

Overview

Purpose:

- To establish a method for analysis of orange essential oil in the Vernier Mini GC Plus for a high school laboratory experiment.
- To see if silylation successfully increases the volatility of orange oil.

*An assumption was made that D-Limonene was the main component of orange oil.

*MSTFA is known as N-Methyl-N-(trimethylsilyl)-trifluoroacetamide.

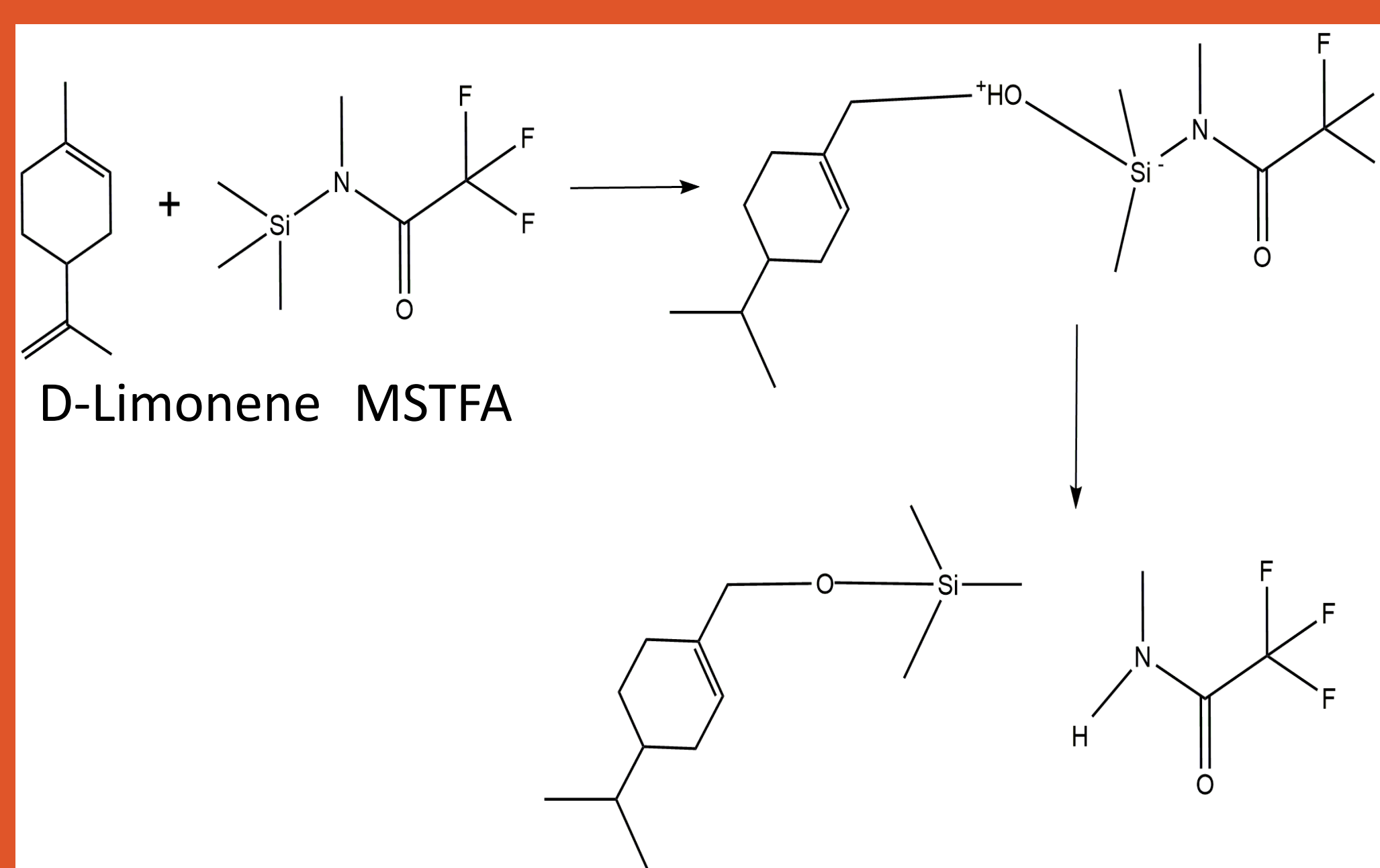


Figure 1. The proposed reaction mechanism for D-Limonene and N-Methyl-N-(trimethylsilyl)-trifluoroacetamide (MSTFA).⁵



Figure 2. The HP 5890 Gas Chromatograph. (28" x 20")



Figure 3. The Vernier Mini GC Plus Gas Chromatograph. (5" x 7")

SILYLATION AND ANALYSIS OF ORANGE OIL IN THE VERNIER MINI GC PLUS

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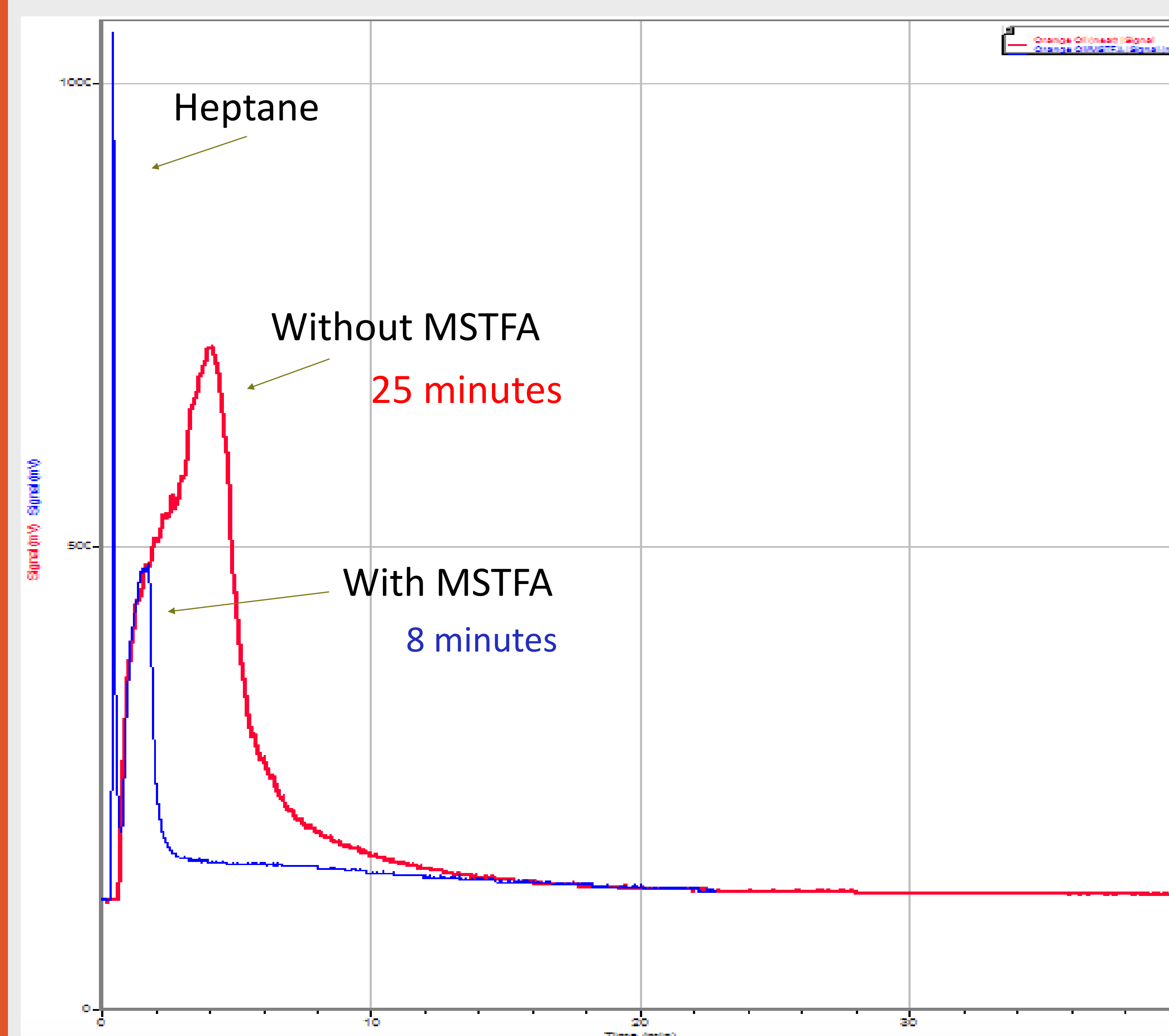


Figure 4. Logger Pro analysis of orange oil with and without MSTFA

Experiments

Note: The orange oil used for all experiments was extracted using EssenEx-100 Microwave Extron.

Mini GC Plus A baseline of methanol and ethanol retention times and peak areas was obtained by running 0.1uL five times each. Methanol was also run at varying volumes from 0.05uL to 1.0uL to determine the resolution of the Mini GC.

- Pure orange oil was run in the Mini GC at 150°C, 21kPa, and High Sensitivity at varying volumes from 0.1uL to 1.0uL. Ten runs were also conducted at 0.8uL and the retention times and areas were analyzed.
- 100mg of orange oil was silylated using 100uL MSTFA in a solution of 3mL heptane for 20 minutes. The same tests conducted on pure orange oil were run on the silylated sample of orange oil for comparison of retention times and peak areas.

APPLICATIONS IN THE CLASSROOM

- 10 min:** Grating orange peels.
- 5 min:** Assemble reactor and place in microwave
- 5+2 min:** 5 minutes microwave extraction, 2 minutes to cool down
- 3 min:** Collect oil
- *20 min:** Allow oil to react with MSTFA in heptane
- 10 min:** Inject oil into Mini GC Plus and analyze with Logger Pro
- Total: 55 minutes** (35 with MSTFA solution already available)

*By having pre-prepared orange oil/MSTFA solutions, this step can be omitted.



Figure 5. Orange oil before and after MSTFA reaction.

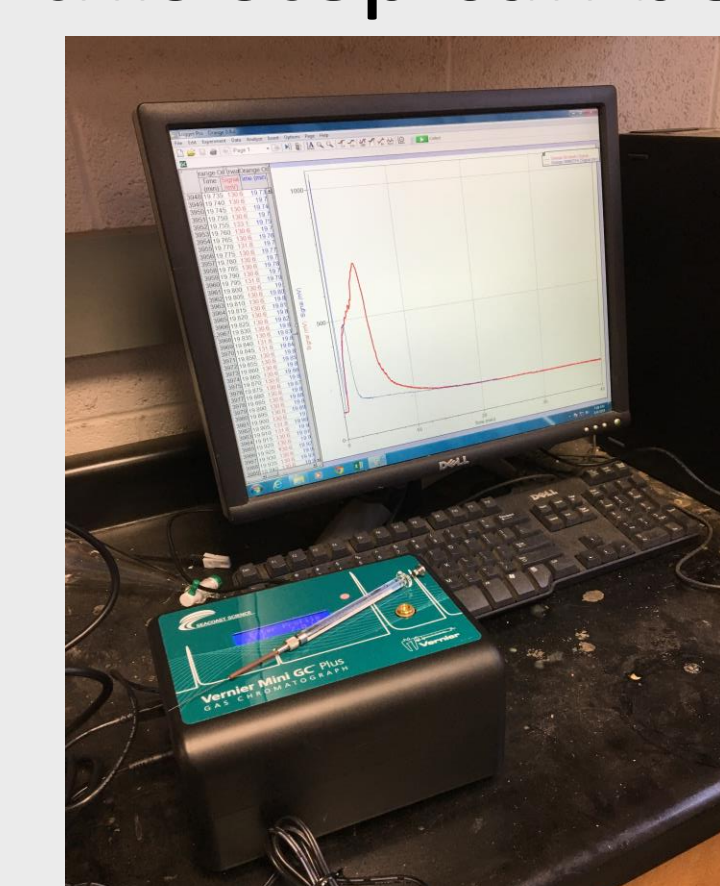


Figure 6. Experimental setup.

Run #	Retention Time of Orange Oil Without MSTFA (min)	Retention Time of Orange Oil With MSTFA (min)
1	3.41	1.77
2	3.62	1.75
3	4.24	1.77
4	3.83	1.56
5	3.96	1.76
6	4.25	1.64
7	3.83	1.55
8	3.98	1.71
9	3.89	1.60
10	3.77	1.49
Average	3.88 ± 0.26	1.66 ± 0.11

All runs were conducted at 21kPa, 150°C, and High Sensitivity on the Mini GC Plus for up to 50 minutes per run.

Table 1. Retention times of orange oil with and without MSTFA

Discussion & Results

The retention time of Orange Oil decreased by over half after MSTFA was injected into the sample. Silylation of D-Limonene was successful despite the lack of an -OH group on D-Limonene. Successful analysis of orange oil provides an expansion of the capabilities of the Mini GC Plus. The decrease in time allows for a potential high school laboratory experiment.

Conclusion

Silylation of orange oil successfully increases its volatility for analysis in the Mini GC Plus. The objective of the research was achieved.

Future Work

Silylation and analysis of other essential oils in the Mini GC Plus, such as lavender and peppermint oil. Additional silylation reagents for orange oil will be tested to optimize process for educational laboratory experiments.

Acknowledgments

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References

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Figure 7. The Hamilton Mini GC 1.0uL Syringe.