GC-MS Analysis of Acetone, Methyl Isobutyl Ketone, and Methyl Ethyl Ketone in Ambient Air Samples from Shelby County

by Verina Rezk

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Thesis Committee:

Dr. Ngee Sing Chong, Thesis Director

Dr. Kevin L Bicker, Second Reader

Dr. Katy Hosbein, Third Reader

Dr. Andrienne C. Friedli, Thesis Committee Chair

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APPROVED:

Dr. Ngee Sing Chong, Thesis Director Professor, Department of Chemistry

Dr. Andrienne C. Friedli, Thesis Committee Chair Professor, Interim Chair, Department of Chemistry

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Abstract:

There is a requirement for industries to report the quantity of toxic compounds emitted from each factory released under the Toxic Substances Control Act. Specifically, industrial manufacturing facilities are required under the EPA to submit data on gases leaked and released annually. Some of these factories are very close to where residents live, threatening their health. Also, the gases leaked are not limited to the county released but affect surrounding counties. This research analyzes samples from Shelby County, Tennessee, and focuses on acetone and methyl isobutyl ketone. This research used Gas Chromatography interfaced to mass spectrometry (GC-MS) to quantify the concentrations of volatile organic compounds in ambient air using the extracted ion chromatography signals. We measured acetone at a higher concentration than MIBK and MEK for air samples analyzed in Shelby County or the metropolitan areas of Memphis, Tennessee. Overall, the concentrations of the ketones were higher in the summer than in the winter.

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Introduction:

Monitoring the emissions of toxins in the air is essential for protecting human health. It could help us understand the risks of the toxins before they become a threat to public health. Those harmful chemicals released in the air are volatile organic compounds (VOC). VOCs are compounds emitted into the air as gases. Some compounds are toxic alone, and others become harmful after reacting with other atmospheric molecules. These compounds are regulated by the United States Environmental Protection Agency (USEPA) and Occupational Safety and Health Administration (OSHA).¹

Factories and companies have been required to report the amount of gasses produced and leaked every year to the USEPA since 1998.² However, collecting the data about chemicals emitted is not enough. Analyzing the air samples at different locations is needed to understand the components that make up the ambient air. Sample analysis allows for better preparation and helps workers and residents from getting sick from these toxins. Preventive measures are better than treating problems after damage, especially since some VOC can cause cancer in later stages.³

The two types of sampling for analyzing air toxicants are on-site and off-site. The on-site is for analyzing the air samples within the factories to estimate the extent of inhalation exposure to VOCs for the workers. For off-site or ambient air samples collected outside industrial facilities, these concentrations of the air toxicants in the area around these factories, thereby affecting nearby residents and the populations of the surrounding counties. The methods for measuring VOCs are gas chromatography with mass spectrometry (GC-MS) which requires a significant amount of time to analyze for chromatographic separation.⁴ The data collected in this research originates from Shelby County, Tennessee. These samples are analyzed by GC-MS and then compared to the Toxics Release Inventory data from USEPA in Shelby County for 2014.

The goal is to focus on ketone's effects when they are present in high concentrations. Ketones are organic compounds that are composed of carbonyl functional groups with carbon-based groups on both sides. Ketones are good solvents and are low-cost factors used as chemical intermediates and solvents for vinyl polymers, resins, cotton, dyes, and pigments.⁵ Factories rely heavily on ketones during their manufacturing and production, which causes an increase in the concentration of ketones in the areas around factories. The primary toxicity of ketone is a central nervous system depressant, irritating the eyes, skin, and mucous membranes. Ketones are absorbed by inhalation, ingestion, and dermal exposure, and absorption is usually rapid.⁵

For example, short-term exposure to MEK can irritate the skin and cause a rash or burning feeling on contact, which could lead to permanent damage. Inhaling MEK can irritate the throat and the nose and cause coughing and wheezing. These symptoms could occur immediately or shortly after the exposure.⁶ The goal is to control and limit how much the residents are exposed to ketones. Not only are the workers affected by the inhalation of ketones, but also residents of these areas are sometimes exposed to ketones leaked into the air.

According to the USEPA, acetone is non-carcinogenic and does not lead to death. Side effects of high concentrations of acetone occur from constant exposure. Some workers exposed to it experienced mood disorders, irritability, memory difficulties, sleep disturbances, headache, numb hands or feet, eye, and nose irritation, bone, joint, or muscle pain, nausea, and abdominal pain.⁷

The increase in the release of MIBK has many effects, especially for on-site releases. MIK short-term effects include eye and mucus membrane irritation, weakness, nausea, headache, dizziness, vomiting, and incoordination narcosis in humans. However, exposure to it often could cause long-term effects, including nausea, headache, burning of the eyes, insomnia, weakness, intestinal pain, and slight liver enlargement. As a result, "EPA has classified it as Group D [carcinogen]" .⁸ This means that the EPA classified it as a VOC that does not cause cancer; however, it has the effects mentioned above.

This research focuses on the ketone VOC effects on residents in Shelby County, TN. Ketone compounds include methyl isobutyl ketone (MIK), methyl ethyl ketone (MEK), and acetone. We will analyze ketones from 50 locations in residential areas and public buildings. The goal is to calculate and understand how much the residents are affected by all the factories surrounding them. We hypothesis that the concentration of the ketones will be higher in the summer due to the increase in the temperature. Also, we are attempting to test two techniques for analysis and try to determine which one is more accurate to use for the sample analysis.

Methods

I. Sample collection:

The on-site samples were previously collected from different locations and at various times. The data gathered for this research is from Shelby County, Tennessee. We analyzed the data through the Target View software on the GC-MS and examined each compound and its peaks. Those compounds are volatile organic compounds and have high release rates based on Toxic Release Inventory (TRI Explorer) data from the United States Environmental Protection Agency (USEPA).

II. Analysis of the Standards:

We started with the analysis of the standards prepared in the lab. First, the peak for the compound MIBK, for example, was found on the standard sample on the GC-MS by estimating its retention time. Then, use the estimated retention for MIBK to see it in the standard samples. Once we find the compound peak, we refine the search. Then search for the compound in 4 different volumes. The volumes are 150 mL, 100 mL, 75 mL, 50 mL, 25 mL, 10 mL, and 5 mL. As the volume decreased, it was harder to find the peaks for the compound. However, it was essential to note if the compound was found even in the smallest sample.

III. Testing the two techniques for the GC-MS analysis (EIC vs. TIC)

The two techniques used to find the concentration of the VOCs were EIC and TIC. EIC stands for Extracted Ion Chromatography, where we set the Target view system for a specific mass for each compound. This mass is determined by having the highest peak and being unique in the compound. For some compounds, the highest peak was the last ion. The other technique is Total Ion Chromatography, which searches for the peaks based on each compound's retention time without specifying any unique ions.

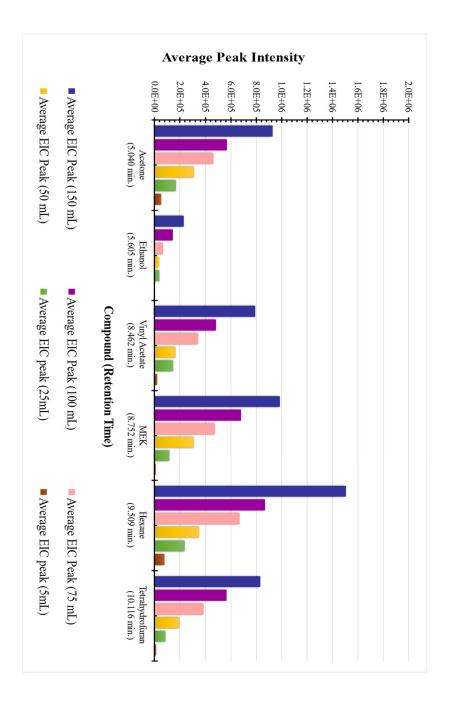
After collecting all the data for the standards, we calculated the average of all the volumes and their peaks. Then, we reported the percent RSD (Relative standard deviation), the R squared value, the standard deviation, and the equation for the graph. These calculations were done using Excel to ensure the data collected was accurate and see if any data were outliers.

IV. Analysis of the samples collected from Shelby County using EIC

After gathering the equations for each compound using EIC and TIC, the samples from Shelby, Tennessee, are analyzed for the concentrations of ketones. We went through each location and searched for MIBK, acetone, MEK, and acetone peaks. After the data was analyzed, the peaks for each compound were used to calculate the ppbv using the Eurofins calculator to convert the concentrations from the microgram/ cubic meter to ppbv.⁹ After the concentrations are determined, the data is compared to the reported TRI data from various Memphis sampling locations in Shelby County.

Results and Discussion:

I. <u>EIC VS. TIC</u>



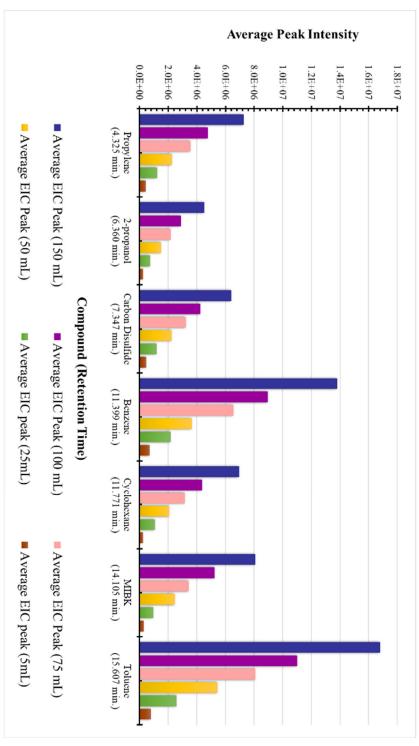


Figure 1: The concentration of fourteen VOCs standards prepared in the lab at

Figure 1 This chart is all 14 compounds analyzed on the x-axis with their retention time versus their standard concentration on the y-axis. Those compounds are benzene, toluene, ethanol, acetone, hexane, carbon disulfide, methyl ethyl ketone, methyl isobutyl ketone, propylene, isopropyl alcohol, p-xylene, cyclohexane, tetrahydrofuran, and vinyl acetate. GC-MS was used to detect the retention time of these VOC compounds. Since there are various volumes, they were color-coded based on their volumes. The highest volume provided the highest peaks on this graph, 150 mL. The x-axis is the retention time, and the y-axis is the concentration/intensity. This data was obtained from the EIC peak area.

Methyl Isobutyl ketone: EIC-43

Table 1: GC-MS calibration data of methyl isobutyl ketone standards using EIC at m/z 43

Std. Vol (mL)	EIC-43	mass (ng)
150	8.08E+06	2.46E+00
100	5.25E+06	1.64E+00
75	3.44E+06	1.23E+00
50	2.47E+06	8.19E-01
25	9.48E+05	4.10E-01
5	2.75E+05	8.19E-02

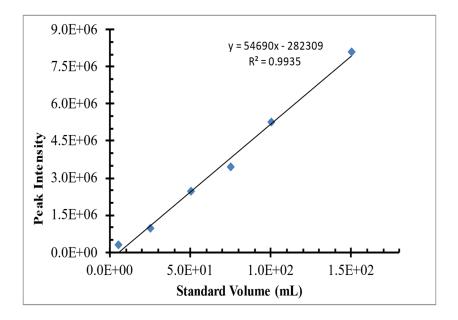


Figure 2: Methyl Isobutyl ketone volume standards using EIC at m/z 43

Figure 2 focuses on the extracted ion chromatography of MIBK. The EIC was set to 43 amu because the 43 ions give more precise results in the data. This graph includes the volume on the x-axis. The volume was calculated using the average of the four samples per volume. The y-axis indicates the peak, giving the compound concentration present in the sample. The R2 value is 0.9935, meaning that the data fit the line and are accurate and precise. The closer the R2 value to 1, the more precise the data. Using *Table 1*, the graph in *Figure 3* was drawn. The equation for the graph (y=mx+b) is in the top left.

Table 1 has volumes on the left in mL, the EIC-43 in the middle, and the percent RSD on the left. The 43 Ion was the most unique for MIBK. The distinctive peak is not at the end because similar isomers and other compounds have equal molecular weights. The peak with the highest intensity is 43, which makes it the most unique.¹¹ The average intensity at the retention time of 14.105min for MIBK is recorded in the middle column. In the data reported by the EPA, MIBK is the 31st highest VOC with 3162 pounds of release in 2014.¹ This shows that each on-site area should be regularly monitored because it is not always the reported VOC that was the highest concentration and was released the most. The mass in ug was calculated by using a 4 ppbv standard for methyl isobutyl ketone.

Methyl Isobutyl ketone TIC:

Std. Vol (mL)	TIC	mass (ng)
150	1.88E+07	2.46E+00
100	1.20E+07	1.64E+00
75	8.32E+06	1.23E+00
50	5.22E+06	8.19E-01
25	6.18E+06	4.10E-01
5	3.58E+06	8.19E-02

Table 2: GC-MS calibration data of methyl isobutyl ketone standards using TIC.

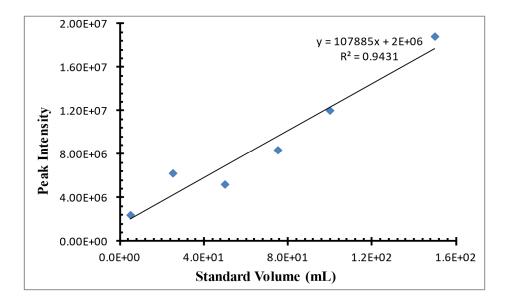


Figure 3: Methyl Isobutyl ketone volume standards using TIC

In Table 2, The Total Ion Chromatogram (TIC) mode of GC-MS was used, and the average was calculated for the data. The TIC is not as accurate as EIC because it does not focus on a specific ion. This data was recorded based on MIBK retention time and its peaks. The average was calculated by taking the average of the four different samples of each volume. For MIBK, the 10 mL was considered an outlier because the compound was not found at the expected retention time but at 13.314 min, which is not the retention time for MIBK. The mass in ug was calculated by using a 4 ppbv standard for methyl isobutyl ketone. Figure 3 is focused on the Total Ion chromatography of acetone. This graph includes the volume on the x-axis. The volume was calculated using the average of the four samples per volume. The y-axis indicates the peak, giving the concentration of the compound present in the sample. The R2 value is 0.9431, which indicates that the data fit the line and are accurate and precise. The closer the R2 value to 1.000, the more precise

the data. Using *Table 4*, the graph in *Figure 5* was drawn. The equation for the graph (y=mx+b) is on the top left. Using *Table 2*, the graph in *Figure 3* was drawn, and the equation for the graph (Y=mx+b) is in the top left.

Acetone: EIC-58

Figure 4 focuses on the extracted ion chromatogram of acetone. The EIC was set to the m/z value of 58 because the ion corresponds to the molecular ion of acetone. This graph includes the volume on the x-axis. We calculated the volume using the average of the four samples per volume. The y-axis indicates the peak, which also refers to the concentration of the compound present in the sample. The R² value is 0.9934, proving that the data fit the line. The closer the R² value to 1.000, the more significant the correlation between analyte concentrations and the EIC signals. Using the data from *Table 3*, the graph in *Figure 4* was drawn. The equation for the graph (Y=mx+b) is in the top left.

Std. Vol (mL)	EIC-58	mass (ng)
150	9.30E+05	1.43E+00
100	5.68E+05	9.50E-01
75	4.63E+05	7.13E-01
50	3.13E+05	4.75E-01
25	1.69E+05	2.38E-01

Table 3: GC-MS calibration data of acetone standards using EIC at m/z 58

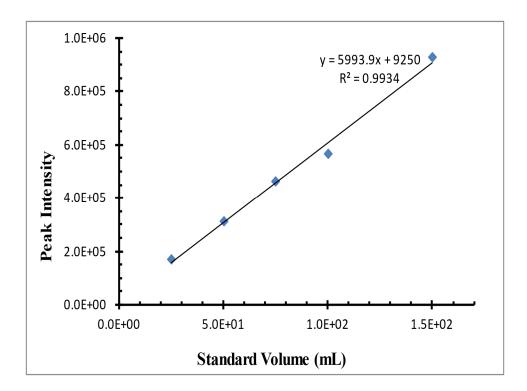


Figure 4: Acetone volume standards using EIC at m/z 58

In *Table 3*, The Extracted Ion Chromatography (EIC) was set to 58 amu, and the average was calculated for the data. This data was recorded based on acetone retention time and its peaks and then calculated the average by taking the average of the four different samples of each volume. For acetone, the 10 mL and the 5 mL was an outlier because the concentration compound increased when it was supposed to decrease as the volume decreased. The mass in ug was calculated by using a 4 ppbv standard for acetone.

Acetone TIC:

Table 4: GC-MS calibration data of acetone standards using TIC

Std. Vol (mL)	TIC	mass (ng)
150	1.85E+07	1.43E+00
100	1.14E+07	9.50E-01
75	8.76E+06	7.13E-01
50	6.20E+06	4.75E-01
25	3.74E+06	2.38E-01

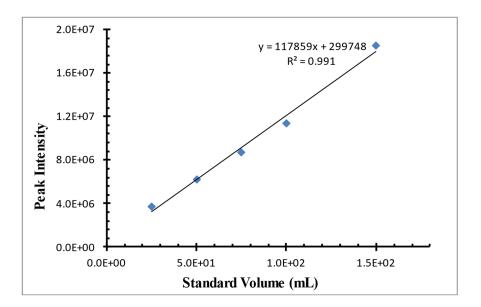


Figure 5: Acetone volume standards using TIC

In *Table 4*, The Total Ion Chromatogram (TIC) mode was used, and the average was calculated for the data. The TIC is less accurate than EIC because it is more susceptible to isobaric interferences. This data was recorded based on Acetone retention time and its peaks. The average was calculated by taking the average of the four different samples of each volume. For acetone, the 10 mL and the 5 mL was an outlier because the concentration compound increased when it was supposed to decrease as the volume decreased. The mass in ug was calculated by using a 4 ppbv standard for acetone.

Figure 5 is focused on the Total Ion chromatography of acetone. This graph includes the volume on the x-axis. The volume was calculated using the average of the four samples per volume. The y-axis indicates the peak, giving the concentration of the compound present in the sample. The R2 value is 0.991, which indicates that the data fit the line and are accurate and precise. The closer the R2 value to 1.000, the more precise the data. Using *Table 4*, the graph in *Figure 5* was drawn. The equation for the graph (y=mx+b) is on the top left.

We set the Target View system for a specific mass for each compound. This mass is determined by having the highest peak and being unique in the compound. The other technique is Total Ion Chromatography, which searches for the peaks based on each compound's retention time without specifying any individual ions. After looking at both graphs for each compound using EIC and TIC techniques, we realized that the R² value for the EIC was always better. The EIC was a more specific technique for each compound and gave us more accurate data because the ions we picked were specific to their respective compounds. While with TIC, we can have another compound different from what we are searching for and have a peak. After seeing these results, we agreed to proceed with the research and calculate the concentrations of each ketone using the equation from the EIC technique and the peak of the compound.

II. Summer vs. Winter data

Using the equation calculated by the standards using the EIC technique, the parts per billion volume (ppbv) was calculated for the Shelby County locations. **Figure 6** shows the data from the winter samples, and **Figure 7** shows the results from the summer samples. After calculating Acetone, MEK, and MIBK concentrations, I used a tri-explorer to locate all the factories that emit these three VOCs in Shelby County. ¹⁰ The purpose of locating these factories is to understand where those emitted VOCs come from. **Figure 9** shows the winter locations with the factories found on TRI Explorer for 2014.¹⁰ The factories are in a tetrahedron shape (upside-down pyramid) and are color-coded based on the ketones they emit. Overall, we can see that the factories that are in black are the ones that emit all three ketones and are the ones surrounded by all the high peaks.

Looking at both graphs, it is evident that the concentration of ketones in the summer is higher than in the winter. **Figure 8** shows both the summer and winter sample concentrations on the same map. The ppbv values for acetone in the summer could reach up to 380 ppbv, while the highest in the winter samples did not exceed 30 ppbv and averaged around 28.4 for July and August, 3.45 for December-February. MEK ppbv values were much lower than the acetone values. In the summer, MEK averaged around 0.94 and

as low as 0.09 for the winter. MEK was not found in most locations for the winter, and when it was found, it had very small peak areas. MIBK was the most challenging to find because it was present in very low concentrations; it was only found in 5 locations in the summer out of all the locations used. The average concentration for MIBK is 2.0 ppbv in the summer. **Figure 8** shows that the summer concentrations were much higher than the winter concentrations in Memphis.

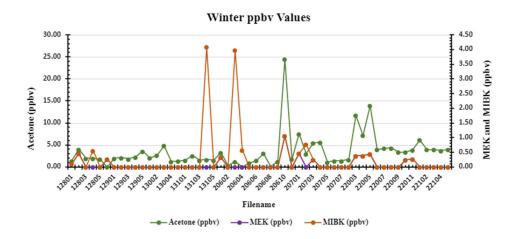


Figure 6: Concentration of acetone. MIBK, MEK in the winter (Dec – Feb)

Figure 6 shows the results of the ppbv concentrations on the y-axis and the file name of the locations on the x-axis. The y-axis on the left is for acetone because it shows higher concentration values than the other two ketones on the right of the graph.

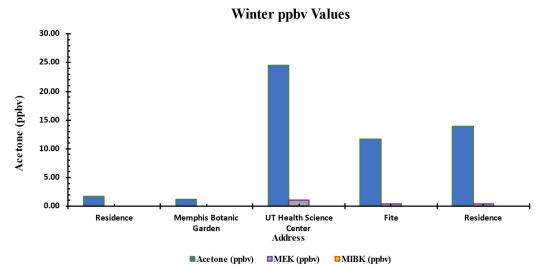


Figure 6.1 A closer look at the highest concentrations from Figure 6

Figure 6.1 A closer look at the highest concentrations from the winter

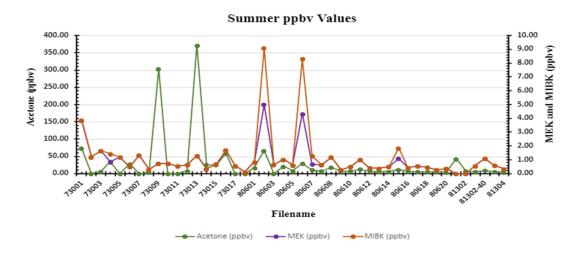


Figure 7: Concentration of acetone. MIBK, MEK in the summer (Jul - Aug):

Figure 7 shows the results of the ppbv calculations for the summer samples collected. Again, the left y-axis is for acetone, and the right y-axis is for MEK and MIBK. Again, the concentration of acetone was much higher than MIBK and MEK.

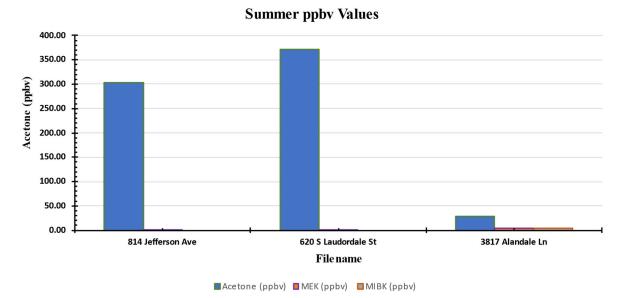


Figure 7.1 A closer look at the highest concentrations from Figure 7



Figure 8: Winter vs. summer concentrations on the map:

Acetone, MEK, and MIBK were much higher in the summer since, with higher temperatures, ketones evaporate and remain in the ambient air. Also, this may be due to sampling in downwind locations. In the winter, the temperature was low, so it did not evaporate as much as it did in the summer. Acetone (C₃H₆O) does not exhibit hydrogen bonding, and the molecules are attracted to each other using dipole-dipole interactions and London forces, which require 0.5-1 kcal/mol, which is very low compared to water for reference, which requires 4-5 kcal/mol to break its hydrogen bonds. MEK (C₄H ₈O) and MIBK (C₆H₁₂O) have the same forces as acetone, but since they are larger compounds with more carbon and hydrogens, they require more energy to evaporate. ¹³ Also, the summer has a higher vapor pressure than the winter; higher vapor pressure is proportional to the compound's volatility. This means that it will stay longer and the air.

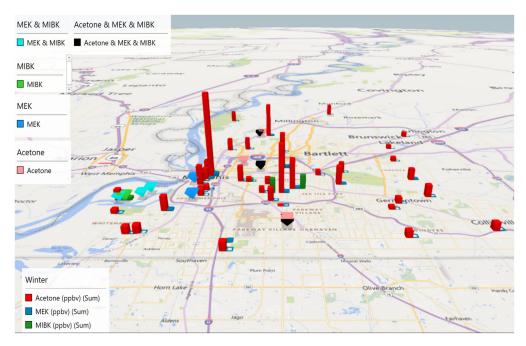


Figure 9: Winter concentrations with factories for Shelby County:

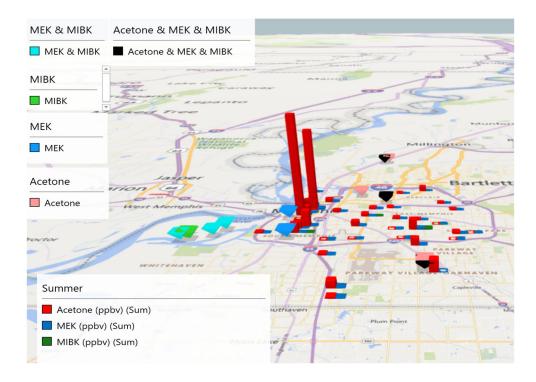


Figure 10: Summer concentrations with factories for Shelby County:

The volatile organic substance detected from the GC-MS analysis is a health threat for the workers of these factories and residents of Shelby County and surrounding counties. Breathing these compounds could irritate the eye, nose, and throat and cause difficulty breathing and nausea. Some of those compounds could cause cancer, but not all ("Volatile organic compounds, "2020).¹²

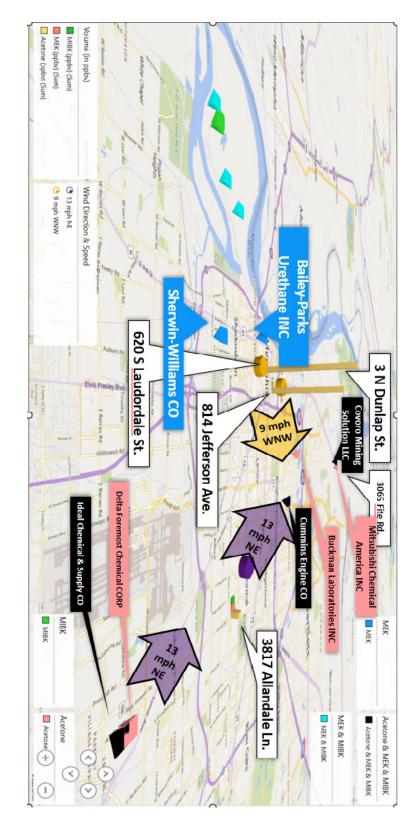


Figure 11: Summer wind speed and direction (July – August)

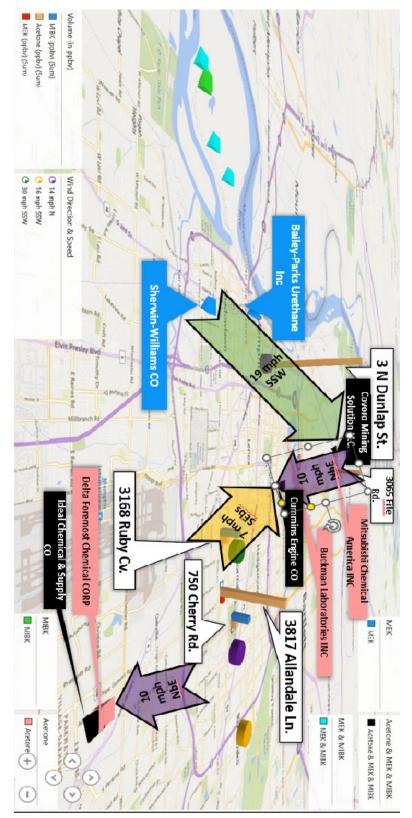


Figure 12: Winter wind speed and direction (December -February)

Figures 11 and 12 show the wind directions for Memphis, TN, showing the reason why the highest concentrations of each compound are in their specific locations.

III. Side Effect of High Concentrations of Ketones

According to the USEPA, side effects for acetone occur from constant exposure to 416-890 ppm.⁷ However, from our analysis, the acetone concentration in Shelby County did not reach the range where it would cause a risk. The highest concentration detected for acetone was 372 ppb which is 0.372 ppm, which is well below the risk range.⁷ Secondly, Methyl Isobutyl ketone was the second compound focused on in this research. MIBK was expected to have a high concentration because of the tri-explorer data of the reported concentrations of VOCs in Shelby County. MIBK was the only ketone that showed up on the TRI-explorer-reported table of the most compounds released, and it was the 31st in Shelby, TN.¹⁰ However, from the samples analyzed in this research, MIBK had the lowest concentrations and was not detected under the prevailing wind conditions. In case of an increase in MIBK to 7.50E+05 ppbv, the side effects start appearing, primarily affecting the workers of these factories.¹⁴

Methyl Ethyl ketone was the third compound analyzed in this research. MEK was very similar to MIBK in results; however, it was detected more and had a slightly higher concentration. The Reference Concentration (RFC) for MEK is 1 mg/m³, which is 334 ppb

based on the data reported by the USEPA from the decreased fetal birth weight in mice. Against MEK, the data from research does not reach the risk concentration to have any effect on the residents.¹⁵

Conclusion:

The USEPA is aware of the health risks that the VOCs pose to public health, which is why they have specific regulations for releasing these compounds. Companies must limit their VOC release and report the gasses released annually. GC-MS analyzes the data collected to find the concentration of those VOC present. Thankfully, the ketones analyzed in this research did not reach a high concentration which would be a health risk and cause side effects to public health.

This research concluded that the EIC technique was the best for our data, getting better results. This was indicated by better R² values and the fact that we are more confident that the compound will exist at its specific ion. Therefore, we can rely on the accuracy of the data collected for this research. Although the technique takes a long time to perform, I think it is worth using it to ensure the data's accuracy. If we had followed the TIC technique by only checking the compound at its retention time, we would have missed many peaks. The compounds sometimes appeared at a slightly different retention time. Also, with the TIC technique, we could have many peaks around very similar retention times. So, the EIC solves this issue by detecting the peak area at a specific unique ion for each ketone plus using the retention time.

Lastly, it was very significant to realize how the emission of ketones varies in different seasons, as seen from the data of this research. For example, the concentrations of ketones were significantly higher in the summer than in the winter. This may be due to the higher temperature that causes the ketones to remain in the vapor phase in the summer. And the wind directions played a major role in explaining why the concentration was higher in some locations.

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