

Gas Chromatography

Ilana Berlin (worked with Gina Dazzo)

Tuesday January 23rd 2024

The purpose of this lab is to

- Record the molecular properties, of five molecules
- Run gas chromatography of each of the five compounds to determine retention time (T_r) in triplicates.
- Run gas chromatography for an unknown mixture to determine composition and percent abundance
- inject compounds into a gas chromatographer and read a ch [omit]
- Model compounds and calculate dipoles in Spartan Student v9
- Increase understanding of the function of intermolecular forces and role in det of T_r

Reference:

- (1) Aldrich Chemical Company. Aldrich Catalogue Handbook of Fine Chemicals; Milwaukee, WI : Sigma-Aldrich, **2002**.
- (2) Kateley, L. J., *Introduction to Chemistry in the Laboratory*, 20th Ed., Lake Forest College, **2021**, Experiment 1, Appendix I.

Observations and Data:

1. GC of Pure Compound*

*See GC table (Figure 3)

To determine the retention time, used needle bleed of pure substances in a gas chromatographer.

2. Separating and Quantifying Components in the Mixture

Using needle bleed, a small amount of mixture one was injected into a gas chromatograph. The mixture smelled sweet and fruity, akin to sweet tarts candy.

Channel 1														
Peak	Fill	Peak Name	t _R (min)	t _S (min)	t _E (min)	H (V)	H _{Norm}	A (V.s)	A _{Norm}	W _{0.1}	Asym	Effic	Res	Type
1		1	0.15	0.10	0.17	0.02	18.52	0.02	13.45	0.02	2.85	728		BD
2		2	0.21	0.17	0.24	0.02	19.72	0.02	17.88	0.03	0.52	1268	2.65	DV
3		3	0.27	0.24	0.32	0.03	27.69	0.03	24.17	0.03	0.75	1925	2.69	VB
4		4	0.52	0.46	0.55	0.02	18.92	0.03	23.09	0.04	1.10	3036	7.99	BD
5		5	0.58	0.55	0.65	0.02	15.15	0.03	21.40	0.05	1.02	2808	1.42	DB
			0.11	100.00	0.13	100.00								

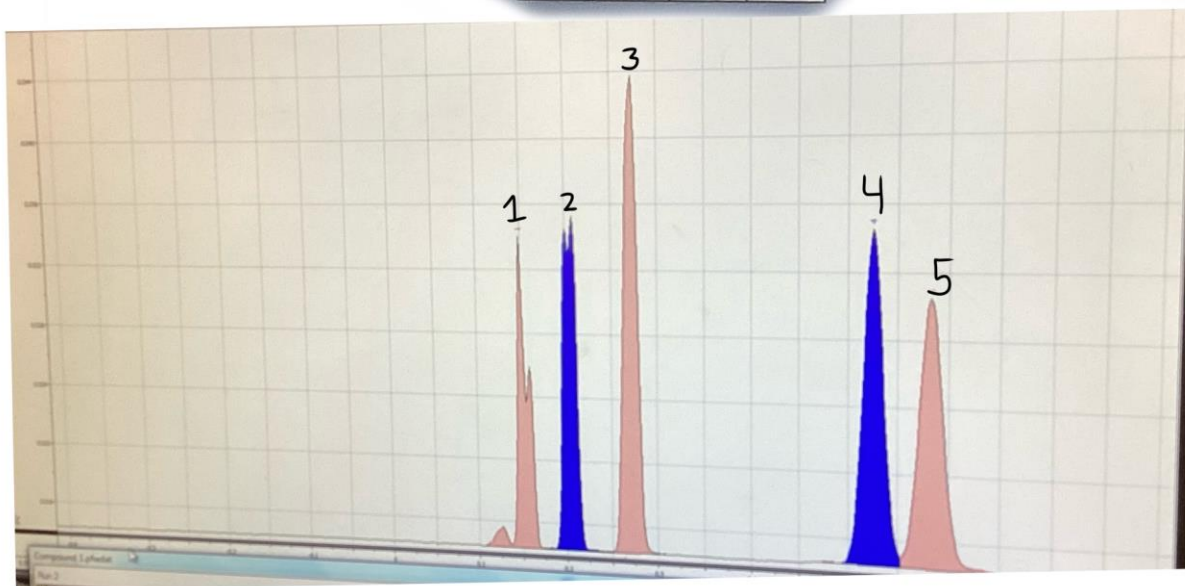


Figure 1. Chromatogram

Compound	Results(T _r (min))	% Composition of Mixture (A _{Norm})
Pentane	0.15	13.45
2-butanone	0.21	17.88
1-butanol	0.27	24.17
Isobutyl acetate	0.52	23.09
Ethyl trimethylacetate	0.58	21.40

Table 1. Mixture Results

GC Parameters Apparatus-Better image of instument available

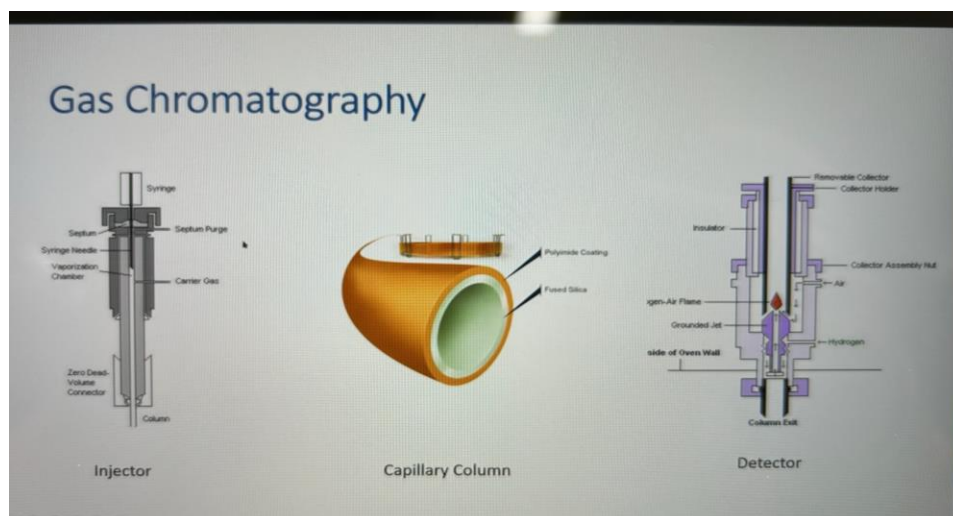


Figure 2. Gas Chromatography

Instrument	HP5890
Oven	50°C
Injector	120°C
Detector	150°C
Split vent flow	44-45mL/min
Column flow	35mL/min
Sample size	needle bleed
Column Restek 5	Crossbond® 5%diphenyl/95%dimethy polysiloxane
Column length	15 meters
Column inside diameter	0.53mm
Column catalog number	10252
Serial number	118671A

Table 2. Instrument information

GC Table. Structures and Properties

add IMF
Ilana Berlin

TURP in Tablae (Pepo)

GAS CHROMATOGRAPHY: STRUCTURES AND PROPERTIES
The compounds for the following table, in alphabetical order, are: 1-butanol, 2-butanone, ethyl trimethylacetate, isobutyl acetate, and pentane. Use online resources or the Aldrich Handbook to find the molecular weight (MW) and boiling point for each. Obtain dipole moments from your computational chemistry exercise. Use GC class data for t_r values. Dipole moments for the two acetates are values from a previous computational chemistry exercise. It will be most convenient if you list the compounds in order of increasing retention times once these values are available from the class data.

Compound	Line Formula	Structure (showing molecular dipole)	Molecular weight	bp, °C	dipole moment, debye	retention time, min.	Order of elution + IMF
1-butanol	$\text{CH}_3(\text{CH}_2)_3\text{OH}$ <i>CH₃OH</i>		74.12	117.73	1.87	0.303 0.292 0.312 0.302	3 D, D, H, B
2-butanone	$\text{CH}_3\text{COCH}_2\text{CH}_3$		72.11	79.6	3.00	0.211 0.211 0.209 0.210	2 D, D, D
ethyl trimethylacetate	$(\text{CH}_3)_3\text{CCOOCH}_2\text{CH}_3$ <i>C₂H₅</i>		130.18	118	1.78	0.596 0.600 0.601 0.599	5 D, D, D
isobutyl acetate	$\text{CH}_3\text{COOCH}_2\text{CH}(\text{CH}_3)_2$ <i>CH(CH₃)₂</i>		116.16	115-117	4.86	0.545 0.547 0.539 0.544	4 D, D, D
pentane	$\text{CH}_3(\text{CH}_2)_3\text{CH}_3$		72.15	36.1	0.04	0.165 0.170 0.164 0.166	1 D

91

Figure 3. Structures and properties of compounds

Dipole Moment Calculations

The dipole moment was modeled using Spartan Student v9.

Questions

- 1) Peaks were identified based on retention time (T_r)
- 2) Retention time for a given compound is reproducible given unchanged conditions. This means the helium flow should be consistent and the coil of the gas chromatographer has been kept at a consistent temperature among other factors.

The percent values are reproducible given the same unchanged conditions and if the sample has been kept in a sealed container to prevent evaporation.

- 3)
 - a) If the temperature is lowered the T_r will increase because there is less energy in the system so the molecules will move slower. This will not change the proportions?, %?.
 - b) If the helium flow rate is increased the T_r will decrease as the increased flow will carry things through the column faster. This will not change the proportions.
 - c) If the column length is doubled then the T_r will increase as there is a larger distance for the molecules to travel. This will not change the proportions.

Conclusion:

- 1) See Table 1
 - a) The most abundant compound in mixture one was 1-butanol at 24.17%
 - b) The least abundant compound in mixture one was pentane at 13.45%
- 2)

a)

Compound	MW	BP	IMFs	Order of elution (T _r)
Pentane	72.15	36.1	Dispersion	1
2-butanone	72.11	79.6	Dispersion Dipole-dipole	2
1-butanol	74.12	117.73	Dispersion Dipole-Dipole Hydrogen Bonds	3

Pentane is the smallest compound with the least intermolecular forces, and the least polarity so it is the fastest to elute. 2-butanone has stronger dipole-dipole forces with more polarity so it elutes slower than pentane but not as slowly as 1-butanol which has the strongest intermolecular forces, hydrogen bonds.

b)

Compound	MW	BP	IMFs	Order of elution(T _r)
1-butanol	74.12	117.73	Dispersion Dipole-dipole Hydrogen bonds	1
Isobutyl acetate	116.16	116.5	Dispersion Dipole-dipole	2
Ethyl trimethylacetate	130.18	118	Dispersion Dipole-dipole	3

1-butanol is the lightest compound so, despite its stronger intermolecular forces, it elutes the fastest. Ethyl trimethylacetate is the heaviest and largest compound (big surface area) so there is more room for it to interact with the coating of the column, causing it to elute last.

GC use, importance