Gas Chromatography

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ELEC 402: Senior Project
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# Midterm Presentation

- Background Review
    - Gas chromatography and detector
- Fall Semester
    - Update on detector and carrier gasses
    - Previous prototype
- Spring Semester
    - Colorimetric array
    - Current prototype
    - GUI, ADC, and software
- Future

Reminder: Gas Chromatography is a Separatory Technique

- Allows qualitative and quantitative analysis of solutions with volatile components

https://en.wikipedia.org/wiki/Response_factor
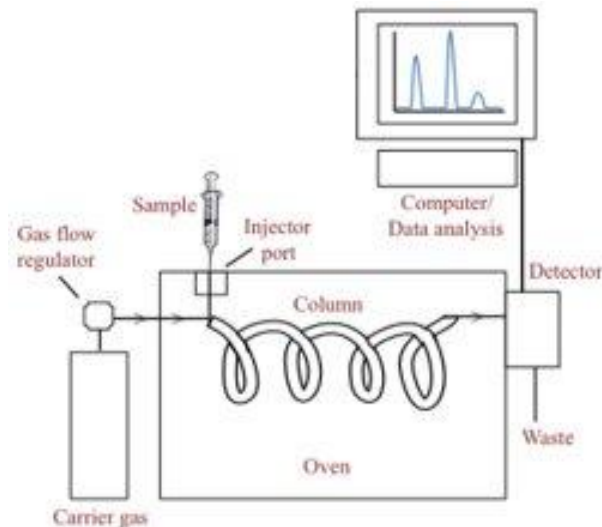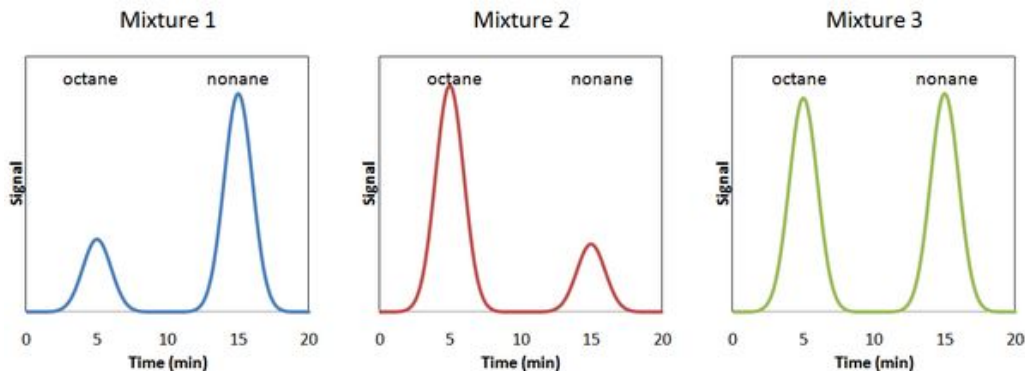
Reminder: Detector Operating Principle

- Diode thermal conductivity detector
  - Does not require an inert carrier gas
  - Rugged enough for classroom use
  - Cheap and simple to replace
  - Poor sensitivity
    - Increased sample volume can mitigate this somewhat
- Note:
  - Dried air (32mW/(m*K) @ 125C)
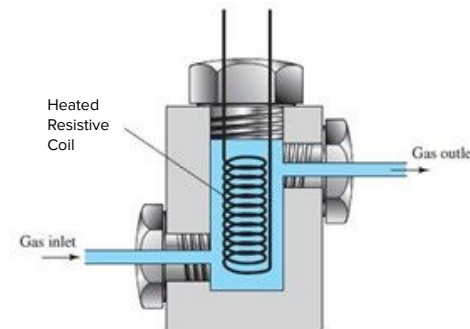
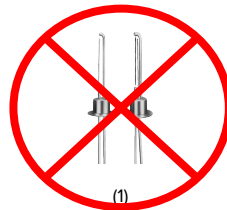| Compound	25°C	125°C	225°C
Acetone	11.5	20.2	30.6
Methane	34.2	49.1	66.5
Methanol	-	26.2	38.6
Ethanol	14.4	25.8	38.4
Hexane	-	23.4	35.4

Thermal Conductivity of Common Analytes (mW/(m*K))
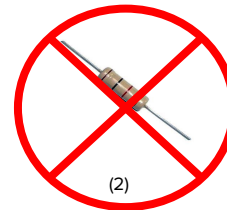
http://www.gaschromatograf.de/documentation_eng.ht ml

Updated Detector Selections

- ~~Rhenium-tungsten~~
- ~~Carbon-film resistor~~
- 1N4148 Diode

Updated Carrier Gas Selections

- Dried air (32 @ 125C)
- ~~Nitrogen (32 @ 125C)~~
- ~~Helium (190 @ 125C)~~

(1)

(2)

(3)

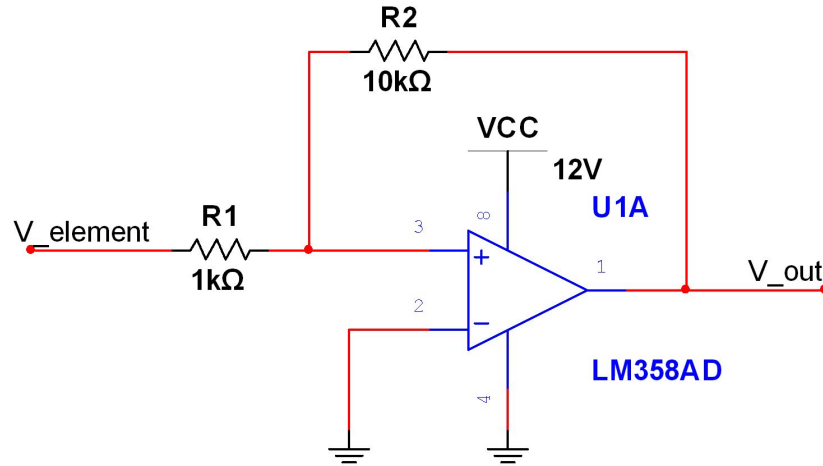(1)   https://www.bucksci.com/products/tcd-filament-tungsten-rhenium-includes-seal
(2)   https://www.westfloridacomponents.com/G530APF08/2W+15K+ohm+Carbon+Film+Resistor+Paccom+RD200T.html
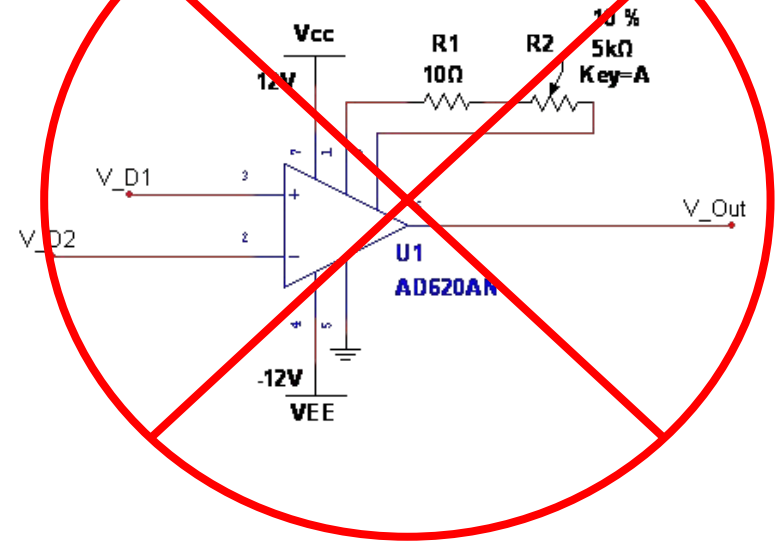(3)   https://www.digikey.com/product-detail/en/on-semiconductor/1N4148TR/1N4148FSCT-ND/9356376
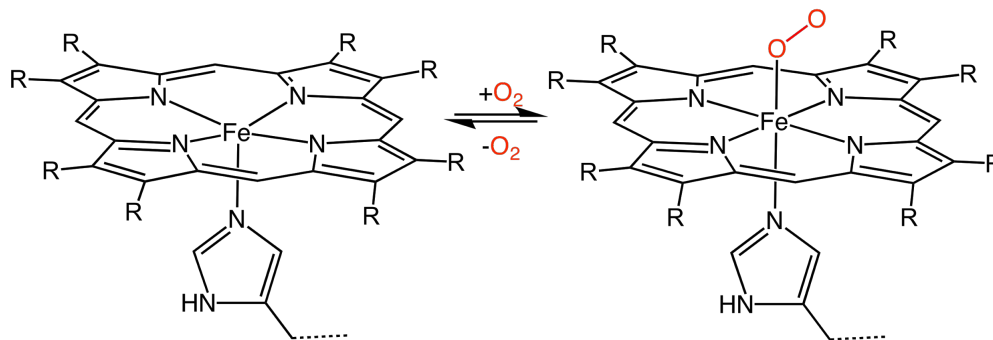
Updated Configuration Selection

Single-ended
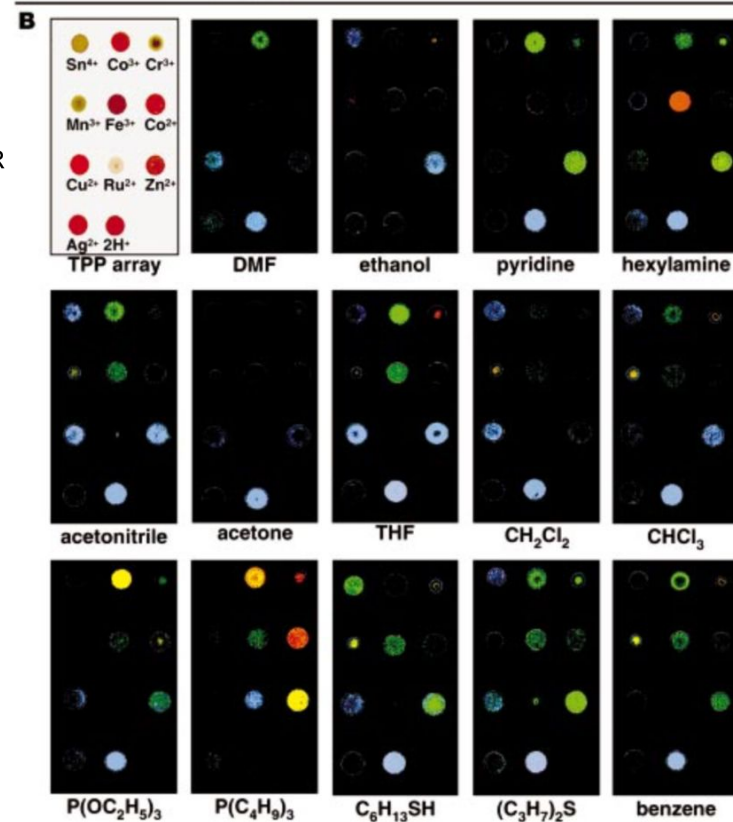

Differential

Reminder: Colorimetric Array Detector

Deoxyhemoglobin
- Dark red
- Fe^{2+} oxidation state

Oxyhemoglobin
- Bright red
- Fe^{3+} oxidation state
- Oxygen ligand

K. Rakow, N. & Suslick. A colorimetric sensor array for odour visualization. Nature, 406:710–713, August 2000.

Progress: Colorimetric Array Detector

- **Innovation**: Sulfonate the metalloporphyrin compounds so they can be dissolved in water.
  - Avoids chloroform, dimethyl sulphoxide (DMSO), and dichloromethane (DCM)
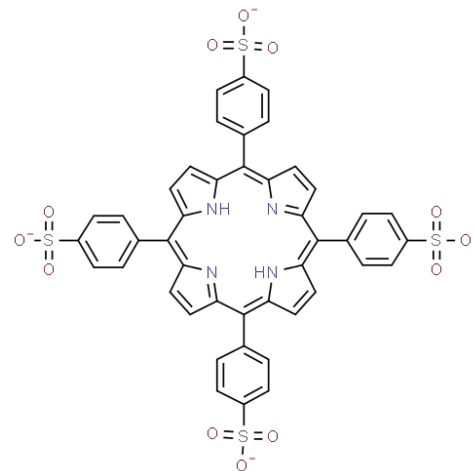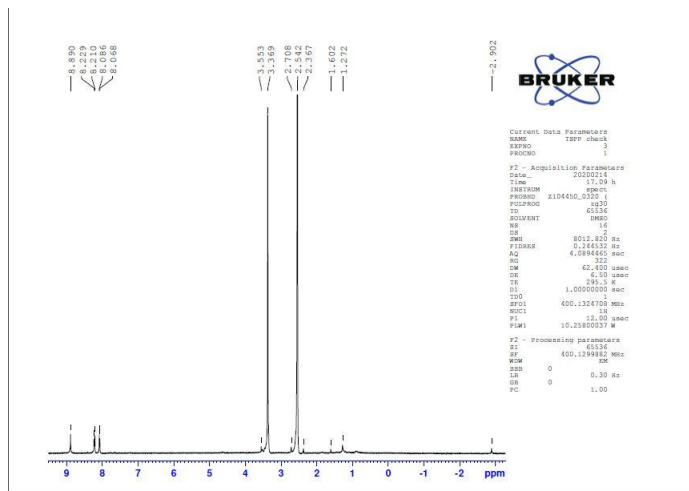
5,10,15,20-tetrakis(4-sulfonatophenyl)porphyrin
(Source: ChemSpider)
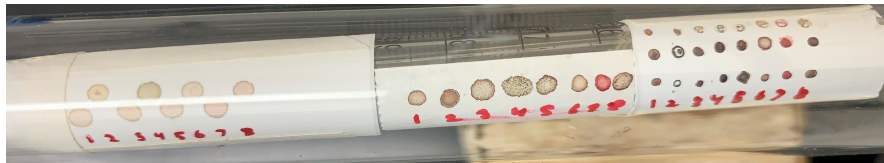
Testing the Colorimetric Array Detector
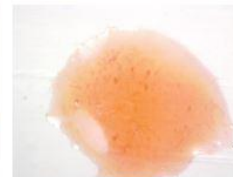

Figure 1: TSPP(Co^{2+}) initial

Figure 2: TSPP(Co^{2+}) w/ acetonitrile

Figure 3: TSPP(Co^{2+}) change

Figure 4: TSPP(Fe^{3+}) initial

Figure 5: TSPP(Fe^{3+}) w/ acetonitrile

Figure 6: TSPP(Fe^{3+}) change

Figure 7: TSPP(Ni^{2+}) initial

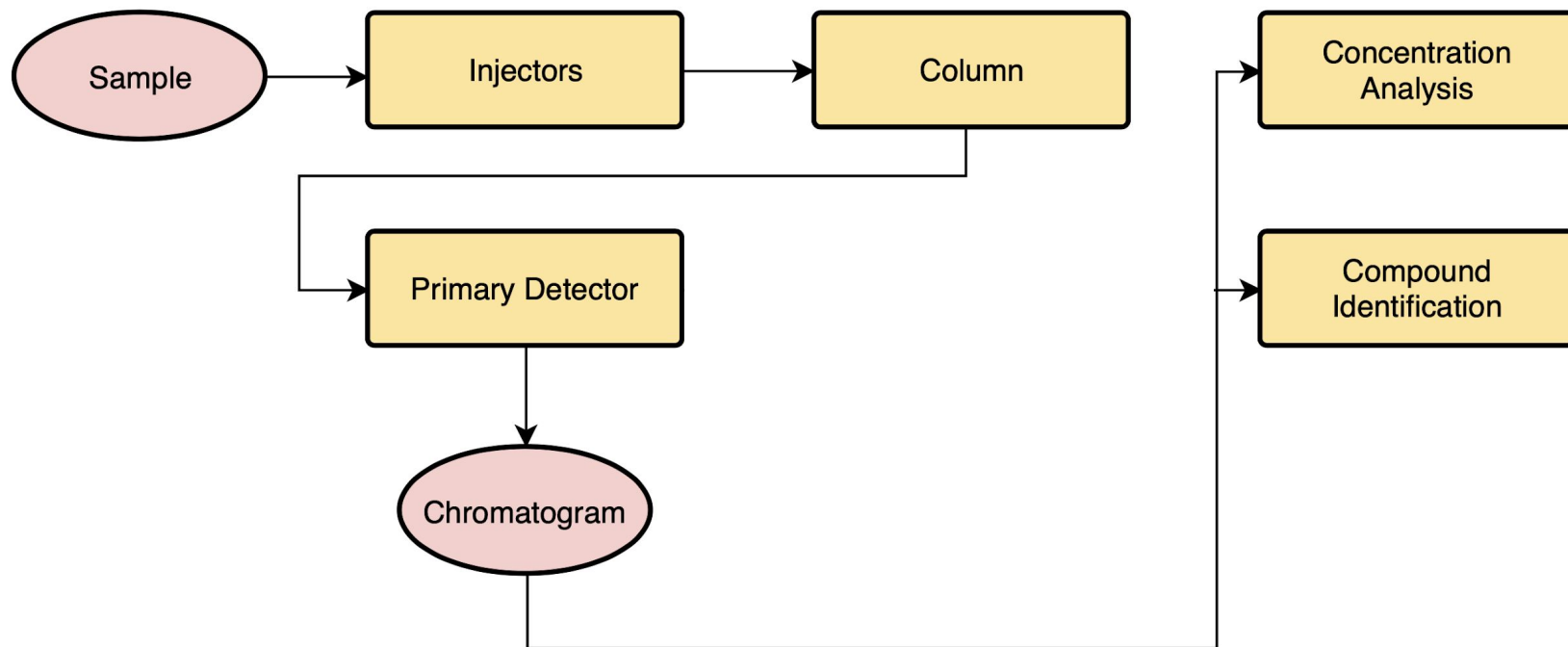Figure 8: TSPP(Ni^{2+}) w/ acetonitrile

Figure 9: TSPP(Ni^{2+}) change

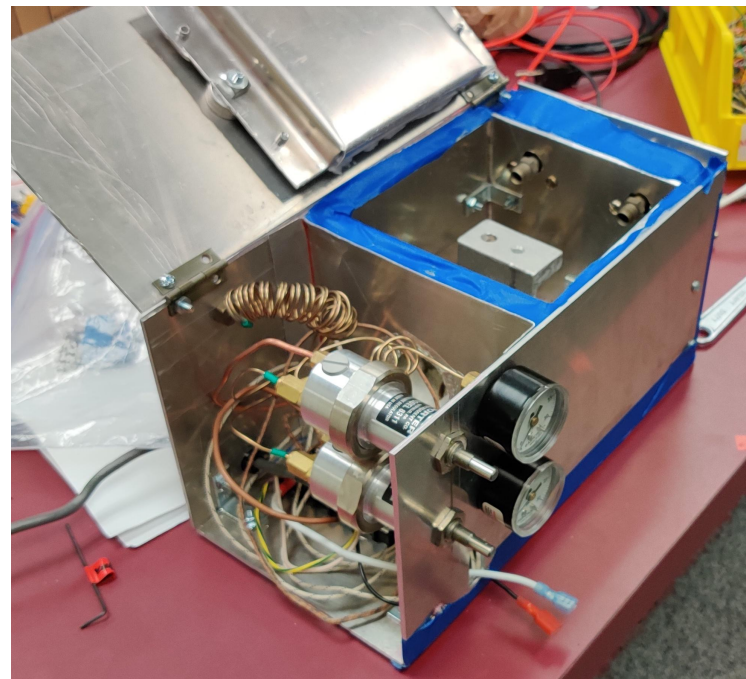Unfortunately, this work can not be continued without lab access.

System and Components

Previous Prototype

- Original design and fabrication
- Functional
  - Heating
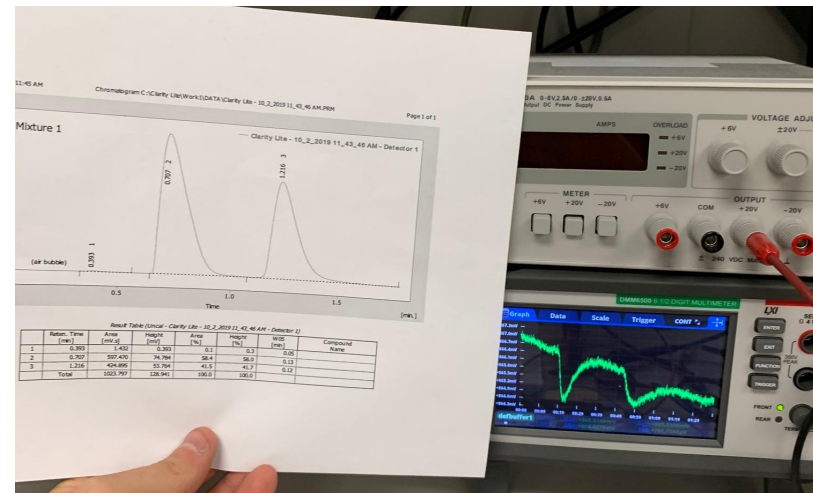 - Insulation
  - Gas flow control
  - Configurable detector
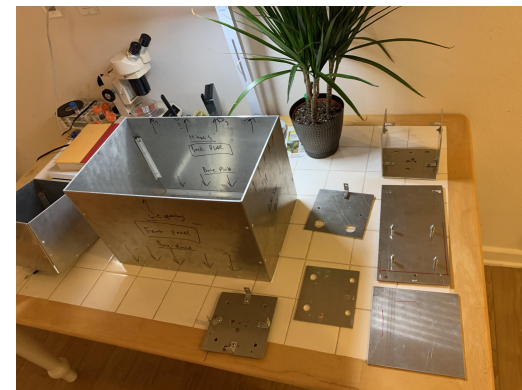
Previous Chromatograms

Single Diode and Nitrogen

Single Diode and Helium

New Prototype (In Progress)

- Much larger than first prototype
  - Supports a larger ¼" silica separatory column
- Better oven temperature gradient
  - Will not overheat parts of the column
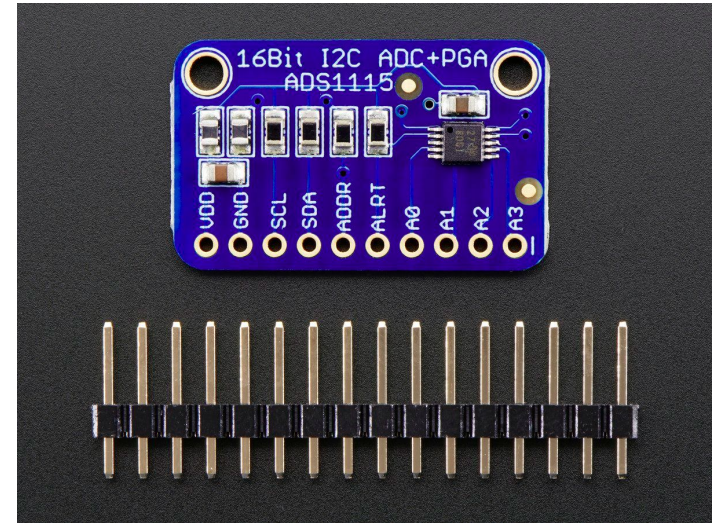- Not yet complete, but work will continue
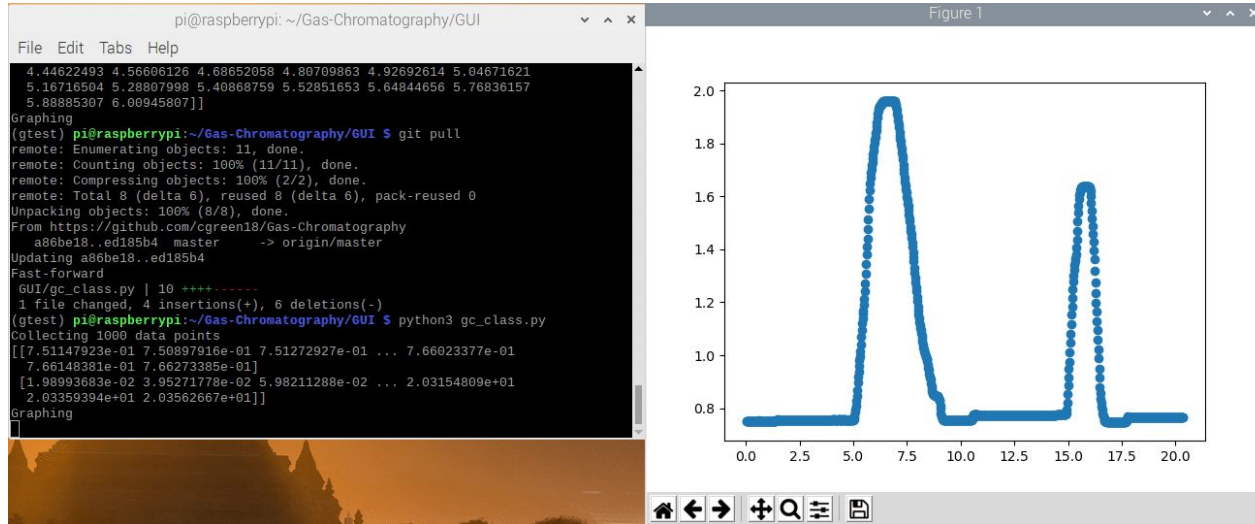
Analog to Digital Conversion

ADS1115

- 16-bit precision analog voltage measurement
- I^{2}C Communication
- Raspberry Pi Library

https://cdn-shop.adafruit.com/1200x900/1085-03.jpg

User Interface and GUI

- Python class inherits wxPython and maintains internal variables of previous runs
- Can perform operations on data (e.g. average)

Full Stack

Fresh Raspberry Pi

Install and
Configure scripts

Reboot and Autorun
on startup

User Interface and GUI

*Scores are out of 5

Design Criteria	Weight (%)	C		Java		Python				
		C#	Visual Basic	AWT	Swing	Qt	TkInter	Pygame	PyGtk	wxPython
Cross-platform compatability	40	1 0.4	1 0.4	3 1.2	3 1.2	5 2	5 2	1 0.4	4 1.6	5 2
Compilable	5	5 0.25	5 0.25	5 0.25	5 0.25	2 0.1	2 0.1	1 0.05	5 0.25	3 0.15
Speed	20	5 1	4 0.8	1 0.2	1 0.2	2 0.4	2 0.4	2 0.4	3 0.6	3 0.6
Codability	10	3 0.3	3 0.3	2 0.2	2 0.2	4 0.4	4 0.4	5 0.5	3 0.3	3 0.3
Range of abilities	25	5 1.25	5 1.25	4 1	4 1	4 1	4 1	3 0.75	5 1.25	5 1.25
Total:		3.2	3	2.85	2.85	3.9	3.9	2.1	4	4.3

- wxPython
- Raspberry Pi
- Interface with Arduino Pro Micro via GPIO for analog voltage measurements

Development Plan

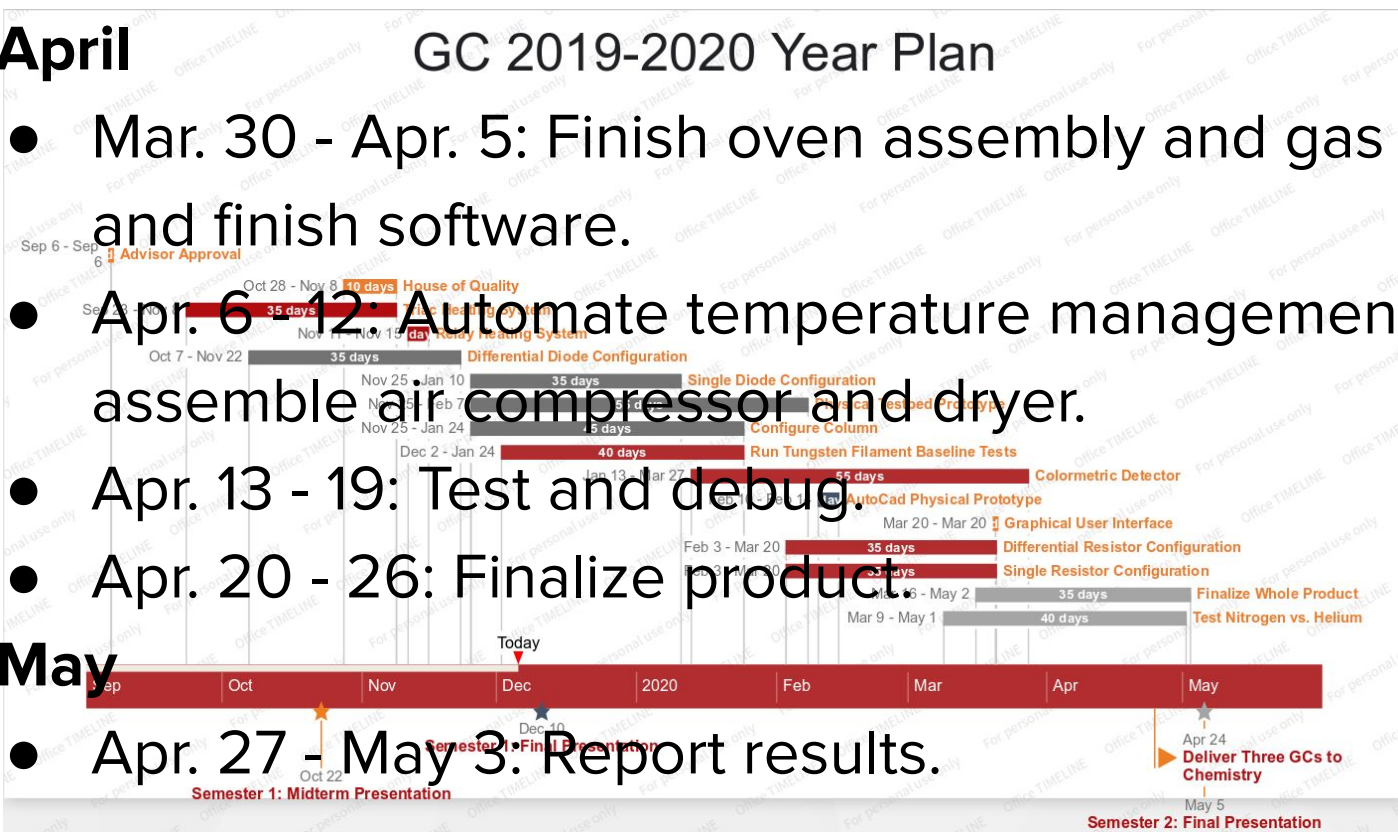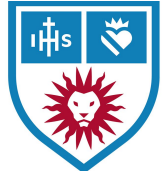**April**

- Mar. 30 - Apr. 5: Finish oven assembly and gas routing and finish software.

- Apr. 6 - 12: Automate temperature management and assemble air compressor and dryer.

- Apr. 13 - 19: Test and debug.

- Apr. 20 - 26: Finalize product.

May

- Apr. 27 - May 3: Report results.

GC 2019-2020 Year Plan

Looking Forward

Date	Objective
Mar. 30 - Apr. 5	Finish oven assembly and gas routing and finish software
Apr. 6 - 12	Automate temperature management and assemble air compressor and dryer
Apr. 13 - 19	Test and debug
Apr. 20 - 26	Finalize product
Apr. 27 - May 3	Report results

References

K. Rakow, N. & Suslick. A colorimetric sensor array for odour visualization. Nature, 406:710–713, August 2000.

M. Jones. A simple-to-build thermal-conductivity gc detector. Journal of Chemical Education, 71:995–996, November 1994.

Objectives

- **Simple**: Student can operate

- **Accurate**: Detects impurities $\geq 5\%$ of substance

- **Carrier gas**: Air > nitrogen > helium

- **Safe**

- **Durable and serviceable**: ≥ 2 weeks uptime / service

- **Economic**: $\leq \$500$/instrument

- **Documentation**