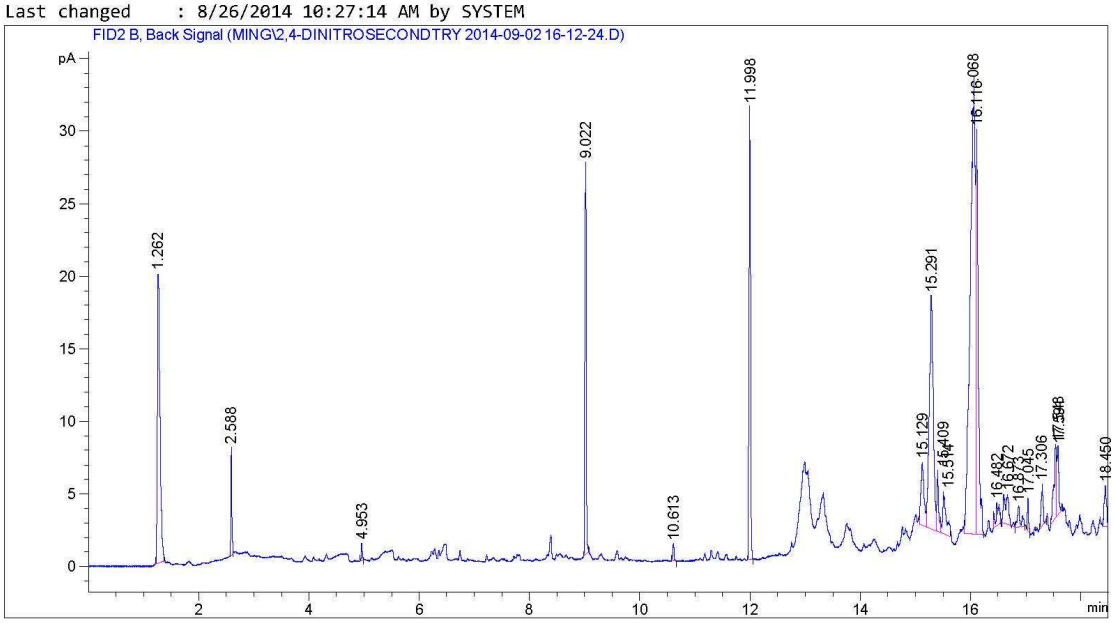

```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]         %
----|-------|----|-------|----------|----------|--------|
   1   1.262 BB    0.0510   66.55877   19.92396 10.29144
   2   2.588 BB    0.0189    8.87983    7.37149  1.37302
   3   4.953 BB    0.0203    1.50119    1.13180  0.23212
   4   9.022 BB    0.0244   41.50256   26.93688  6.41720
   5  10.613 BB    0.0271    1.95303    1.14678  0.30198
   6  11.998 BB    0.0318   61.39054   31.14692  9.49232
   7  15.129 BV    0.0506   16.74017    4.33878  2.58840
   8  15.291 VV    0.0635   79.48488   16.01171 12.29010
   9  15.409 VB    0.0313    8.68930    4.21052  1.34356
  10  15.514 BB    0.0589   13.26252    2.86249  2.05068

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]         %
----|-------|----|-------|----------|----------|--------|
  11  16.068 BV    0.0797  204.43306   31.49420 31.60982
  12  16.116 VB    0.0383   76.07550   27.85968 11.76294
  13  16.482 BB    0.0490    5.76361    1.52293  0.89118
  14  16.672 BV    0.0698   11.69947    2.07280  1.80899
  15  16.873 VB    0.0470    5.17173    1.45876  0.79966
  16  17.045 BB    0.0201    2.90692    2.21321  0.44947
  17  17.306 BB    0.0330    6.39064    2.72067  0.98813
  18  17.548 BV    0.0395   14.57195    5.01773  2.25314
  19  17.591 VB    0.0329   11.66923    4.58618  1.80432
  20  18.450 BBA   0.0398    8.09415    2.82427  1.25153

Totals :                   646.73906  196.85176

=====================================================================
                        *** End of Report ***
```

```
==========================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/2/2014 5:10:36 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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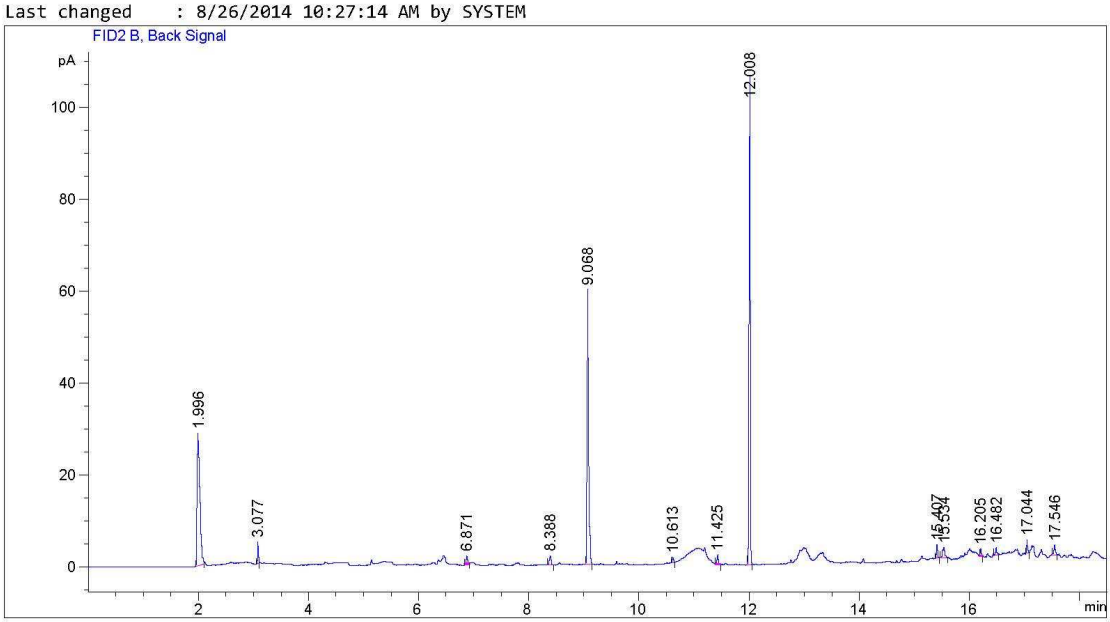


```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   1.996 BB    0.0491   99.37468   28.73645 23.29948
   2   3.077 BB    0.0183    5.55749    4.80020  1.30301
   3   6.871 BB    0.0276    3.10898    1.78194  0.72893
   4   8.388 BB    0.0353    4.24786    1.87100  0.99596
   5   9.068 BB    0.0284  112.70622   59.75512 26.42521
   6  10.613 BB    0.0243    1.87421    1.22118  0.43943
   7  11.425 BB    0.0244    2.98847    1.93461  0.70068
   8  12.008 BB    0.0265  133.82628  104.83421 40.75547
   9  15.407 BB    0.0236    4.40938    2.86213  1.03383
  10  15.534 BB    0.0386    6.22812    2.14928  1.46025
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	16.205	BB	0.0197	1.89088	1.56864	0.44334
12	16.482	BB	0.0248	2.83915	1.59160	0.66567
13	17.044	BB	0.0190	3.54390	2.90958	0.83091
14	17.546	BB	0.0282	3.91465	2.09024	0.91783
Totals :				426.51027	218.10617	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/2/2014 4:40:49 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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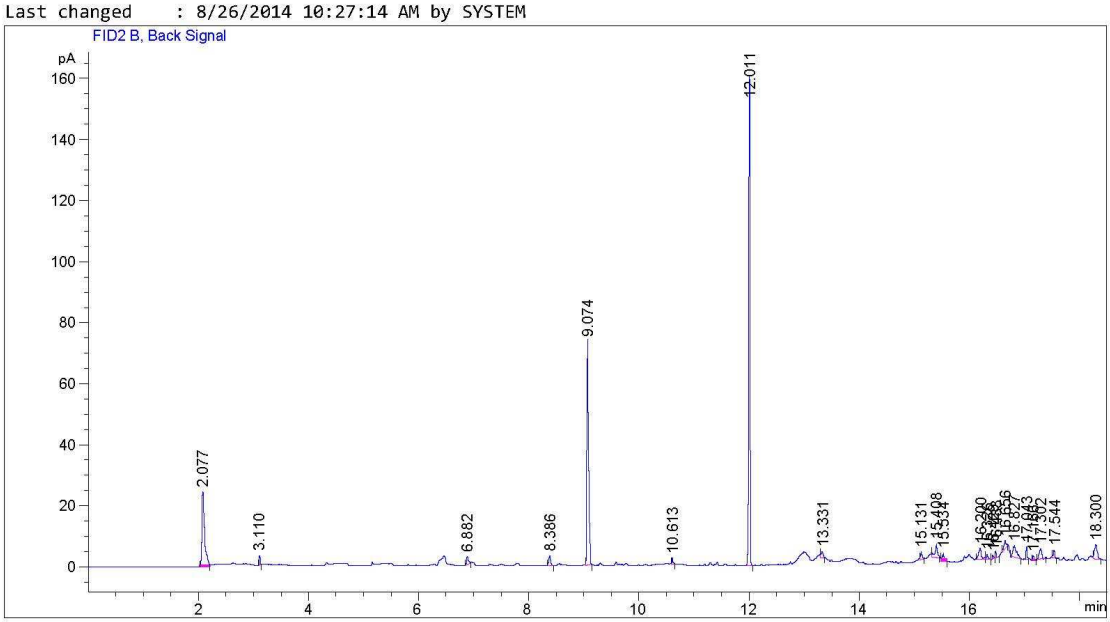


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	2.077	BB	0.0506	82.24731	24.35964	12.85801
2	3.110	BB	0.0184	3.59610	3.07769	0.56219
3	6.882	BB	0.0332	5.64572	2.53530	0.88261
4	8.386	BB	0.0353	6.88889	3.12714	1.07696
5	9.074	BB	0.0325	84.96680	73.24505	24.85945
6	10.613	BB	0.0242	2.97963	1.87115	0.46582
7	12.011	BB	0.0262	137.89635	158.90376	42.07844
8	13.331	BB	0.0328	4.86125	2.08583	0.75998
9	15.131	BB	0.0297	5.05978	2.37346	0.79101
10	15.408	BB	0.0444	14.33725	4.40397	2.24139

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	15.534	BB	0.0242	3.78222	2.27991	0.59129
12	16.200	BB	0.0456	12.07781	3.59047	1.88817
13	16.326	BB	0.0303	3.40187	1.60907	0.53183
14	16.429	BV	0.0183	1.27177	1.16597	0.19882
15	16.483	VB	0.0192	2.69210	2.18386	0.42086
16	16.656	BB	0.0338	6.57324	2.44187	1.02762
17	16.827	BB	0.0540	17.24153	3.96272	2.69543
18	17.043	BB	0.0204	5.33293	3.97647	0.83372
19	17.166	BB	0.0218	1.81636	1.37248	0.28396
20	17.302	BB	0.0419	10.23135	3.28929	1.59950
21	17.544	BB	0.0264	4.59040	2.57574	0.71763
22	18.300	BB	0.0450	16.85707	4.61315	2.63532
Totals :				639.65835	309.04400	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/3/2014 1:45:51 PM
                                                  Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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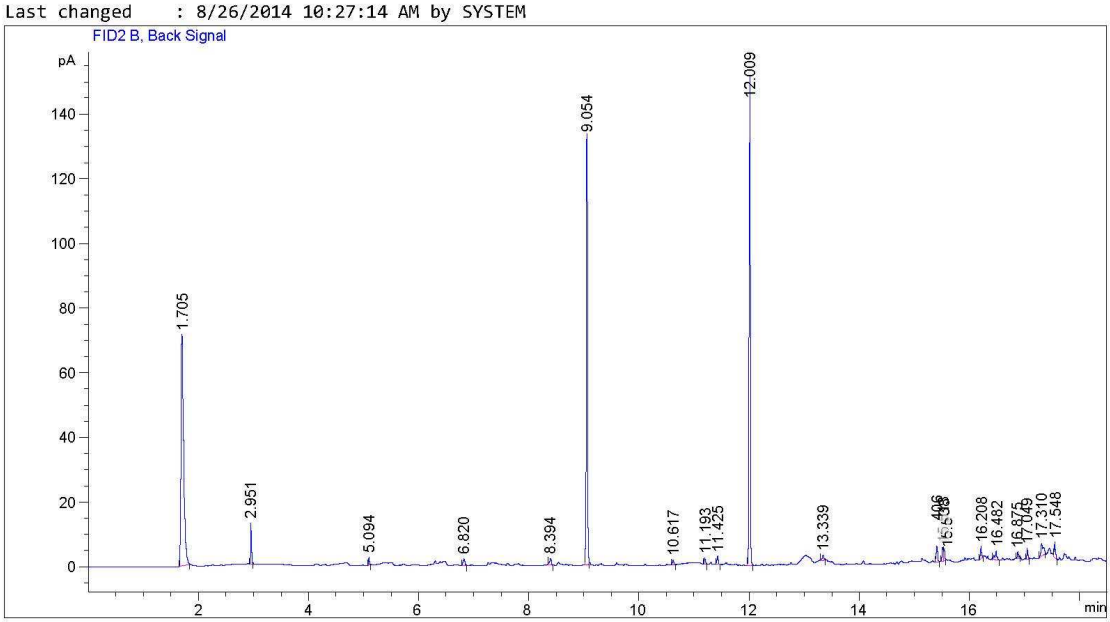


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	1.705	BB	0.0505	240.54088	71.44025	29.69033
2	2.951	BB	0.0173	14.40396	12.59237	1.77790
3	5.094	BB	0.0199	3.32482	2.56402	0.41039
4	6.820	BB	0.0313	4.31655	2.08620	0.53280
5	8.394	BB	0.0343	4.37408	1.88022	0.53990
6	9.054	BB	0.0252	213.89001	132.43100	26.40077
7	10.617	BB	0.0258	2.23591	1.40621	0.27598
8	11.193	BB	0.0242	3.05202	1.91517	0.37671
9	11.425	BB	0.0268	4.49948	2.57592	0.55538
10	12.009	BB	0.0261	254.16539	150.33827	31.37202

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	13.339	BB	0.0328	3.99725	1.66635	0.49339
12	15.406	BB	0.0279	8.99624	4.87046	1.11042
13	15.513	BV	0.0240	6.82049	4.33278	0.84186
14	15.538	VB	0.0204	4.92587	3.49678	0.60801
15	16.208	BB	0.0252	5.87492	3.64220	0.72515
16	16.482	BB	0.0355	7.00468	2.66497	0.86460
17	16.875	BB	0.0288	4.17447	2.09653	0.51526
18	17.049	BB	0.0209	4.31595	3.29646	0.53272
19	17.310	BB	0.0430	12.26715	3.91403	1.51415
20	17.548	BB	0.0243	6.98569	4.54360	0.86225
Totals :				810.16582	413.75379	

==========

*** End of Report ***

```
==========================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/3/2014 6:50:15 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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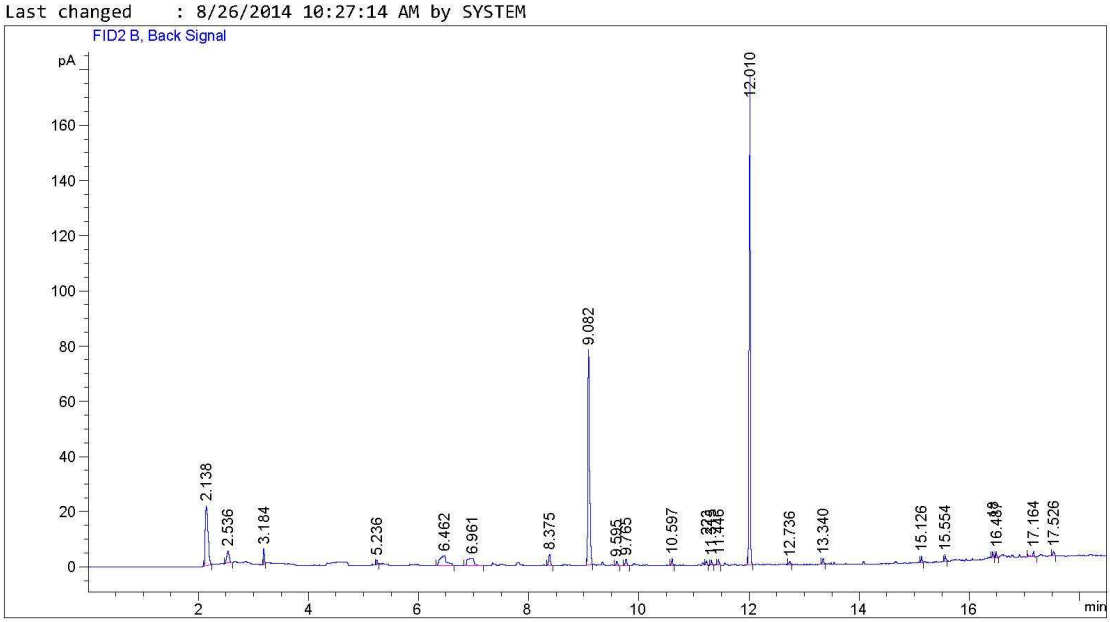



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

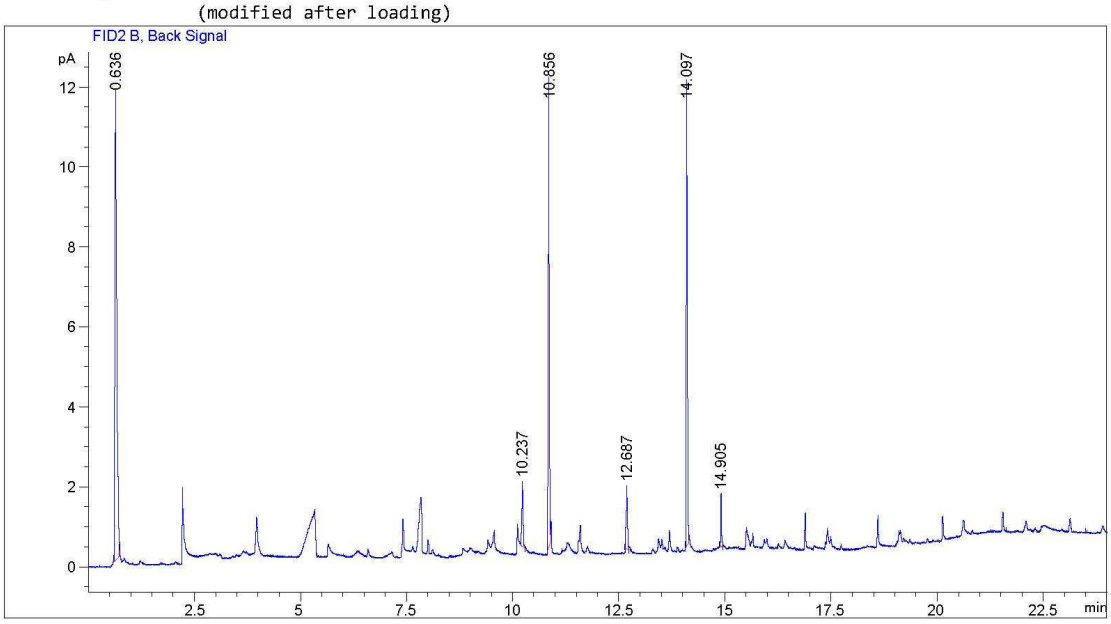

Signal 1: FID2 B, Back Signal
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	2.138	BB	0.0563	75.37549	21.79466	11.36451
2	2.536	BB	0.0414	13.19777	4.49593	1.98985
3	3.184	BB	0.0185	6.57942	5.60655	0.99199
4	5.236	BB	0.0217	2.34656	1.70111	0.35380
5	6.462	BB	0.1052	29.43476	3.59097	4.43794
6	6.961	BB	0.0979	20.23513	2.51422	3.05089
7	8.375	BB	0.0352	8.79771	3.89197	1.32645
8	9.082	BB	0.0318	203.58347	77.88902	24.74721
9	9.595	BB	0.0296	2.68116	1.44572	0.40424
10	9.765	BB	0.0351	4.59495	2.09929	0.69279

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	10.597	BB	0.0247	4.01246	2.55150	0.60497
12	11.223	BB	0.0235	2.33175	1.51858	0.35156
13	11.315	BB	0.0253	2.75424	1.70174	0.41526
14	11.446	BB	0.0241	3.25376	2.14424	0.49058
15	12.010	BB	0.0255	230.65030	175.46466	45.12008
16	12.736	BB	0.0258	1.93345	1.11430	0.29151
17	13.340	BB	0.0281	3.94568	2.04698	0.59490
18	15.126	BB	0.0204	2.91370	2.29385	0.43930
19	15.554	BB	0.0218	3.44514	2.36819	0.51943
20	16.418	BB	0.0225	3.28049	2.16059	0.49461
21	16.487	BB	0.0199	2.49764	2.03665	0.37657
22	17.164	BB	0.0241	2.65263	1.74363	0.39994
23	17.526	BB	0.0269	3.59217	1.89541	0.54160
Totals :				663.25302	324.06976	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/12/2014 1:42:06 PM
                                                  Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBUR1.M
Last changed    : 3/13/2014 1:08:32 PM by SYSTEM
                  (modified after loading)
```




```
======================================================================
                        Area Percent Report
======================================================================

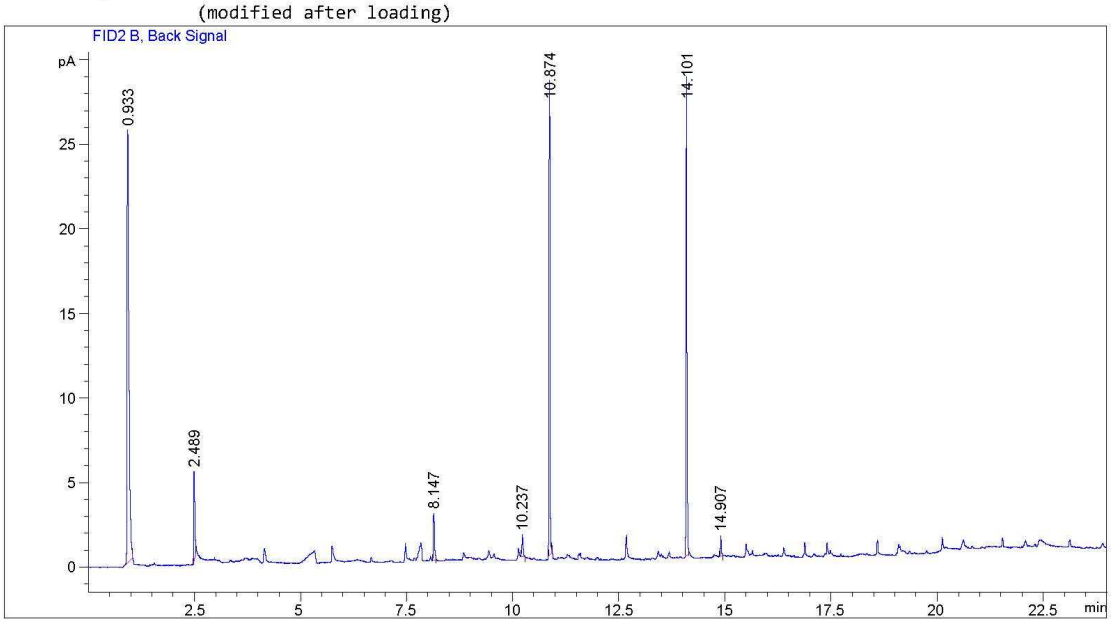
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   0.636 BB    0.0443   35.68917   11.74312 41.44328
   2  10.237 BB    0.0330    3.34893    1.61453  3.88888
   3  10.856 BB    0.0262   19.85652   11.70268 23.05796
   4  12.687 BB    0.0318    3.43237    1.57728  3.98576
   5  14.097 BB    0.0288   21.55905   11.62199 25.03498
   6  14.905 BB    0.0252    2.22966    1.32399  2.58914

Totals :                    86.11570   39.58360
```

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/12/2014 2:37:01 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBUR1.M
Last changed    : 3/12/2014 1:48:29 PM by SYSTEM
                  (modified after loading)
```




```
======================================================================
                        Area Percent Report
======================================================================

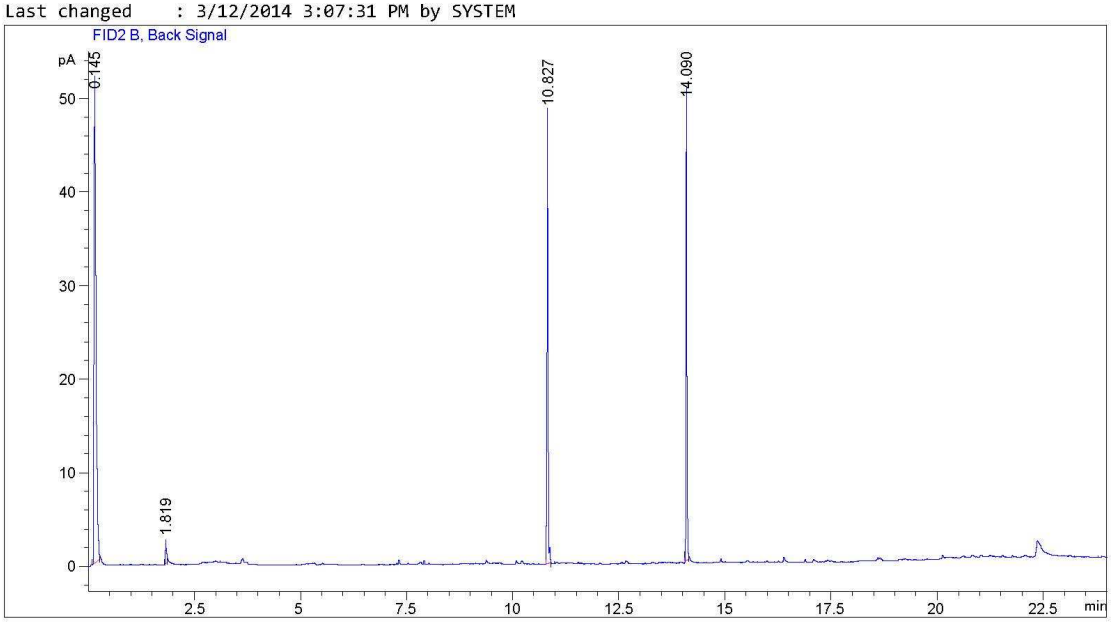
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   0.933 BB    0.0434   75.83232   25.54385 39.65561
   2   2.489 BB    0.0249    8.74024    5.29266  4.57060
   3   8.147 BB    0.0294    5.39771    2.74328  2.82267
   4  10.237 BB    0.0318    2.61209    1.28072  1.36596
   5  10.874 BB    0.0260   46.96103   27.90120 24.55771
   6  14.101 BB    0.0277   49.61527   28.17915 25.94571
   7  14.907 BB    0.0271    2.06858    1.16563  1.08174

Totals :                   191.22724   92.10649
```

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/12/2014 3:12:19 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBUR1.M
Last changed    : 3/12/2014 3:07:31 PM by SYSTEM
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

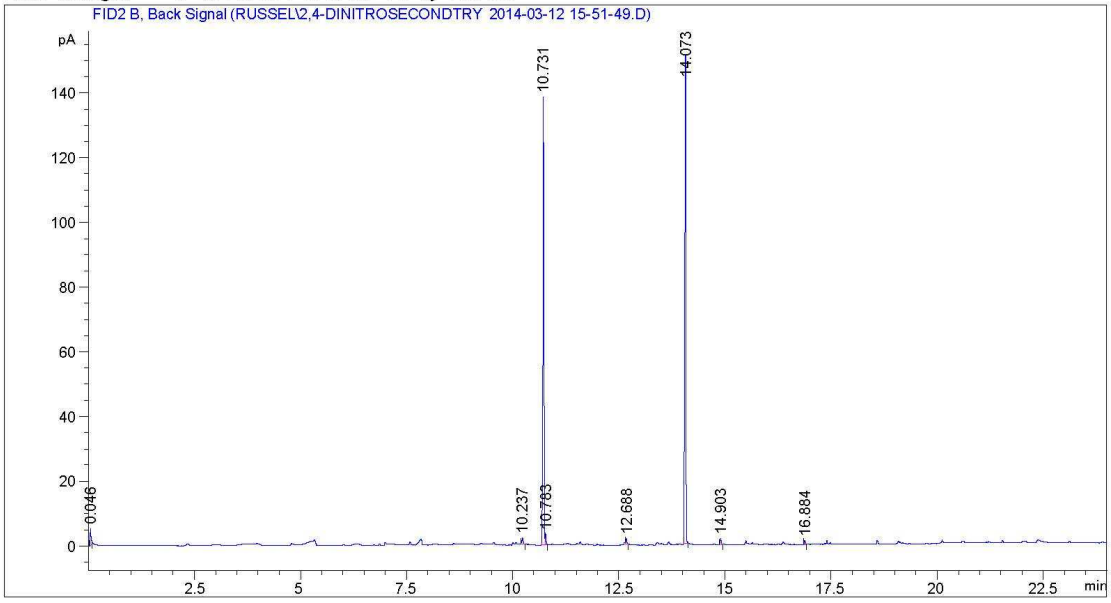
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   0.145 BB    0.0480  167.47295   51.97122 48.57034
   2   1.819 BB    0.0264    4.46890    2.50878  1.29607
   3  10.827 BB    0.0269   95.70641   48.46788 24.64199
   4  14.090 BB    0.0276  110.06602   50.31339 25.49161

Totals :                   344.80499  153.26126


======================================================================
                        *** End of Report ***
```

Data File C:\CHEM32\1\DATA\RUSSEL\2,4-DINITROSECONDTRY 2014-03-12 15-51-49.D
Sample Name: 2,4-dinitrosecondtry

```
==========================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/12/2014 3:51:49 PM
                                                  Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBUR1.M
Last changed    : 3/12/2014 3:07:31 PM by SYSTEM
```




```
==========================================================================
                          Area Percent Report
==========================================================================

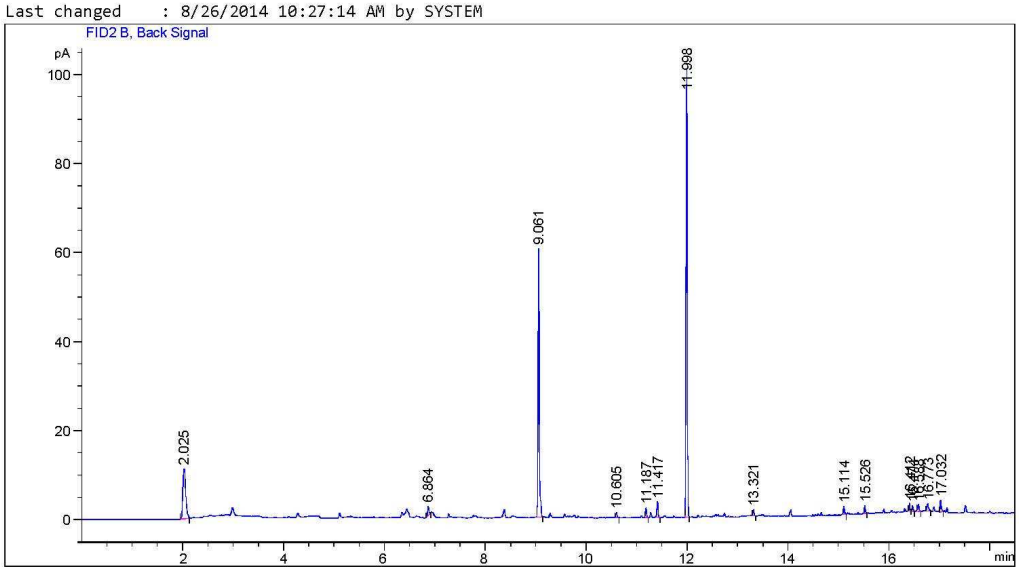
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   0.046 BB    0.0266    8.71883    5.04195  1.66404
   2  10.237 BB    0.0295    3.72343    1.94605  0.71064
   3  10.731 BV    0.0265  205.07344  138.37621 45.41830
   4  10.783 VB    0.0252    5.23641    3.24237  0.99940
   5  12.688 BB    0.0304    3.79333    1.84558  0.72398
   6  14.073 BB    0.0272  232.90698  150.68500 49.42045
   7  14.903 BB    0.0282    3.48836    1.86296  0.66577
   8  16.884 BB    0.0259    2.08229    1.24461  0.39742

Totals :                   523.95561  304.24471
```

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/9/2014 5:20:32 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

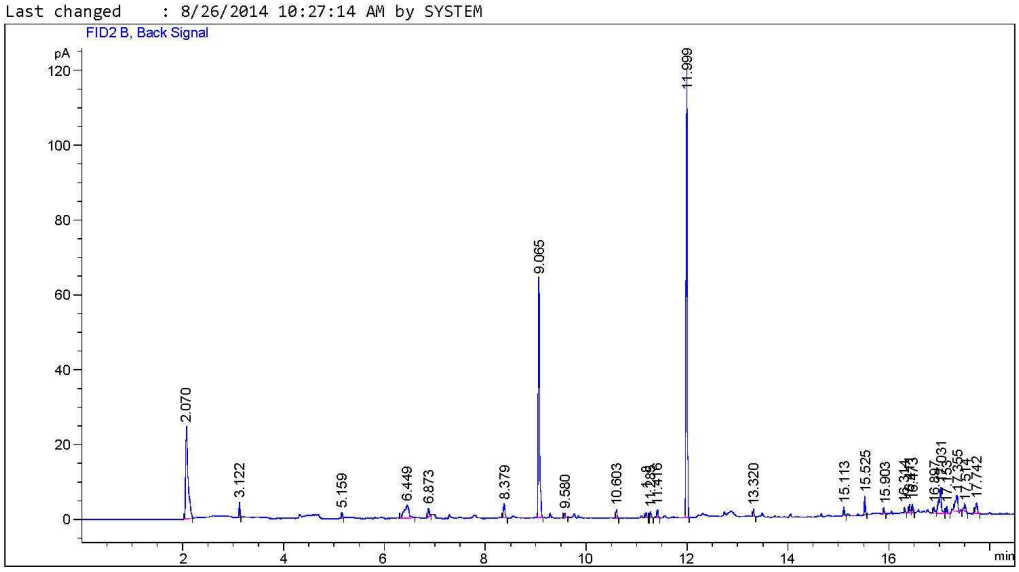

Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   2.025 BB    0.0628   44.10668   11.19005 12.50010
   2   6.864 BB    0.0294    4.08716    2.07560  1.15833
   3   9.061 BB    0.0265  113.08270   60.21674 29.41775
   4  10.605 BB    0.0253    1.85928    1.19553  0.52693
   5  11.187 BB    0.0256    3.39809    2.06575  0.96304
   6  11.417 BB    0.0261    5.55225    3.41963  1.57354
   7  11.998 BB    0.0270  169.31157   99.51895 47.98393
   8  13.321 BB    0.0230    2.10905    1.35306  0.59772
   9  15.114 BB    0.0217    2.47796    1.71255  0.70227
  10  15.526 BB    0.0195    2.28050    1.71241  0.64631
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	16.412	BB	0.0190	2.25238	1.74849	0.63834
12	16.474	BB	0.0197	1.56293	1.16371	0.44294
13	16.588	BB	0.0290	2.64427	1.36516	0.74940
14	16.773	BB	0.0316	3.55029	1.59045	1.00617
15	17.032	BB	0.0223	3.85742	2.56995	1.09322
Totals :				352.85054	192.89802	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/9/2014 5:51:08 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

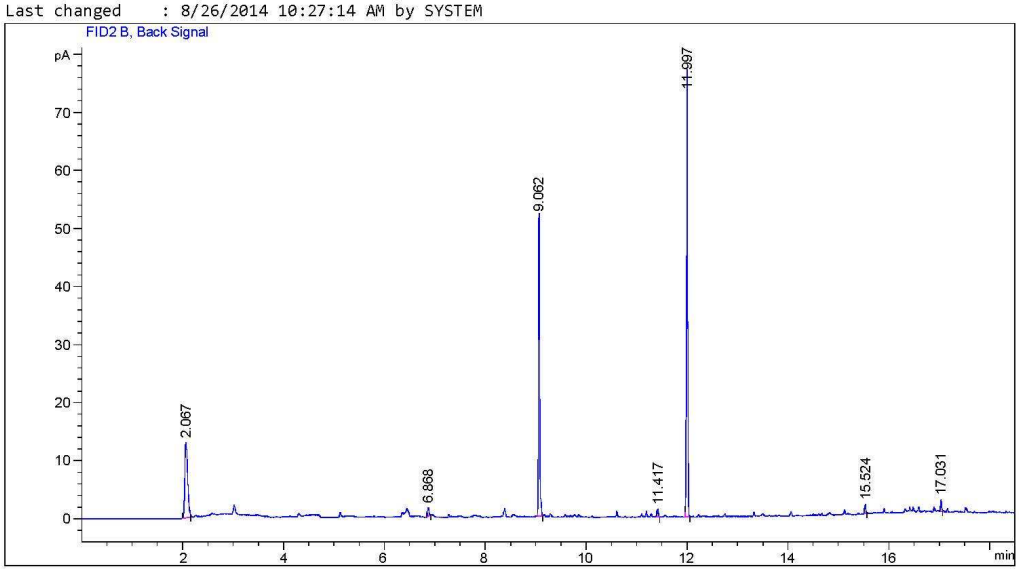

Signal 1: FID2 B, Back Signal
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	2.070	BB	0.0489	82.95724	24.61293	15.66761
2	3.122	BB	0.0184	4.76463	4.09442	0.89987
3	5.159	BB	0.0191	1.84534	1.50896	0.34852
4	6.449	BB	0.0801	21.99085	3.44291	4.15327
5	6.873	BB	0.0300	4.43133	2.27065	0.83692
6	8.379	BB	0.0346	8.34553	3.78081	1.57617
7	9.065	BB	0.0281	118.59631	63.86641	22.39854
8	9.580	BB	0.0278	2.18821	1.23705	0.41327
9	10.603	BB	0.0241	3.42149	2.25303	0.64619
10	11.188	BB	0.0260	2.13530	1.32691	0.40328

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	11.285	BB	0.0254	2.57703	1.57893	0.48671
12	11.416	BB	0.0237	3.20129	2.06425	0.60461
13	11.999	BB	0.0266	197.67497	118.90600	37.33362
14	13.320	BB	0.0235	2.90073	1.89333	0.54784
15	15.113	BB	0.0220	3.11550	2.21397	0.58840
16	15.525	BB	0.0204	6.39644	4.79287	1.20806
17	15.903	BB	0.0233	2.43929	1.60931	0.46069
18	16.314	BB	0.0202	1.87580	1.49581	0.35427
19	16.411	BB	0.0213	3.13910	2.21623	0.59286
20	16.473	BB	0.0213	3.18366	2.24859	0.60128
21	16.897	BB	0.0278	3.00516	1.57938	0.56757
22	17.031	BB	0.0399	20.88881	6.93789	3.94514
23	17.153	BB	0.0238	2.79219	1.78939	0.52734
24	17.355	BB	0.0511	15.04972	4.14946	2.84235
25	17.514	BB	0.0281	3.16755	1.63930	0.59823
26	17.742	BB	0.0335	7.39897	2.84893	1.39740
Totals :				529.48243	266.35772	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/10/2014 4:39:03 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
```




```
======================================================================
                        Area Percent Report
======================================================================

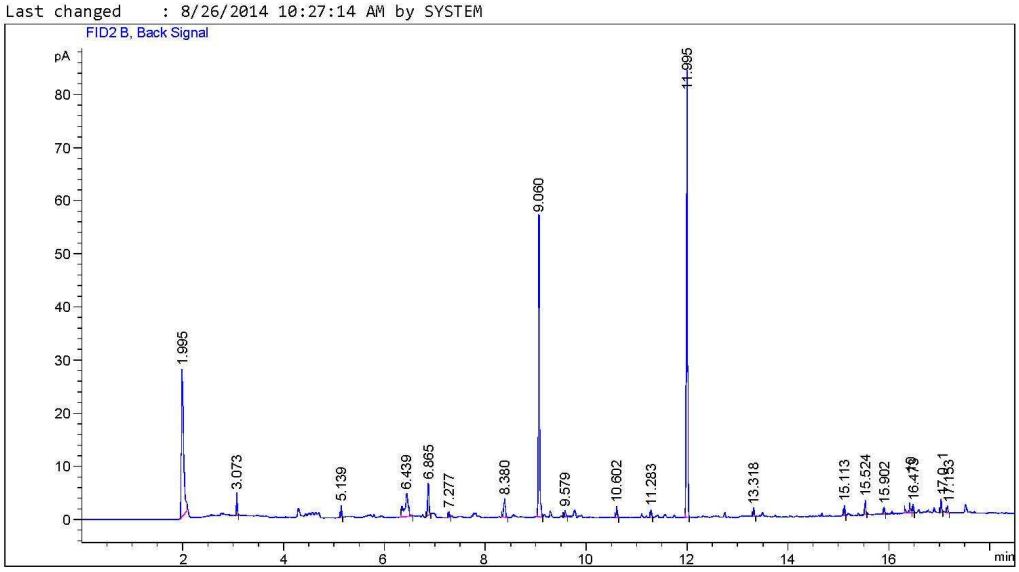
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   2.067 BB    0.0567   46.82768   12.91628 17.38420
   2   6.868 BB    0.0283    2.67705    1.47780  0.99382
   3   9.062 BB    0.0258   86.10667   51.73782 31.96604
   4  11.417 BB    0.0236    2.00403    1.29768  0.74397
   5  11.997 BB    0.0256  128.38260   76.89127 47.01545
   6  15.524 BB    0.0227    2.38456    1.62604  0.88524
   7  17.031 BB    0.0198    2.72407    2.01081  1.01128

Totals :                   269.36923  147.95770
```

```
==========================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/10/2014 5:01:53 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

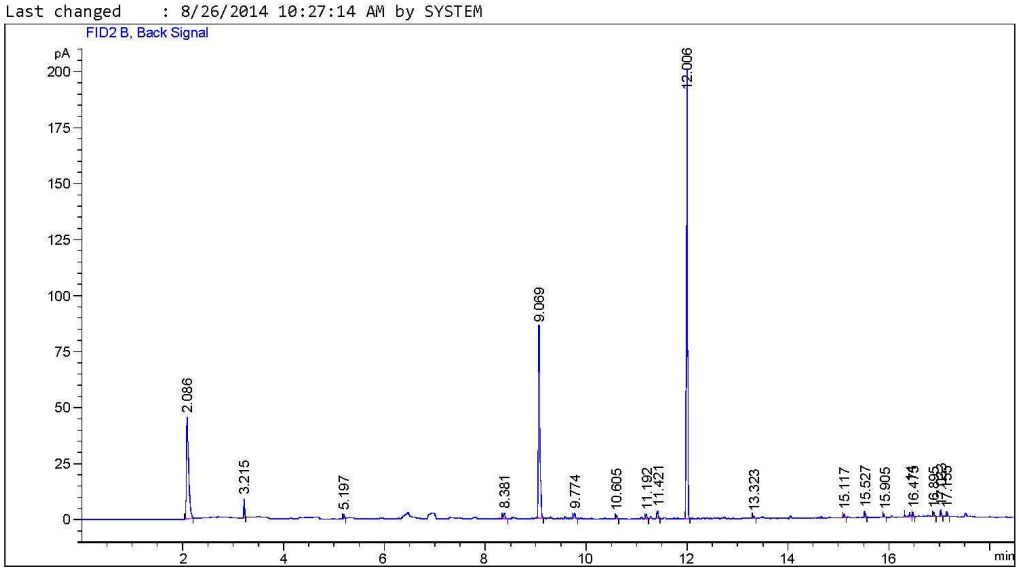

Signal 1: FID2 B, Back Signal
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	1.995	BB	0.0441	81.48457	27.55722	20.76828
2	3.073	BB	0.0176	4.88649	4.18833	1.24544
3	5.139	BB	0.0177	2.41557	2.18612	0.61566
4	6.439	BB	0.0651	20.65745	4.27875	5.26504
5	6.865	BB	0.0279	10.69673	6.02866	2.72632
6	7.277	BB	0.0226	1.53926	1.05465	0.39232
7	8.380	BB	0.0366	7.77819	3.36649	1.98246
8	9.060	BB	0.0254	91.97424	56.48001	23.44182
9	9.579	BB	0.0283	2.13639	1.17991	0.54451
10	10.602	BB	0.0248	3.18571	2.02463	0.81195

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	11.283	BB	0.0242	2.04782	1.28180	0.52194
12	11.995	BB	0.0261	144.36490	83.93921	36.08651
13	13.318	BB	0.0236	2.33481	1.51389	0.59508
14	15.113	BB	0.0209	2.53509	1.83867	0.64613
15	15.524	BB	0.0211	3.61244	2.58884	0.92072
16	15.902	BB	0.0214	1.72198	1.21304	0.43889
17	16.410	BV	0.0336	4.48210	1.81479	1.14237
18	16.473	VB	0.0201	2.06956	1.50037	0.52748
19	17.031	BB	0.0207	3.41875	2.50348	0.87135
20	17.153	BB	0.0227	1.78814	1.21916	0.45575
Totals :				392.35117	207.75801	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/11/2014 4:40:25 PM
                                                  Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

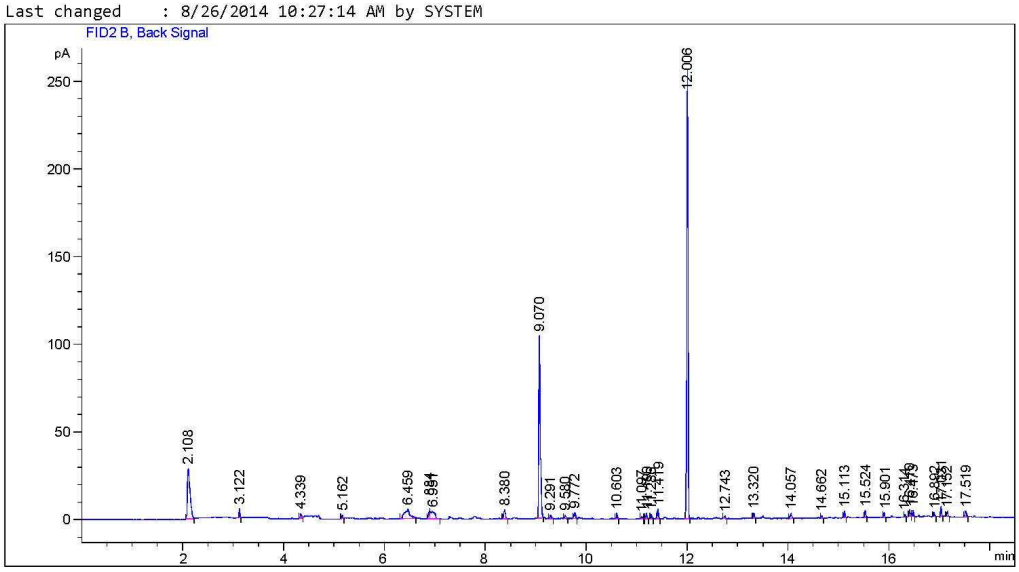

Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   2.086 BB    0.0492  147.40874   45.29082 20.44886
   2   3.215 BB    0.0170    9.13790    8.21869  1.26763
   3   5.197 BB    0.0194    2.19881    1.76182  0.30502
   4   8.381 BB    0.0344    5.49711    2.42794  0.76257
   5   9.069 BB    0.0316  179.39140   85.65450 24.88556
   6   9.774 BB    0.0312    4.80034    2.41038  0.66591
   7  10.605 BB    0.0244    2.41775    1.57094  0.33540
   8  11.192 BB    0.0254    3.18338    1.95879  0.44160
   9  11.421 BB    0.0247    5.34929    3.41301  0.74206
  10  12.006 BB    0.0261  336.37372  198.79266 46.66249
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	13.323	BB	0.0264	1.97242	1.15292	0.27362
12	15.117	BB	0.0214	2.12410	1.57146	0.29466
13	15.527	BB	0.0209	3.42595	2.47875	0.47525
14	15.905	BB	0.0206	1.67913	1.30838	0.23293
15	16.414	BV	0.0316	4.14678	1.80398	0.57525
16	16.475	VB	0.0221	2.89009	2.04562	0.40092
17	16.895	BB	0.0222	2.38195	1.67338	0.33043
18	17.033	BB	0.0211	3.93607	2.82037	0.54602
19	17.155	BB	0.0210	2.55045	1.93419	0.35380
Totals :				720.86537	368.28859	

*** End of Report ***

```
================================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/11/2014 5:01:23 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
```




```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	2.108	BB	0.0523	103.20856	28.74597	10.85605
2	3.122	BB	0.0180	5.86885	5.18483	0.61732
3	4.339	BB	0.0279	3.85106	2.09068	0.40508
4	5.162	BB	0.0214	2.31091	1.70282	0.24307
5	6.459	BB	0.0964	38.93384	5.23090	4.09528
6	6.884	BV	0.0295	8.71383	4.24639	0.91657
7	6.951	VB	0.0732	23.93051	4.07968	2.51714
8	8.380	BB	0.0354	10.94492	4.94635	1.15125
9	9.070	BB	0.0312	221.29782	103.94129	23.27732
10	9.291	BB	0.0314	3.86040	1.86108	0.40606

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	9.580	BB	0.0273	3.30927	1.92298	0.34809
12	9.772	BV	0.0331	6.82815	3.27709	0.71822
13	10.603	BB	0.0242	4.45910	2.92609	0.46903
14	11.097	BB	0.0260	1.96248	1.16671	0.20642
15	11.190	BB	0.0253	5.11701	3.15757	0.53823
16	11.286	BB	0.0236	3.81763	2.37524	0.40156
17	11.419	BB	0.0246	8.44276	5.42198	0.88806
18	12.006	BB	0.0271	434.43048	255.03728	45.69579
19	12.743	BB	0.0249	2.07817	1.31067	0.21859
20	13.320	BB	0.0241	4.60586	3.02752	0.48447
21	14.057	BB	0.0292	5.53784	2.74343	0.58250
22	14.662	BB	0.0213	1.92005	1.35702	0.20196
23	15.113	BB	0.0217	5.05553	3.65338	0.53177
24	15.524	BB	0.0217	5.42680	3.94297	0.57082
25	15.901	BB	0.0209	3.18140	2.43065	0.33464
26	16.314	BB	0.0191	1.65979	1.35670	0.17459
27	16.410	BB	0.0210	5.13589	3.69687	0.54022
28	16.473	BB	0.0217	4.71741	3.42068	0.49620
29	16.892	BB	0.0211	2.87150	2.15956	0.30204
30	17.031	BB	0.0194	7.29094	5.51761	0.76690
31	17.152	BB	0.0230	4.00102	2.81614	0.42085
32	17.519	BB	0.0278	5.93148	3.11134	0.62391
Totals :				950.70128	477.85948	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/12/2014 4:30:30 PM
                                                  Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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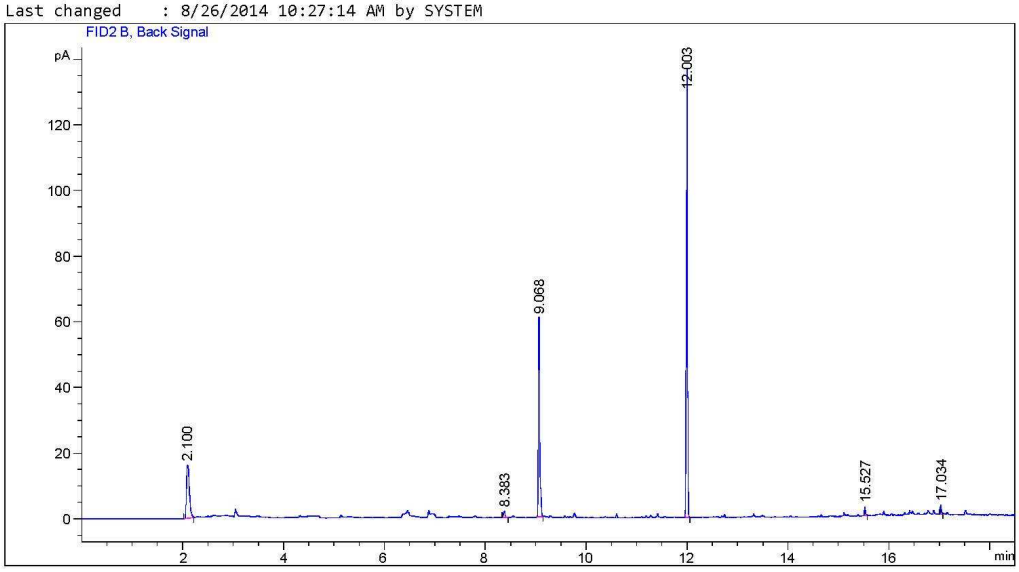



```
======================================================================
                        Area Percent Report
======================================================================

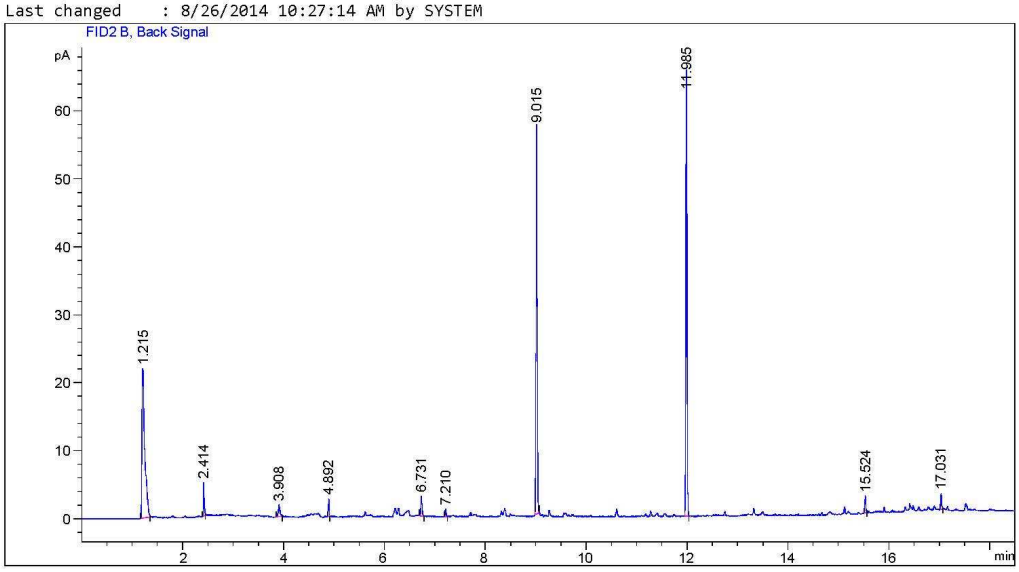
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   2.100 BB    0.0670   67.08669   16.11106 16.03610
   2   8.383 BB    0.0358    4.11630    1.83320  0.98394
   3   9.068 BB    0.0295  115.70696   60.62465 27.65806
   4  12.003 BB    0.0266  224.10210  135.10495 53.56834
   5  15.527 BB    0.0212    3.54513    2.64566  0.84741
   6  17.034 BB    0.0216    3.79082    2.75905  0.90614

Totals :                   418.34800  219.07857
```

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/12/2014 4:52:23 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	1.215	BB	0.0568	85.71198	21.95323	27.75679
2	2.414	BB	0.0196	6.24177	4.91347	2.02132
3	3.908	BB	0.0400	4.38299	1.68416	1.41938
4	4.892	BB	0.0160	2.63781	2.57458	0.85422
5	6.731	BB	0.0271	4.91543	2.88066	1.59180
6	7.210	BB	0.0203	1.46025	1.09952	0.47289
7	9.015	BB	0.0247	89.56506	56.99088	29.00456
8	11.985	BB	0.0255	107.44609	65.57436	34.79512
9	15.524	BB	0.0206	3.36414	2.48707	1.08944
10	17.031	BB	0.0203	3.07097	2.30758	0.99450

Data File C:\CHEM32\1\DATA\MING\2 4-DINITR05ECONDTRY 2014-09-12 16-52-21.D
Sample Name: 2 4-dinitrosecondtry

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area
Totals				308.79648	162.46550	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/13/2014 6:48:30 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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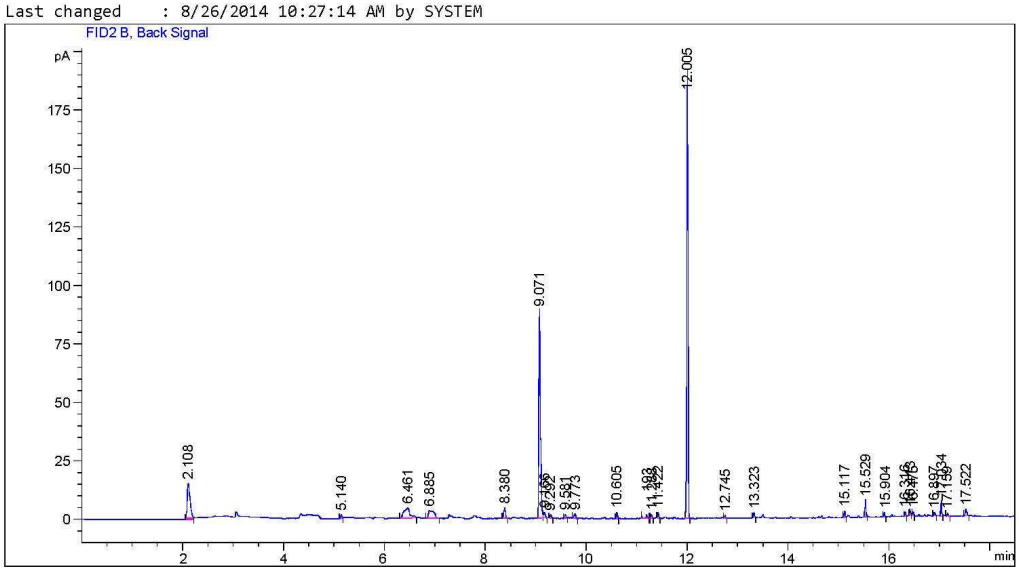


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   2.108 BB    0.0566   58.83424   15.11181  8.07528
   2   5.140 BB    0.0266    2.68857    1.49025  0.36902
   3   6.461 BB    0.1073   35.11559    4.29664  4.81978
   4   6.885 BB    0.0957   25.17822    3.34956  3.45583
   5   8.380 BB    0.0351   10.06313    4.45732  1.38121
   6   9.071 BV    0.0332  197.79253   88.74552 27.14796
   7   9.166 VB    0.0362    5.53806    2.43237  0.76012
   8   9.292 BB    0.0280    2.60350    1.40899  0.35734
   9   9.581 BB    0.0260    2.78219    1.65223  0.38187
  10   9.773 BB    0.0329    3.85140    1.80379  0.52862
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	10.605	BB	0.0239	4.04720	2.69572	0.55550
12	11.193	BB	0.0347	3.40999	1.40501	0.46804
13	11.288	BB	0.0259	2.83283	1.62948	0.38882
14	11.422	BB	0.0266	4.26175	2.45876	0.58495
15	12.005	BB	0.0259	320.07379	191.76987	43.93164
16	12.745	BB	0.0243	1.95292	1.21597	0.26805
17	13.323	BB	0.0238	3.40426	2.17737	0.46725
18	15.117	BB	0.0222	3.74290	2.62447	0.51373
19	15.529	BB	0.0217	9.66845	7.01533	1.32704
20	15.904	BB	0.0214	2.52959	1.86947	0.34720
21	16.316	BB	0.0191	2.24841	1.83435	0.30861
22	16.413	BB	0.0215	4.30446	2.87701	0.59081
23	16.475	BB	0.0247	2.90856	1.70643	0.39921
24	16.897	BB	0.0255	2.59633	1.58560	0.35636
25	17.034	BB	0.0214	8.79710	6.47902	1.20744
26	17.159	BB	0.0255	1.72507	1.14725	0.23677
27	17.522	BB	0.0338	5.62136	2.62519	0.77156
Totals :				728.57239	357.86478	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/13/2014 7:11:29 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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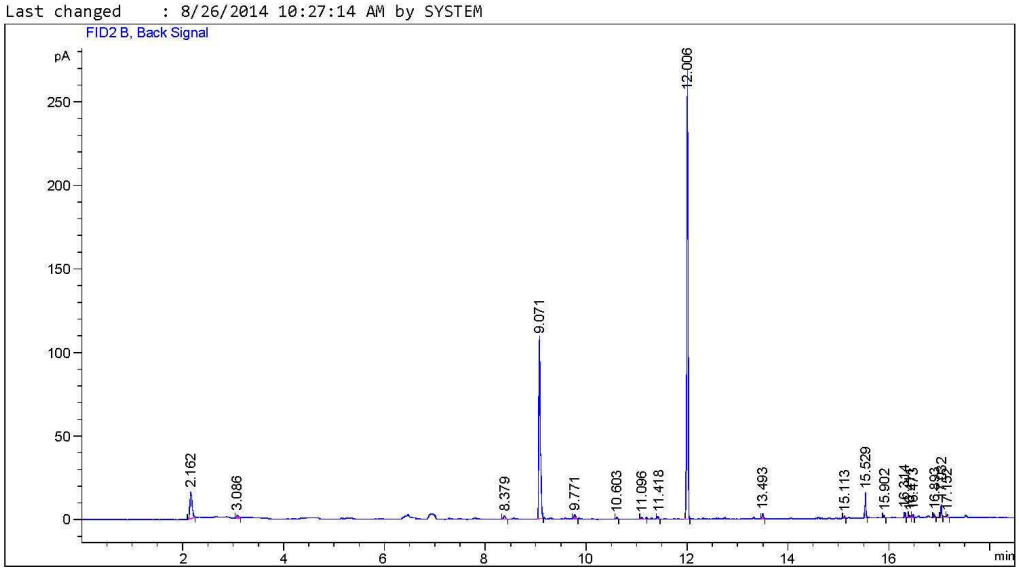


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	2.162	BB	0.0489	48.28025	15.61774	5.98378
2	3.086	BB	0.0364	4.18327	1.77143	0.51847
3	8.379	BB	0.0352	4.59561	2.09201	0.56957
4	9.071	BB	0.0317	234.36523	108.12711	29.04686
5	9.771	BB	0.0307	4.28203	2.37274	0.53071
6	10.603	BB	0.0242	2.11071	1.38660	0.26160
7	11.096	BB	0.0417	3.25310	1.07609	0.40318
8	11.418	BB	0.0249	2.48133	1.56340	0.30753
9	12.006	BB	0.0262	452.29761	266.91202	56.05707
10	13.493	BB	0.0252	4.55957	2.71565	0.56511

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	15.113	BB	0.0223	1.77462	1.23928	0.21994
12	15.529	BB	0.0215	19.97675	14.68700	2.47589
13	15.902	BB	0.0201	2.06063	1.56855	0.25539
14	16.314	BB	0.0180	3.69983	3.08807	0.45855
15	16.411	BB	0.0208	1.64892	1.20154	0.20436
16	16.473	BB	0.0205	2.44364	1.81034	0.30286
17	16.893	BB	0.0226	2.67310	1.83579	0.33130
18	17.032	BB	0.0209	10.06904	7.66357	1.24794
19	17.152	BB	0.0214	2.09687	1.47047	0.25988
Totals :				806.85210	438.19940	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/13/2014 9:24:51 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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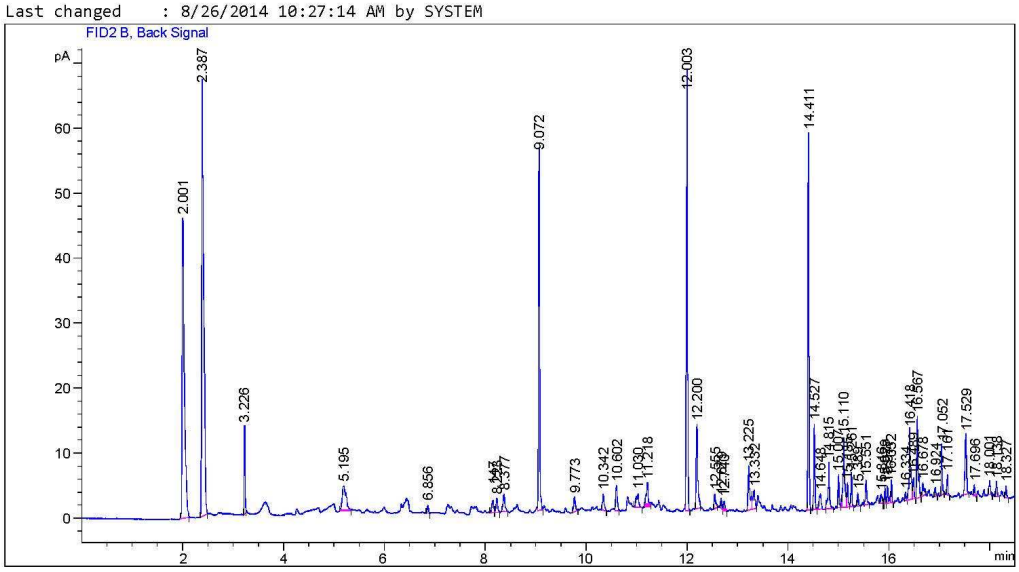


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   2.001 BB    0.0434  140.10239   46.14653 14.24395
   2   2.387 BB    0.0406  192.25746   67.12746 19.54646
   3   3.226 BB    0.0175   15.58609   13.48276  1.58461
   4   5.195 BB    0.0666   20.42891    3.76210  2.07697
   5   6.856 BB    0.0255    2.01737    1.22941  0.20510
   6   8.147 BV    0.0374    4.30304    1.85791  0.43748
   7   8.223 VB    0.0345    4.80848    2.05247  0.48887
   8   8.377 BB    0.0408    7.27087    2.65355  0.73922
   9   9.072 BB    0.0260  103.80070   54.82354  8.97500
  10   9.773 BB    0.0354    5.18717    2.14683  0.52737
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	10.342	BB	0.0350	5.88804	2.47038	0.59863
12	10.602	BB	0.0331	8.53153	3.84004	0.86738
13	11.030	BB	0.0452	6.94851	2.08861	0.70644
14	11.218	BB	0.0375	9.11681	3.50638	0.92689
15	12.003	BB	0.0252	112.36878	66.95345	11.42433
16	12.200	BB	0.0312	27.58734	12.96386	2.80475
17	12.555	BB	0.0306	4.57194	2.20846	0.46482
18	12.679	BV	0.0232	2.35856	1.49420	0.23979
19	12.740	VB	0.0253	2.27217	1.40136	0.23101
20	13.225	BB	0.0330	15.86551	6.74568	1.61302
21	13.332	BV	0.0436	9.08153	2.84817	0.92330
22	14.411	BB	0.0222	82.73916	57.97815	8.41194
23	14.527	BB	0.0242	20.01177	13.11591	2.03456
24	14.648	BB	0.0389	6.62765	2.31910	0.67382
25	14.815	BB	0.0294	14.34967	7.02182	1.45890
26	15.007	BB	0.0230	7.37944	4.95483	0.75025
27	15.110	BV	0.0320	24.48396	10.51713	2.48924
28	15.185	VB	0.0260	6.47405	3.56137	0.65820
29	15.261	BB	0.0225	7.47380	5.14855	0.75985
30	15.389	BB	0.0235	2.90861	1.98655	0.29571
31	15.551	BB	0.0300	6.73395	3.69970	0.68463
32	15.846	BB	0.0305	2.94513	1.33588	0.29943
33	15.929	BV	0.0183	2.94480	2.53907	0.29939
34	15.977	VB	0.0255	4.39148	2.57219	0.44647
35	16.052	BB	0.0211	4.57392	3.43346	0.46502
36	16.334	BV	0.0218	1.77224	1.27778	0.18018
37	16.418	VV	0.0284	22.13166	10.57922	2.25008
38	16.489	VB	0.0254	5.33611	3.15087	0.54251
39	16.567	BB	0.0267	22.73583	12.55242	2.31151
40	16.678	BB	0.0296	4.37948	1.99353	0.44525
41	16.924	BB	0.0251	2.14014	1.28249	0.21758
42	17.052	BB	0.0199	10.64530	7.83391	1.08229
43	17.161	BB	0.0218	4.08163	3.09767	0.41497
44	17.529	BB	0.0325	19.37610	9.23235	1.96993
45	17.696	BB	0.0209	1.82833	1.56241	0.18588
46	18.001	BB	0.0285	3.87423	2.12573	0.39389
47	18.138	BB	0.0243	3.73651	2.23402	0.37988
48	18.327	BB	0.0241	2.68733	1.77206	0.27322
Totals :				983.59225	480.68131	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/13/2014 9:52:08 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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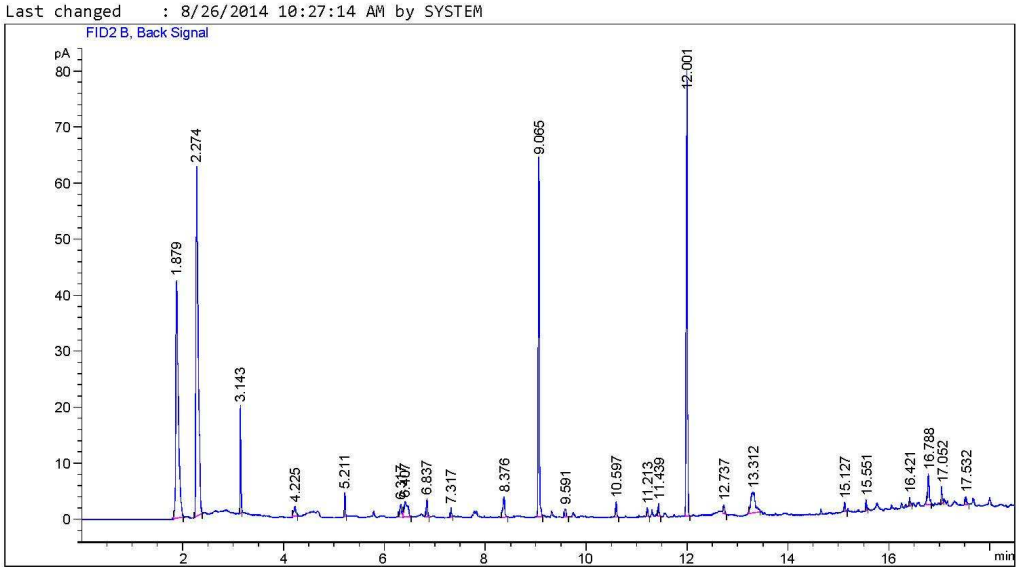


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   1.879 BB    0.0484  143.92323   42.40129 20.34004
   2   2.274 BB    0.0468  194.73294   62.40850 27.52076
   3   3.143 BB    0.0172   21.67665   19.11968  3.06347
   4   4.225 BB    0.0410    4.92301    1.66065  0.69575
   5   5.211 BB    0.0192    5.21390    4.21057  0.73686
   6   6.317 BB    0.0364    5.47464    2.24803  0.77371
   7   6.407 BB    0.0646   13.12827    2.66915  1.85536
   8   6.837 BB    0.0266    5.37545    3.10838  0.75969
   9   7.317 BB    0.0237    2.47547    1.59444  0.34985
  10   8.376 BB    0.0385    9.37196    3.58585  1.32450
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	9.065	BB	0.0251	102.58749	63.86723	14.49824
12	9.591	BB	0.0315	2.88795	1.43628	0.40814
13	10.597	BB	0.0253	4.37239	2.69470	0.61793
14	11.213	BB	0.0235	2.27982	1.48596	0.32220
15	11.439	BB	0.0241	3.49813	2.20174	0.49437
16	12.001	BB	0.0261	133.63589	79.15663	18.88618
17	12.737	BB	0.0278	2.81734	1.53799	0.39816
18	13.312	BB	0.0783	23.72131	3.68701	3.35243
19	15.127	BB	0.0219	2.09494	1.57988	0.29607
20	15.551	BB	0.0225	2.71780	1.87942	0.38409
21	16.421	BB	0.0191	1.91474	1.47863	0.27060
22	16.788	BB	0.0330	12.38568	5.42998	1.75041
23	17.052	BB	0.0207	3.96404	2.90013	0.56022
24	17.532	BB	0.0285	2.41259	1.31947	0.34096
Totals :				707.58562	313.66160	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/14/2014 9:58:51 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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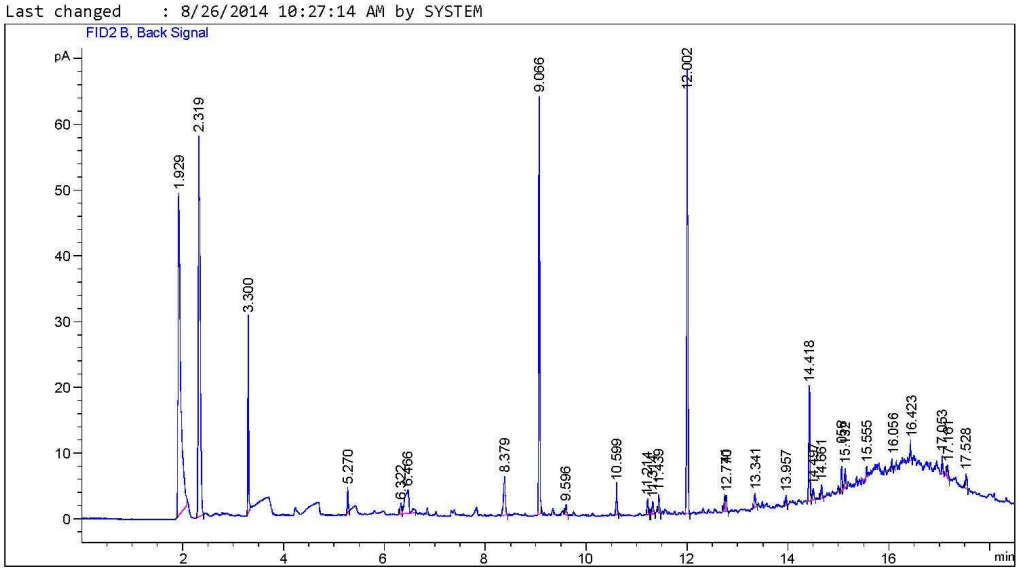


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   1.929 BB    0.0507  175.29761   48.84111 24.41719
   2   2.319 BB    0.0404  160.57156   57.89339 22.36600
   3   3.300 BB    0.0171   33.56493   29.95918  4.67526
   4   5.270 BB    0.0195    4.76688    3.78911  0.66398
   5   6.322 BV    0.0318    3.21503    1.57674  0.44782
   6   6.466 VB    0.0791   21.68123    3.51990  3.01998
   7   8.379 BB    0.0362   14.22355    5.87800  1.98120
   8   9.066 BB    0.0244   97.90993   63.57361 13.63787
   9   9.596 BB    0.0276    2.70537    1.49074  0.37683
  10  10.599 BB    0.0233    6.87974    4.54539  0.95828
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	11.214	BB	0.0247	3.67583	2.34879	0.51201
12	11.314	BB	0.0265	3.22440	1.80098	0.44913
13	11.439	BB	0.0238	3.99587	2.68096	0.55658
14	12.002	BB	0.0257	111.44655	67.24886	15.52338
15	12.741	BV	0.0230	4.06985	2.61059	0.56689
16	12.770	VB	0.0235	3.75555	2.44809	0.52311
17	13.341	BB	0.0280	4.10118	2.13830	0.57125
18	13.957	BB	0.0221	2.05422	1.45330	0.28613
19	14.418	BB	0.0232	27.43809	17.42595	3.82185
20	14.497	BB	0.0291	3.11522	1.65716	0.43392
21	14.661	BB	0.0234	2.98277	1.95365	0.41547
22	15.058	BV	0.0245	5.50793	3.71301	0.76720
23	15.132	VB	0.0227	4.85351	2.90808	0.67604
24	15.555	BB	0.0138	1.26149	1.63122	0.17571
25	16.056	BB	0.0197	2.11377	1.65412	0.29443
26	16.423	BB	0.0156	3.17076	2.79984	0.44165
27	17.053	BB	0.0203	3.44465	2.60027	0.47981
28	17.161	BB	0.0244	3.21772	1.77489	0.44820
29	17.528	BB	0.0220	3.68180	2.19277	0.51284
Totals :				717.92701	344.10801	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/14/2014 10:30:48 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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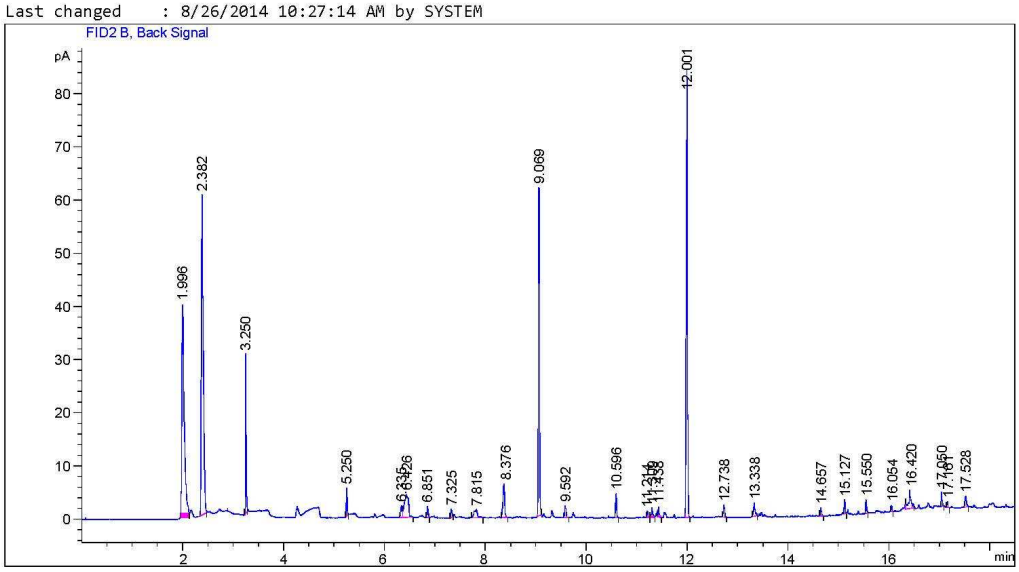


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   1.996 BB    0.0484  129.91026   39.83366 19.02114
   2   2.382 BB    0.0394  162.25148   60.22554 23.75646
   3   3.250 BB    0.0179   33.45779   29.80760  4.89881
   4   5.250 BB    0.0192    6.51272    5.27215  0.95358
   5   6.335 BV    0.0295    4.29870    2.17271  0.62940
   6   6.426 VB    0.0707   24.22807    4.29094  3.54741
   7   6.851 BB    0.0243    3.18308    2.07140  0.46606
   8   7.325 BB    0.0252    2.43295    1.45125  0.35623
   9   7.815 BB    0.0574    7.17917    1.52330  1.05116
  10   8.376 BB    0.0375   14.90336    6.22481  2.18211
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	9.069	BB	0.0237	95.70879	61.69785	14.01344
12	9.592	BB	0.0328	4.31609	2.16991	0.63195
13	10.596	BB	0.0236	6.90910	4.48518	1.01161
14	11.214	BB	0.0245	1.89809	1.22067	0.27791
15	11.309	BB	0.0273	2.87343	1.73861	0.42072
16	11.438	BB	0.0242	2.52175	1.72905	0.36923
17	12.001	BB	0.0260	140.43703	83.69965	20.56244
18	12.738	BB	0.0249	3.72473	2.24783	0.54537
19	13.338	BB	0.0328	5.41987	2.39079	0.79356
20	14.657	BB	0.0233	2.21563	1.45964	0.32441
21	15.127	BB	0.0216	3.52268	2.56547	0.51578
22	15.550	BB	0.0202	3.42746	2.59524	0.50184
23	16.054	BB	0.0202	1.49113	1.12743	0.21833
24	16.420	BB	0.0434	11.60184	3.43227	1.69871
25	17.050	BB	0.0190	3.42114	2.65579	0.50091
26	17.161	BB	0.0193	1.32634	1.07047	0.19420
27	17.528	BB	0.0289	3.80578	2.04687	0.55723
Totals :				682.97843	331.20610	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/15/2014 9:30:52 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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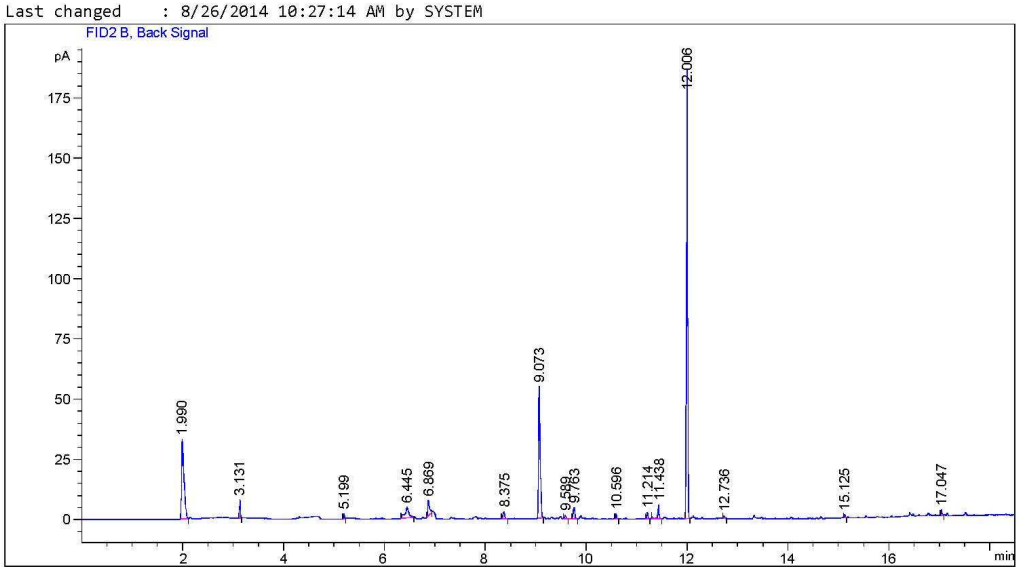


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   1.990 BB    0.0465  108.99707   32.99511 16.82846
   2   3.131 BB    0.0183    8.70213    7.53369  1.34355
   3   5.199 BB    0.0198    2.48698    1.93368  0.38397
   4   6.445 BB    0.0717   24.28575    4.33879  3.74957
   5   6.869 BB    0.0362   16.25850    6.35632  2.51021
   6   8.375 BB    0.0376    6.76949    2.82224  1.04517
   7   9.073 BB    0.0357   91.84613   54.52795 19.41482
   8   9.589 BB    0.0283    2.47487    1.36748  0.38210
   9   9.763 BB    0.0363   10.56210    4.62074  1.63072
  10  10.596 BB    0.0247    3.05272    1.94980  0.47132
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	11.214	BB	0.0260	4.46776	2.65606	0.68979
12	11.438	BB	0.0301	10.98621	5.59255	1.69620
13	12.006	BB	0.0272	126.64516	84.98192	48.86248
14	12.736	BB	0.0247	1.80312	1.10148	0.27839
15	15.125	BB	0.0221	1.69831	1.20198	0.26221
16	17.047	BB	0.0223	2.92129	2.04488	0.45103
Totals :				647.69480	316.02466	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/15/2014 10:40:59 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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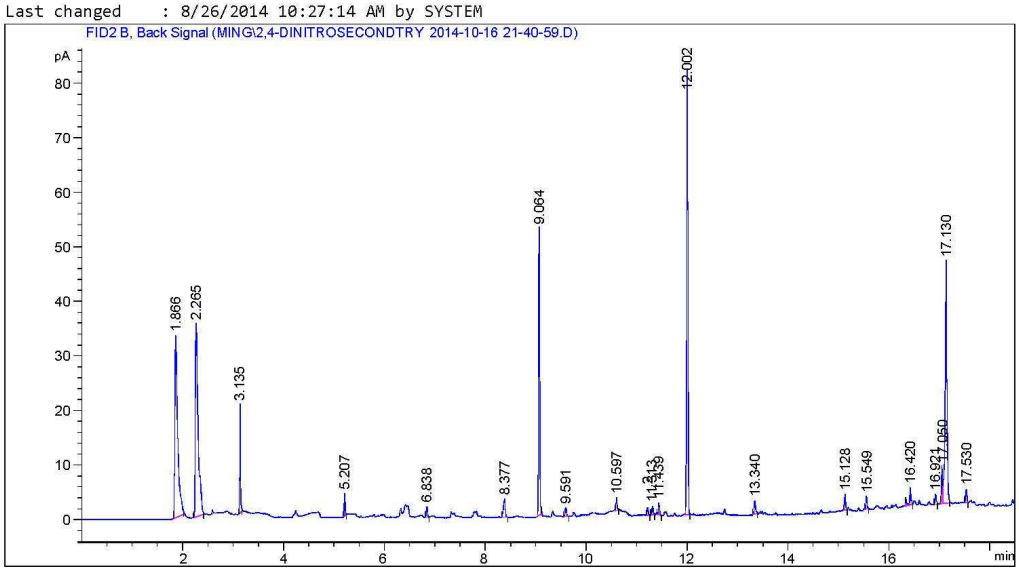


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   1.866 BB    0.0565  131.57460   33.28856 18.93510
   2   2.265 BB    0.0586  139.06401   35.39214 20.01291
   3   3.135 BB    0.0178   23.37821   19.81314  3.36439
   4   5.207 BB    0.0204    5.49243    4.10828  0.79042
   5   6.838 BB    0.0271    3.16302    1.85634  0.45519
   6   8.377 BB    0.0374    7.91763    3.23030  1.13944
   7   9.064 BB    0.0247   94.73284   52.50827 11.87490
   8   9.591 BB    0.0338    3.29905    1.58839  0.47477
   9  10.597 BB    0.0269    4.01619    2.28248  0.57798
  10  11.213 BB    0.0263    2.25804    1.37670  0.32496
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	11.313	BB	0.0257	2.70960	1.57595	0.38994
12	11.439	BB	0.0248	3.24358	2.14515	0.46679
13	12.002	BB	0.0271	138.31130	81.21359	19.95535
14	13.340	BB	0.0301	4.95266	2.43431	0.71275
15	15.128	BB	0.0230	4.00038	2.81962	0.57570
16	15.549	BB	0.0246	3.71371	2.28352	0.53445
17	16.420	BB	0.0283	6.20064	3.08283	0.89234
18	16.921	BB	0.0246	2.98775	1.76366	0.42997
19	17.050	BV	0.0205	9.53179	6.75390	1.37173
20	17.130	VB	0.0376	112.29736	44.33313	16.16088
21	17.530	BB	0.0236	3.89156	2.41904	0.56004
Totals :				694.87155	306.26930	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/16/2014 9:40:59 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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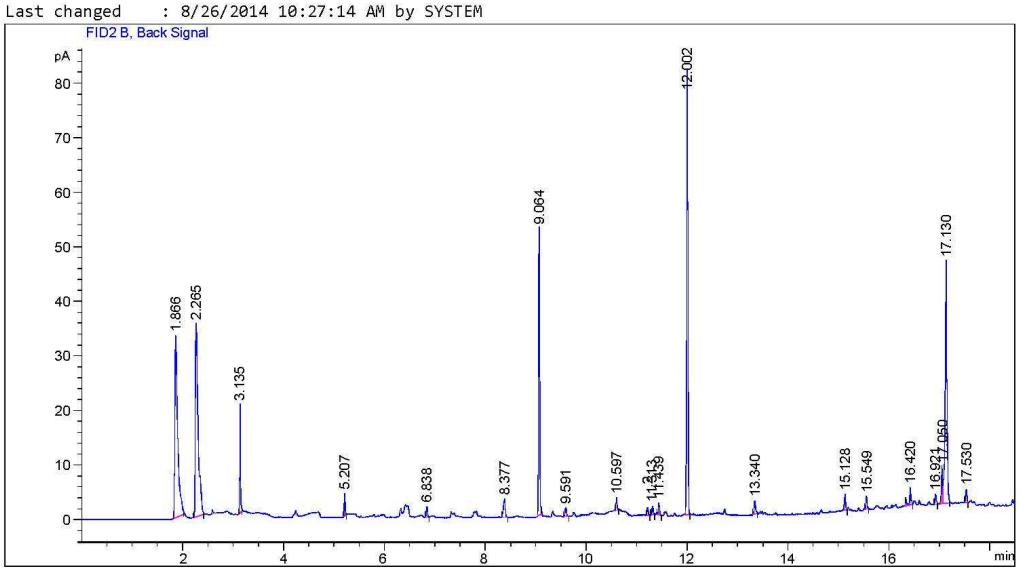


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak  RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   1.866 BB    0.0565  131.57460   33.28856 18.93510
   2   2.265 BB    0.0586  139.06401   35.39214 20.01291
   3   3.135 BB    0.0178   23.37821   19.81314  3.36439
   4   5.207 BB    0.0204    5.49243    4.10828  0.79042
   5   6.838 BB    0.0271    3.16302    1.85634  0.45519
   6   8.377 BB    0.0374    7.91763    3.23030  1.13944
   7   9.064 BB    0.0247   88.27743   52.50827 11.87490
   8   9.591 BB    0.0338    3.29905    1.58839  0.47477
   9  10.597 BB    0.0269    4.01619    2.28248  0.57798
  10  11.213 BB    0.0263    2.25804    1.37670  0.32496
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	11.313	BB	0.0257	2.70960	1.57595	0.38994
12	11.439	BB	0.0248	3.24358	2.14515	0.46679
13	12.002	BB	0.0271	138.66402	81.21359	19.95535
14	13.340	BB	0.0301	4.95266	2.43431	0.71275
15	15.128	BB	0.0230	4.00038	2.81962	0.57570
16	15.549	BB	0.0246	3.71371	2.28352	0.53445
17	16.420	BB	0.0283	6.20064	3.08283	0.89234
18	16.921	BB	0.0246	2.98775	1.76366	0.42997
19	17.050	BV	0.0205	9.53179	6.75390	1.37173
20	17.130	VB	0.0376	112.29736	44.33313	16.16088
21	17.530	BB	0.0236	3.89156	2.41904	0.56004
Totals :				694.87155	306.26930	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/16/2014 10:31:13 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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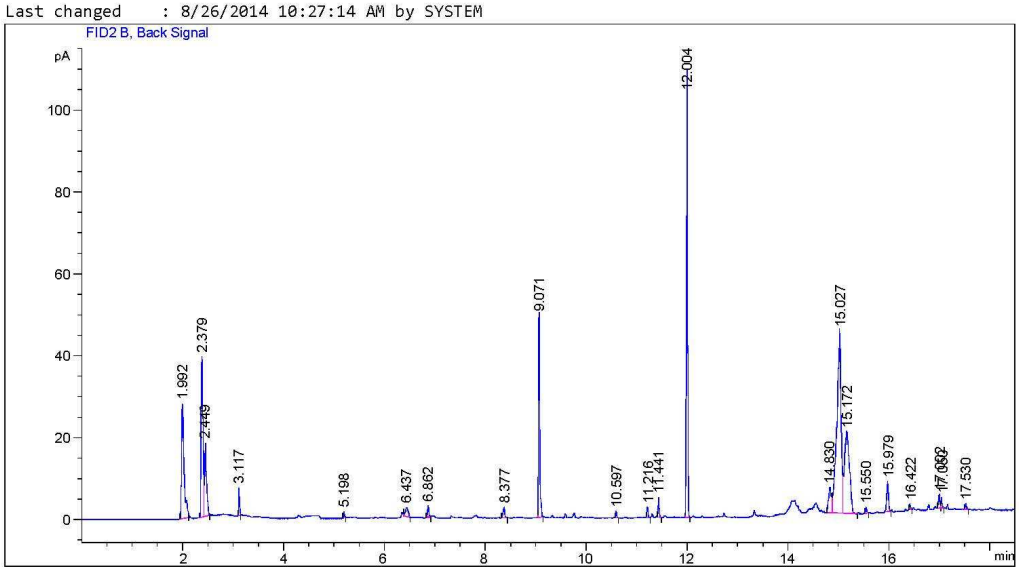


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   1.992 BB    0.0514   96.49081   27.97702  9.52931
   2   2.379 BV    0.0407  101.84367   39.21660 10.05795
   3   2.449 VB    0.0381   47.13363   17.80271  4.65486
   4   3.117 BB    0.0185    7.94811    6.76130  0.78494
   5   5.198 BB    0.0197    1.88865    1.48248  0.18652
   6   6.437 BB    0.0567    8.34805    2.14216  0.82444
   7   6.862 BB    0.0294    5.10303    2.78227  0.50397
   8   8.377 BB    0.0355    5.92430    2.51716  0.58508
   9   9.071 BB    0.0270   86.10667   49.26996  8.58896
  10  10.597 BB    0.0234    2.43221    1.59062  0.24020
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	11.216	BB	0.0265	4.05004	2.44870	0.39998
12	11.441	BB	0.0261	7.80357	4.82664	0.77067
13	12.004	BB	0.0261	141.58585	108.60617	18.10358
14	14.830	BV	0.0471	23.25440	6.18301	2.29657
15	15.027	VV	0.0710	258.12332	44.43538	25.49192
16	15.172	VB	0.0862	138.65268	19.86697	13.69316
17	15.550	BB	0.0246	2.20950	1.41645	0.21821
18	15.979	BB	0.0294	15.82285	7.28161	1.56264
19	16.422	BB	0.0203	1.84049	1.38656	0.18176
20	17.002	BV	0.0342	7.46078	3.32160	0.73682
21	17.050	VB	0.0211	3.60069	2.58424	0.35560
22	17.530	BB	0.0281	2.35788	1.31397	0.23286
Totals :				1012.56912	355.21357	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/17/2014 4:57:12 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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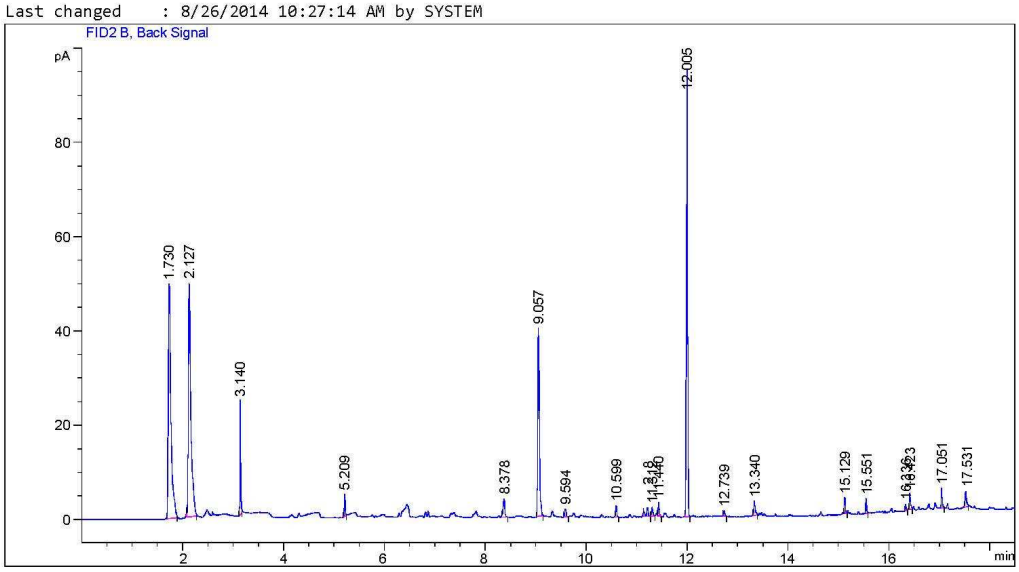


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   1.730 BB    0.0532  179.19995   49.81327 25.28675
   2   2.127 BB    0.0502  172.41650   49.56883 24.32954
   3   3.140 BB    0.0183   28.04461   24.16392  3.95735
   4   5.209 BB    0.0194    5.92093    4.71809  0.83550
   5   8.378 BB    0.0376    9.00611    3.74882  1.27084
   6   9.057 BB    0.0341   82.51533   39.87855 12.96033
   7   9.594 BB    0.0312    3.49552    1.64424  0.49325
   8  10.599 BB    0.0252    3.92619    2.44025  0.55402
   9  11.218 BV    0.0431    5.41394    1.79829  0.76396
  10  11.312 VB    0.0263    3.13074    1.83350  0.44178
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	11.440	BB	0.0256	4.65151	2.82532	0.65637
12	12.005	BB	0.0278	166.48907	94.38515	23.49313
13	12.739	BB	0.0254	1.91725	1.17480	0.27054
14	13.340	BB	0.0290	5.96929	2.97696	0.84232
15	15.129	BB	0.0213	4.36305	3.24344	0.61567
16	15.551	BB	0.0217	4.26304	3.09211	0.60155
17	16.336	BB	0.0208	1.70234	1.24007	0.24022
18	16.423	BB	0.0224	5.14452	3.41645	0.72594
19	17.051	BB	0.0215	5.69554	4.17912	0.80369
20	17.531	BB	0.0277	6.07515	3.20383	0.85726
Totals :				708.67138	299.34504	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 9/17/2014 5:56:27 PM
                                                  Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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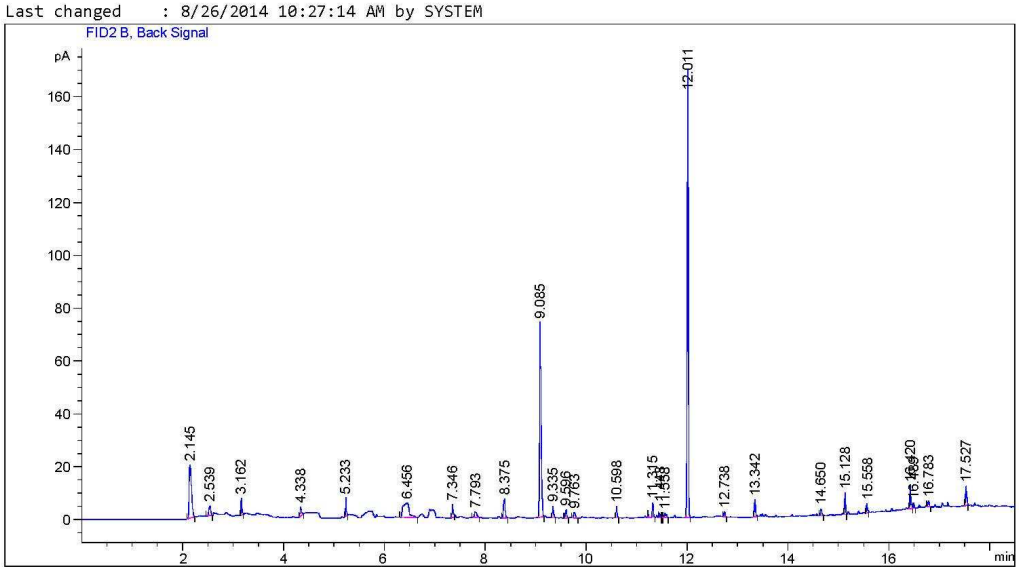


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   2.145 BB    0.0514   64.97404   20.05876  9.05954
   2   2.539 BV    0.0462   10.35441    3.70640  1.44375
   3   3.162 BB    0.0179    7.56556    6.35851  1.05489
   4   4.338 BB    0.0291    6.36027    3.26795  0.88683
   5   5.233 BB    0.0198    8.81958    6.84276  1.22974
   6   6.456 BB    0.1080   45.30408    5.32912  6.31690
   7   7.346 BB    0.0267    8.79564    4.87480  1.22641
   8   7.793 BB    0.0507    7.68926    2.06119  1.07214
   9   8.375 BB    0.0377   16.72225    7.13451  2.33164
  10   9.085 BB    0.0309   86.96916   73.47183 21.53823
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	9.335	BB	0.0276	7.54804	4.31367	1.05245
12	9.596	BB	0.0294	5.52403	3.01791	0.77023
13	9.763	BB	0.0396	5.14692	2.05612	0.71765
14	10.598	BB	0.0245	6.61815	4.27565	0.92279
15	11.315	BB	0.0257	8.86313	5.35372	1.23582
16	11.448	BB	0.0227	2.06144	1.40834	0.28743
17	11.558	BB	0.0363	4.08818	1.59717	0.57003
18	12.011	BB	0.0260	166.81346	87.91168	39.33612
19	12.738	BB	0.0260	3.02729	1.80322	0.42210
20	13.342	BB	0.0260	11.09564	6.33451	1.54710
21	14.650	BB	0.0290	4.67588	2.50559	0.65197
22	15.128	BB	0.0198	10.21627	7.93520	1.42449
23	15.558	BB	0.0229	5.40791	3.48198	0.75404
24	16.420	BB	0.0200	11.54480	8.44347	1.60973
25	16.489	BB	0.0215	2.83566	1.89274	0.39539
26	16.783	BB	0.0256	3.58189	2.01180	0.49944
27	17.527	BB	0.0247	11.78453	7.20252	1.64315
Totals :				717.18900	364.65112	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/20/2014 11:04:56 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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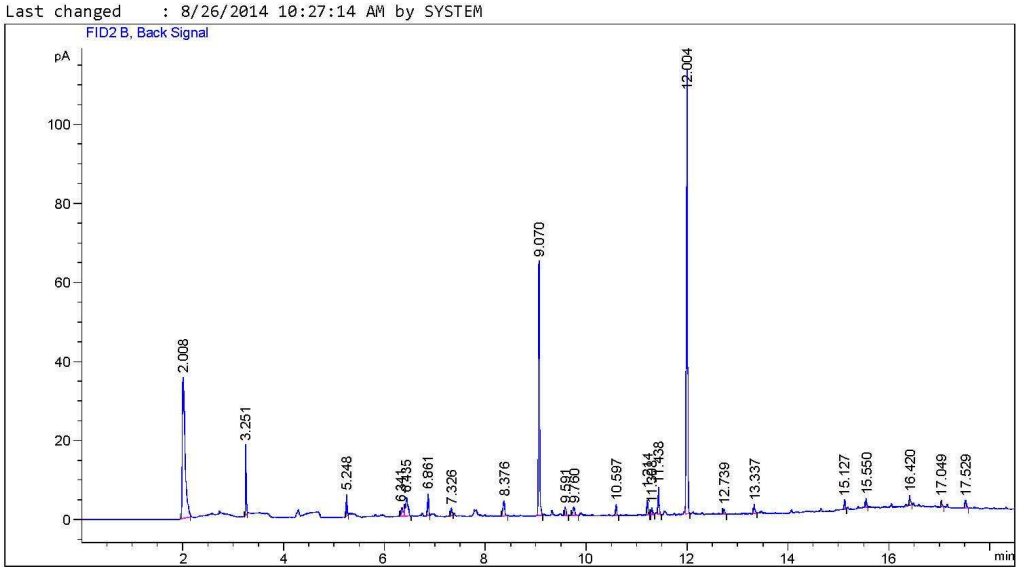


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	2.008	BB	0.0567	134.10135	35.67035	24.24124
2	3.251	BB	0.0179	19.98338	17.77443	3.61236
3	5.248	BB	0.0193	6.63224	5.33564	1.19890
4	6.341	BB	0.0441	6.93547	2.19227	1.25371
5	6.435	BB	0.0465	15.66557	4.56075	2.83183
6	6.861	BB	0.0244	8.14021	5.28685	1.47149
7	7.326	BB	0.0268	3.42179	1.88322	0.61855
8	8.376	BB	0.0391	9.85734	3.79878	1.78189
9	9.070	BB	0.0244	104.06435	64.46195	18.81151
10	9.591	BB	0.0309	3.98781	2.03415	0.72087

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	9.760	BB	0.0423	5.26591	2.08473	0.95191
12	10.597	BB	0.0236	4.11362	2.66401	0.74361
13	11.214	BB	0.0251	5.98155	3.73667	1.08127
14	11.308	BB	0.0252	2.91360	1.73498	0.52669
15	11.438	BB	0.0253	10.36209	6.66871	1.87313
16	12.004	BB	0.0271	189.42577	110.83434	34.24212
17	12.739	BB	0.0252	2.12549	1.31957	0.38422
18	13.337	BB	0.0300	4.31003	2.20315	0.77912
19	15.127	BB	0.0217	3.27060	2.25568	0.59122
20	15.550	BB	0.0223	3.07670	2.15572	0.55617
21	16.420	BB	0.0207	3.41827	2.39279	0.61791
22	17.049	BB	0.0199	2.22434	1.63172	0.40209
23	17.529	BB	0.0297	3.91772	1.95900	0.70820
Totals :				553.19518	284.63945	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/20/2014 11:50:51 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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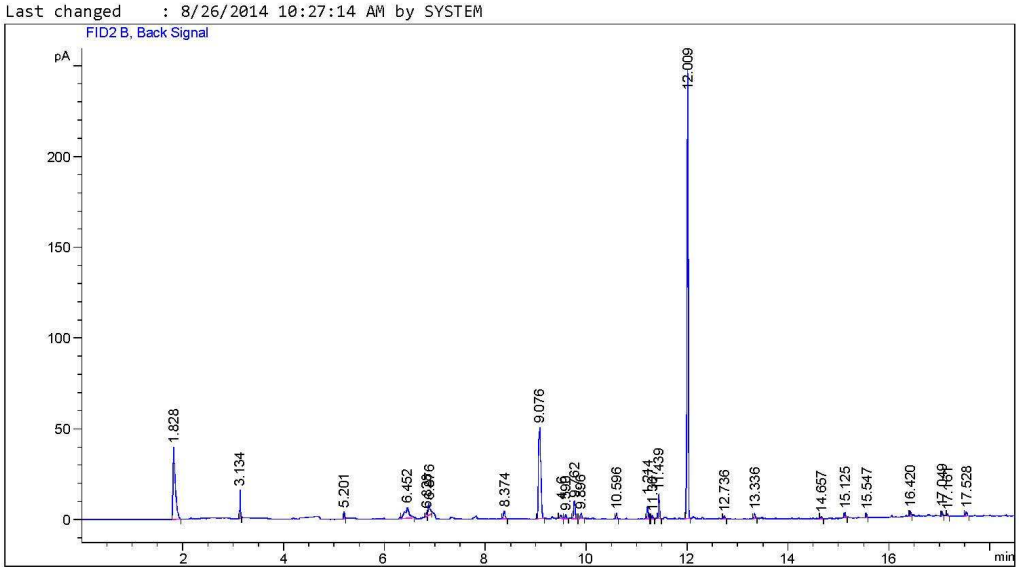


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   1.828 BB    0.0469  131.91736   39.59428 14.45753
   2   3.134 BB    0.0171   17.29792   15.36232  1.89577
   3   5.201 BB    0.0198    5.03567    3.92857  0.55189
   4   6.452 BB    0.0801   35.89951    5.74214  3.93442
   5   6.828 BV    0.0251    3.55618    2.21368  0.38974
   6   6.876 VB    0.0375   17.82897    6.52289  1.95397
   7   8.374 BB    0.0363    9.72227    4.24785  1.06552
   8   9.076 BB    0.0515  125.74884   50.00979 17.82320
   9   9.496 BV    0.0381    4.34109    1.83010  0.47576
  10   9.590 VB    0.0294    3.70948    2.01808  0.40654
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	9.762	BB	0.0356	20.37324	9.42370	2.23281
12	9.896	BB	0.0368	6.40679	2.74696	0.70215
13	10.596	BB	0.0237	4.48120	2.89609	0.49112
14	11.214	BB	0.0263	11.38355	6.68268	1.24758
15	11.307	BB	0.0240	2.94295	1.86167	0.32253
16	11.439	BB	0.0253	20.73900	13.33534	2.27290
17	12.009	BB	0.0275	179.39140	104.08437	46.47412
18	12.736	BB	0.0238	2.45890	1.51113	0.26948
19	13.336	BB	0.0291	5.36403	2.66768	0.58787
20	14.657	BB	0.0241	1.59056	1.00515	0.17432
21	15.125	BB	0.0226	3.75350	2.70463	0.41137
22	15.547	BB	0.0200	2.76358	2.11996	0.30288
23	16.420	BB	0.0248	4.88572	2.84314	0.53545
24	17.049	BB	0.0212	3.79226	2.69706	0.41561
25	17.161	BB	0.0192	1.28581	1.04283	0.14092
26	17.528	BB	0.0298	4.23855	2.26947	0.46453
Totals :				912.44741	431.36156	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/21/2014 8:46:21 PM
                                                  Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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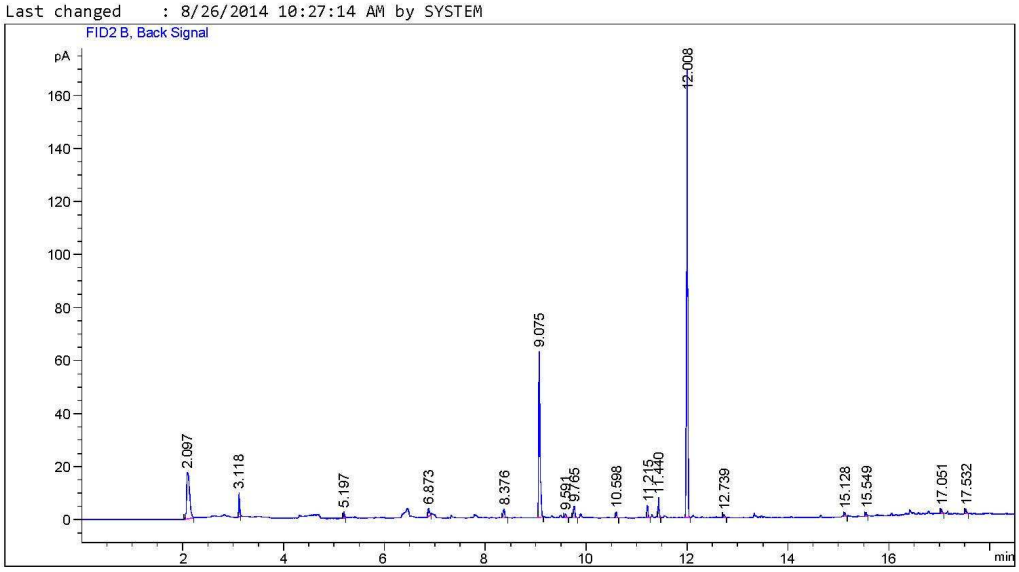


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   2.097 BB    0.0675   73.17124   17.40326 13.30068
   2   3.118 BB    0.0187   10.88050    9.12433  1.97780
   3   5.197 BB    0.0201    2.91229    2.22660  0.52938
   4   6.873 BB    0.0307    6.38587    2.96688  1.16079
   5   8.376 BB    0.0355    7.81971    3.21932  1.42143
   6   9.075 BB    0.0288  120.13661   62.56175 21.83780
   7   9.591 BB    0.0292    2.60918    1.43327  0.47428
   8   9.765 BB    0.0356    9.50515    4.27087  1.72780
   9  10.598 BB    0.0239    3.26254    2.17713  0.59305
  10  11.215 BB    0.0256    6.90413    4.36863  1.25500
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	11.440	BB	0.0247	11.33330	7.22673	2.06011
12	12.008	BB	0.0262	197.67497	147.87675	51.66346
13	12.739	BB	0.0245	1.72005	1.11001	0.31266
14	15.128	BB	0.0236	1.92768	1.30689	0.35040
15	15.549	BB	0.0230	2.17083	1.45371	0.39460
16	17.051	BB	0.0232	2.33802	1.55256	0.42499
17	17.532	BB	0.0259	2.83744	1.56661	0.51577
Totals :				550.13153	291.84531	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/21/2014 9:30:56 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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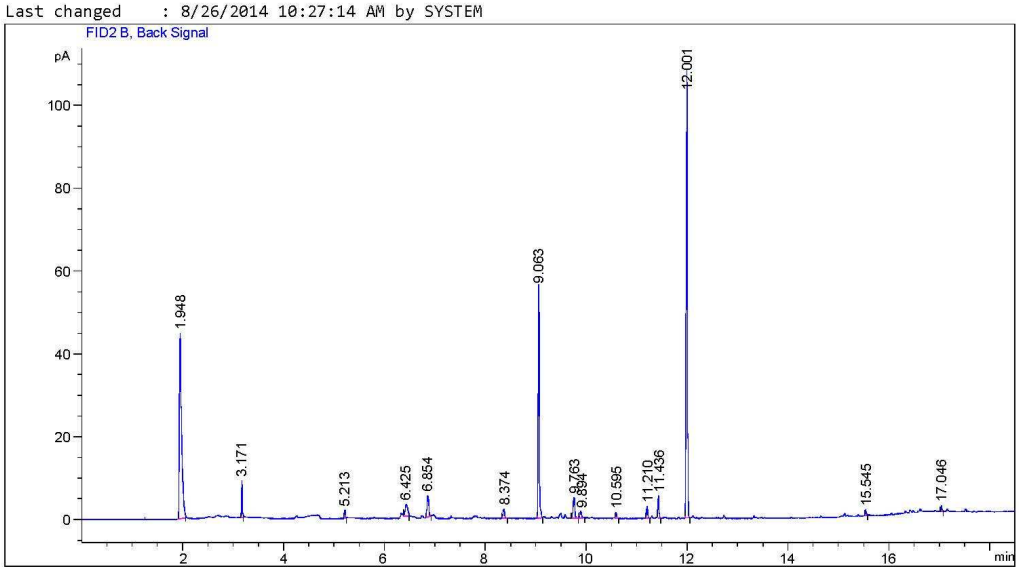


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   1.948 BB    0.0439  134.38713   44.68027 27.96118
   2   3.171 BB    0.0180   10.01532    8.84671  2.08383
   3   5.213 BB    0.0190    2.27423    1.86293  0.47319
   4   6.425 BB    0.0503   10.45949    2.77988  2.17625
   5   6.854 BB    0.0395   12.40485    5.12158  2.58101
   6   8.374 BB    0.0355    5.01677    2.12453  1.04381
   7   9.063 BB    0.0255   91.41241   55.73435 19.01967
   8   9.763 BB    0.0394   11.49781    4.88967  2.39229
   9   9.894 BB    0.0391    3.76772    1.53066  0.78393
  10  10.595 BB    0.0265    2.42165    1.46132  0.50386
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	11.210	BB	0.0268	4.83404	2.87443	1.00579
12	11.436	BB	0.0261	8.63693	5.33855	1.79704
13	12.001	BB	0.0268	179.88387	107.26411	37.42743
14	15.545	BB	0.0208	1.74622	1.27254	0.36333
15	17.046	BB	0.0211	1.86192	1.33467	0.38740
Totals :				480.62035	247.11621	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/22/2014 8:45:20 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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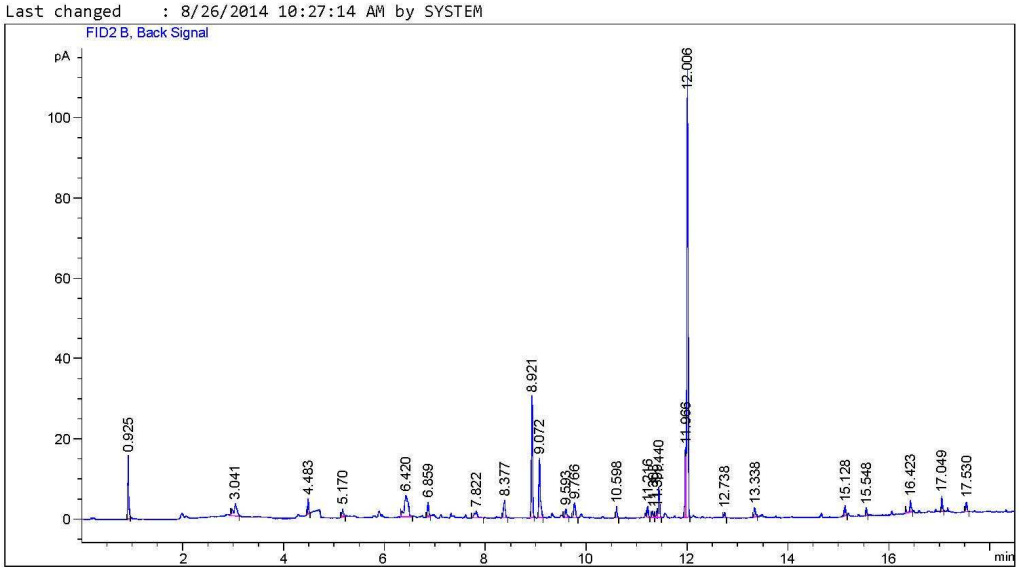


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	0.925	BB	0.0231	23.59494	15.70680	5.14643
2	3.041	BB	0.0618	13.63714	3.00184	2.97448
3	4.483	BB	0.0226	5.36647	3.69236	1.17051
4	5.170	BB	0.0300	4.20780	2.01223	0.91779
5	6.420	BB	0.0745	28.27381	5.27511	6.16697
6	6.859	BB	0.0287	6.87046	3.59869	1.49856
7	7.822	BB	0.0620	6.98170	1.55555	1.52282
8	8.377	BB	0.0366	10.28804	4.31045	2.24399
9	8.921	BB	0.0243	48.45550	30.17313	10.56891
10	9.072	BB	0.0296	29.26741	14.71985	6.38369

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	9.593	BB	0.0349	4.31708	1.99079	0.94162
12	9.766	BB	0.0374	8.93708	3.54434	1.94932
13	10.598	BB	0.0253	4.43318	2.73326	0.96695
14	11.216	BB	0.0260	4.58980	2.72512	1.00111
15	11.308	BB	0.0271	2.68537	1.51596	0.58572
16	11.399	BV	0.0255	2.77957	1.63356	0.60627
17	11.440	VB	0.0267	12.73279	7.62228	2.77722
18	11.966	BV	0.0199	23.52710	17.23587	5.13163
19	12.006	VB	0.0265	189.50531	109.98882	41.33412
20	12.738	BB	0.0238	2.12664	1.36529	0.46385
21	13.338	BB	0.0316	5.11355	2.36863	1.11535
22	15.128	BB	0.0226	3.50435	2.40938	0.76435
23	15.548	BB	0.0212	2.42481	1.81230	0.52889
24	16.423	BB	0.0273	5.44765	2.93550	1.18822
25	17.049	BB	0.0201	4.95974	3.77742	1.08180
26	17.530	BB	0.0291	4.44461	2.45406	0.96944
Totals :				458.47191	250.15860	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/22/2014 9:30:52 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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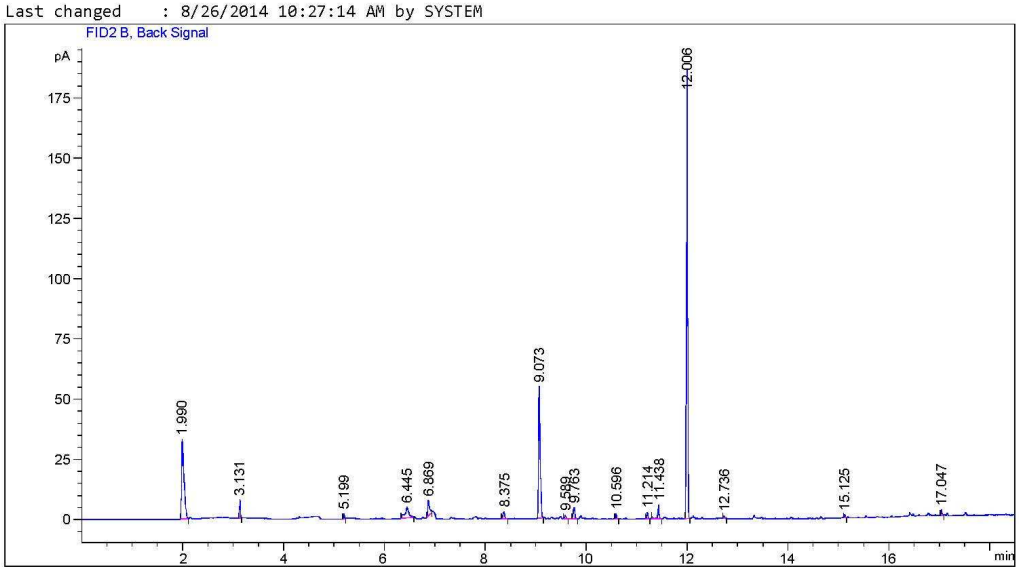


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	1.990	BB	0.0465	108.99707	32.99511	16.82846
2	3.131	BB	0.0183	8.70213	7.53369	1.34355
3	5.199	BB	0.0198	2.48698	1.93368	0.38397
4	6.445	BB	0.0717	24.28575	4.33879	3.74957
5	6.869	BB	0.0362	16.25850	6.35632	2.51021
6	8.375	BB	0.0376	6.76949	2.82224	1.04517
7	9.073	BB	0.0357	122.62742	54.52795	19.41482
8	9.589	BB	0.0283	2.47487	1.36748	0.38210
9	9.763	BB	0.0363	10.56210	4.62074	1.63072
10	10.596	BB	0.0247	3.05272	1.94980	0.47132

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	11.214	BB	0.0260	4.46776	2.65606	0.68979
12	11.438	BB	0.0301	10.98621	5.59255	1.69620
13	12.006	BB	0.0272	185.95951	84.98192	48.86248
14	12.736	BB	0.0247	1.80312	1.10148	0.27839
15	15.125	BB	0.0221	1.69831	1.20198	0.26221
16	17.047	BB	0.0223	2.92129	2.04488	0.45103
Totals :				647.69480	316.02466	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/23/2014 6:37:03 PM
                                                  Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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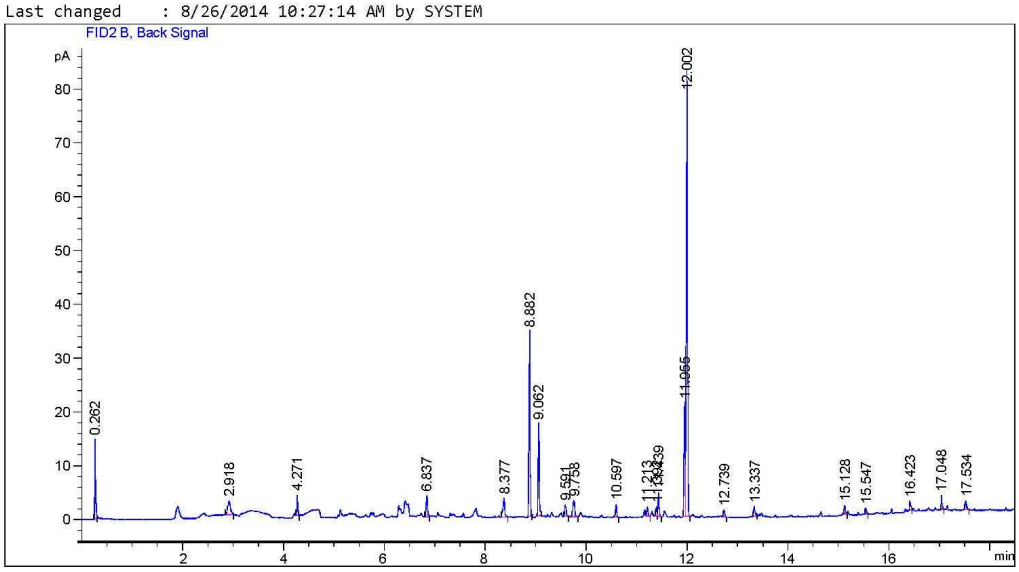


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	0.262	BB	0.0233	21.24831	14.64853	5.90299
2	2.918	BB	0.0529	9.39039	2.48168	2.60874
3	4.271	BB	0.0223	5.25877	3.68165	1.46094
4	6.837	BB	0.0305	7.63764	3.81899	2.12181
5	8.377	BB	0.0359	8.49329	3.36255	2.35952
6	8.882	BB	0.0251	54.92808	34.27856	15.25955
7	9.062	BB	0.0256	27.87810	16.96716	7.74481
8	9.591	BB	0.0317	4.23606	2.08165	1.17682
9	9.758	BB	0.0423	7.57451	2.91749	2.10427
10	10.597	BB	0.0252	3.79977	2.35787	1.05561

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	11.213	BB	0.0270	2.66958	1.63826	0.74164
12	11.392	BV	0.0224	2.33405	1.62002	0.64842
13	11.439	VB	0.0264	7.21614	4.37664	2.00471
14	11.955	BV	0.0250	33.34168	20.96723	9.26264
15	12.002	VB	0.0269	144.78091	82.35538	40.22154
16	12.739	BB	0.0244	1.89187	1.22402	0.52558
17	13.337	BB	0.0331	3.77364	1.70216	1.04835
18	15.128	BB	0.0230	2.30631	1.54499	0.64072
19	15.547	BB	0.0224	1.66397	1.15292	0.46227
20	16.423	BB	0.0235	2.79905	1.67466	0.77760
21	17.048	BB	0.0211	3.55285	2.53795	0.98702
22	17.534	BB	0.0298	3.18369	1.64512	0.88446
Totals :				359.95866	209.03547	

==========

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/23/2014 7:21:06 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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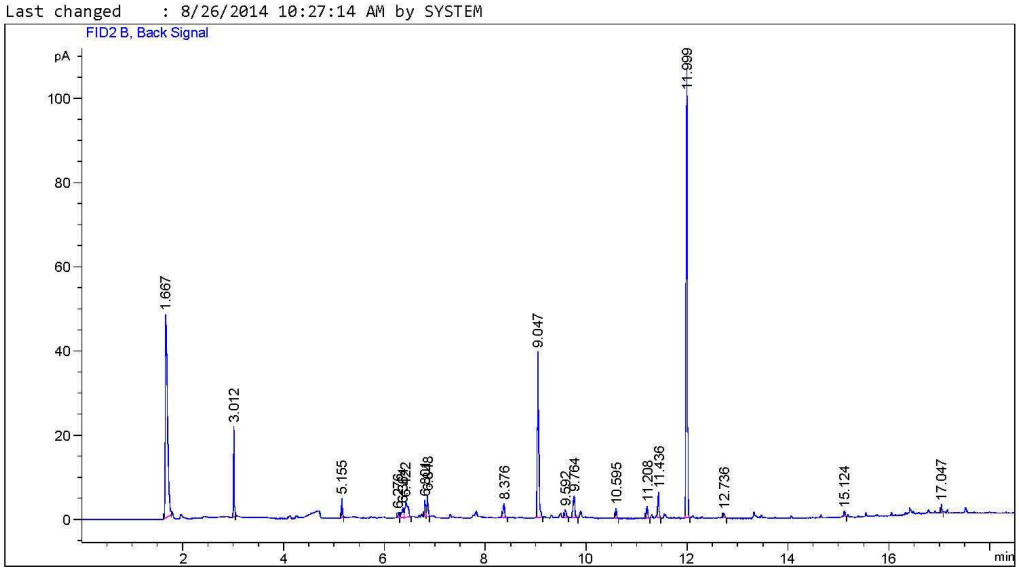


```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   1.667 BB    0.0483  150.35561   48.28679 28.19565
   2   3.012 BB    0.0176   24.89862   21.42891  4.66915
   3   5.155 BB    0.0192    5.54099    4.47690  1.03908
   4   6.276 BV    0.0260    1.88706    1.17398  0.35387
   5   6.364 VV    0.0381    6.36956    2.28618  1.19446
   6   6.422 VB    0.0535   14.85601    3.74560  2.78590
   7   6.801 BV    0.0231    5.67849    3.78469  1.06487
   8   6.848 VB    0.0272    8.97195    5.02230  1.68248
   9   8.376 BB    0.0350    7.25949    3.14183  1.36135
  10   9.047 BB    0.0301   79.78844   39.33666 14.96244
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	9.592	BB	0.0354	4.15442	1.76642	0.77906
12	9.764	BB	0.0396	12.62330	5.04690	2.36720
13	10.595	BB	0.0245	3.65101	2.25575	0.68466
14	11.208	BB	0.0284	5.09943	2.81119	0.95628
15	11.436	BB	0.0262	10.22039	6.01533	1.91659
16	11.999	BB	0.0277	164.13663	105.97335	34.87233
17	12.736	BB	0.0256	1.87921	1.14259	0.35240
18	15.124	BB	0.0226	1.61731	1.11065	0.30329
19	17.047	BB	0.0204	2.44727	1.82802	0.45893
Totals :				533.25810	260.63403	

*** End of Report ***

```
======================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/24/2014 9:38:02 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
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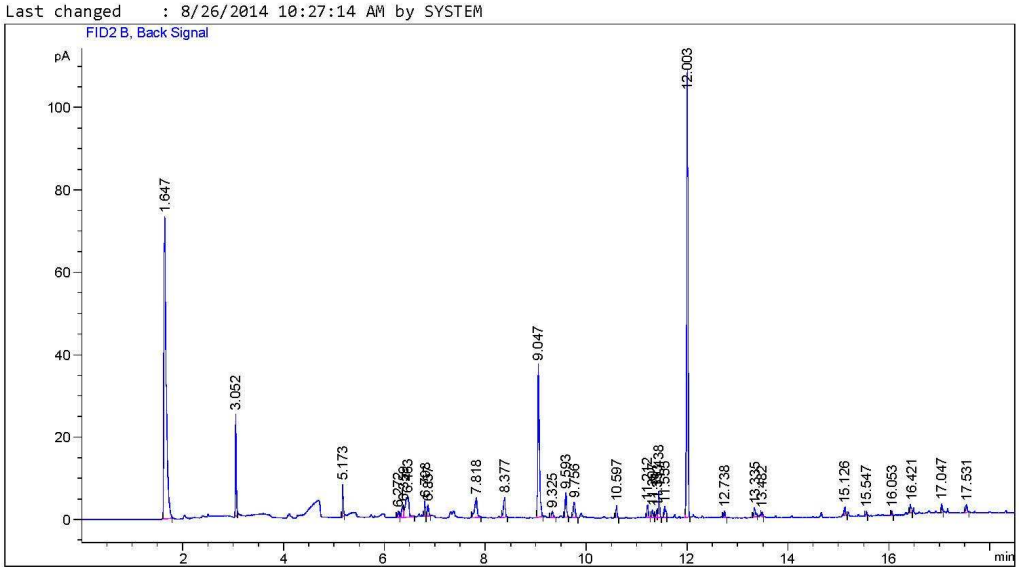



```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

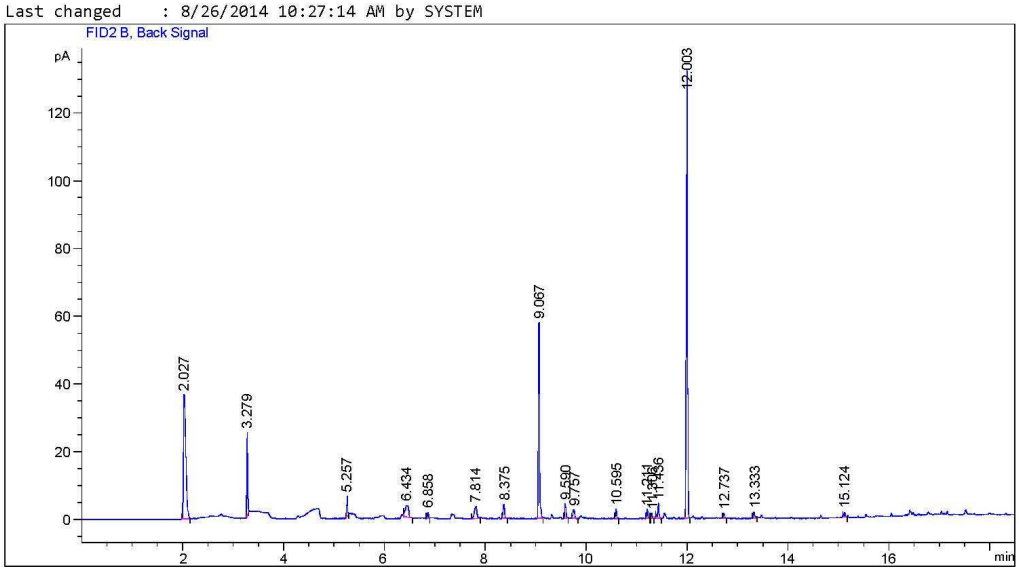

Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   1.647 BB    0.0472  241.36484   73.30944 33.72820
   2   3.052 BB    0.0184   28.75542   24.67099  4.01827
   3   5.173 BB    0.0198    9.91539    7.71529  1.38557
   4   6.272 BV    0.0277    2.69584    1.47331  0.37671
   5   6.359 VV    0.0354    6.99372    2.81170  0.97730
   6   6.463 VB    0.0720   25.96150    4.79336  3.62785
   7   6.798 BV    0.0249    5.45891    3.59549  0.76283
   8   6.857 VB    0.0290    5.01913    2.68540  0.70137
   9   7.818 BB    0.0510   17.14932    4.65790  2.39644
  10   8.377 BB    0.0347   11.30552    4.78814  1.57983
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	9.047	BB	0.0311	80.74574	36.98539	11.28337
12	9.325	BB	0.0374	3.28466	1.41733	0.45900
13	9.593	BB	0.0312	11.72693	5.89464	1.63871
14	9.756	BB	0.0419	9.54665	3.72122	1.33404
15	10.597	BB	0.0256	5.00985	3.04213	0.70007
16	11.212	BB	0.0304	5.56226	3.00513	0.77727
17	11.307	BB	0.0284	3.35879	1.78244	0.46936
18	11.393	BV	0.0239	2.68767	1.70972	0.37557
19	11.438	VB	0.0278	11.20564	6.33587	1.56587
20	11.555	BB	0.0329	5.92567	2.53544	0.82805
21	12.003	BB	0.0285	196.81346	107.86467	27.50261
22	12.738	BB	0.0252	2.33593	1.51632	0.32642
23	13.335	BB	0.0357	5.33021	2.24367	0.74484
24	13.482	BB	0.0204	1.46463	1.09791	0.20467
25	15.126	BB	0.0226	2.63756	1.90037	0.36857
26	15.547	BB	0.0213	1.59104	1.12533	0.22233
27	16.053	BB	0.0206	1.34751	1.04785	0.18830
28	16.421	BB	0.0259	3.76852	2.00493	0.52661
29	17.047	BB	0.0204	2.76636	2.06428	0.38657
30	17.531	BB	0.0327	3.88872	1.83660	0.54341
Totals :				715.61741	319.63227	

*** End of Report ***

```
=====================================================================
Acq. Operator   : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : Agilent 7890B                    Location : Vial 2
Injection Date  : 10/24/2014 10:25:45 PM
                                                   Inj Volume : Manually
Method          : C:\CHEM32\1\METHODS\WILBER.M
Last changed    : 8/26/2014 10:27:14 AM by SYSTEM
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID2 B, Back Signal

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   2.027 BB    0.0526  119.56099   36.55827 21.38602
   2   3.279 BB    0.0170   27.48949   24.62997  4.91708
   3   5.257 BB    0.0199    7.85944    6.06880  1.40583
   4   6.434 BB    0.0626   16.37250    3.39936  2.92857
   5   6.858 BB    0.0248    2.67637    1.69752  0.47873
   6   7.814 BB    0.0527   12.74157    3.51146  2.27910
   7   8.375 BB    0.0338    9.29360    4.08193  1.66236
   8   9.067 BB    0.0257   95.54005   57.71143 17.08936
   9   9.590 BB    0.0289    7.90138    4.24083  1.41333
  10   9.757 BB    0.0394    6.05824    2.50412  1.08364
```

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
11	10.595	BB	0.0259	4.32323	2.70377	0.77330
12	11.211	BB	0.0256	4.30843	2.62019	0.77065
13	11.306	BB	0.0246	2.25025	1.38534	0.40250
14	11.436	BB	0.0293	8.55510	4.36535	1.53026
15	12.003	BB	0.0283	226.44614	130.41386	40.50469
16	12.737	BB	0.0249	2.27479	1.43323	0.40689
17	13.333	BB	0.0309	3.57654	1.76329	0.63974
18	15.124	BB	0.0223	1.83338	1.34191	0.32794
Totals :				559.06149	290.43064	

*** End of Report ***

VITA

Xuewen Wang

Candidate for the Degree of

Master of Science

Thesis: ANALYSIS OF TASTE-AND-ODOR COMPOUNDS USING GAS CHROMATOGRAPHY AND FLAME IONIZATION DETECTION

Major Field: Environmental Engineering

Biographical:

Education:

Completed the requirements for the Master of Science in environmental engineering at Oklahoma State University, Stillwater, Oklahoma in December, 2014.

Completed the requirements for the Bachelor of Science in material science and engineering at Dalian Polytechnic University, Dalian, China in 2012.