Gas Chromatography

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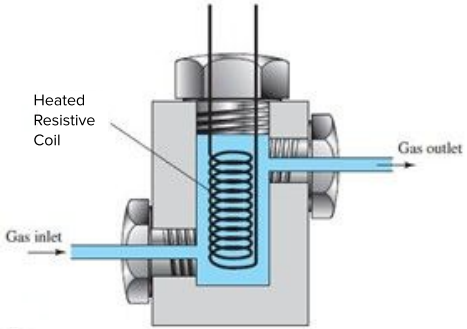
ELEC 402: Senior Project
Dr. Asghari

Final Presentation

- Spring Semester
  - Update on detector and carrier gasses
  - Colorimetric array
  - Current prototype
  - GUI, ADC, and software
- Demonstrations
  - Hardware + Software
- Future

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

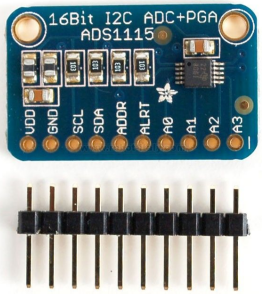
Updated Detector and Carrier Gas Selections

- 1N4148 Diode
- Dried air (32 @ 125C)

(1)

Updated Configuration Selection

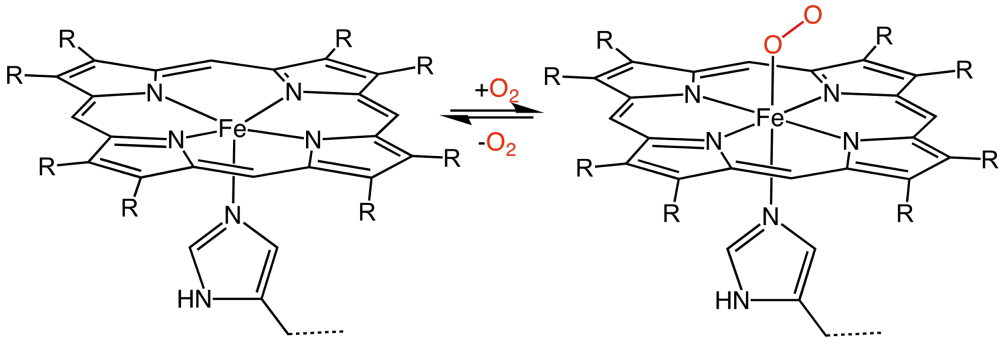
- Single-ended ADS1115
  - 16-bit precision analog voltage measurement
  - I^2C Communication
  - Raspberry Pi Library



(2)

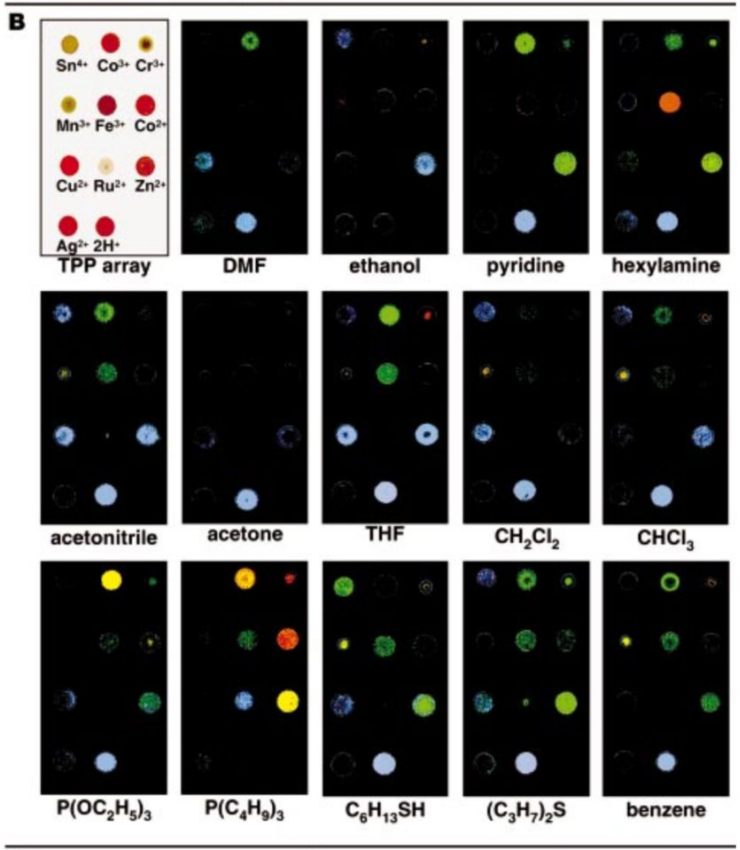
(1) https://www.digikey.com/product-detail/en/on-semiconductor/1N4148TR/1N4148FSCT-ND/9356376
(2) http://www.robot-italy.com/media/catalog/product/cache/4/image/d43192dcd82ea942982b4b1d2a6e2479/w/i/window.jpeg

Reminder: Colorimetric Array Detector



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

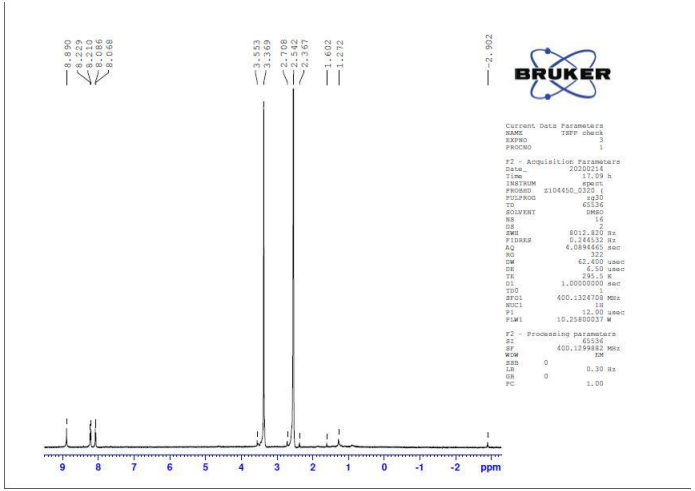
Oxyhemoglobin
- Bright red
- Fe^{3+} oxidation state
- Oxygen ligand

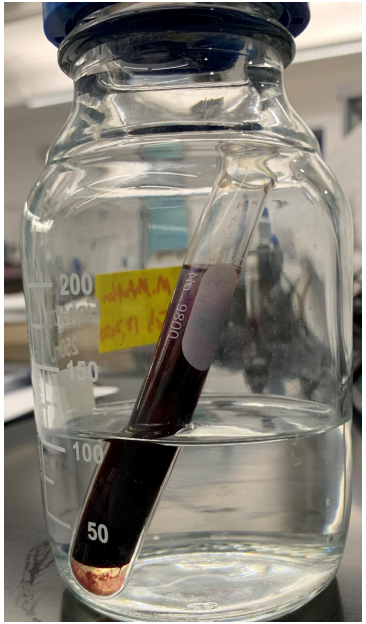


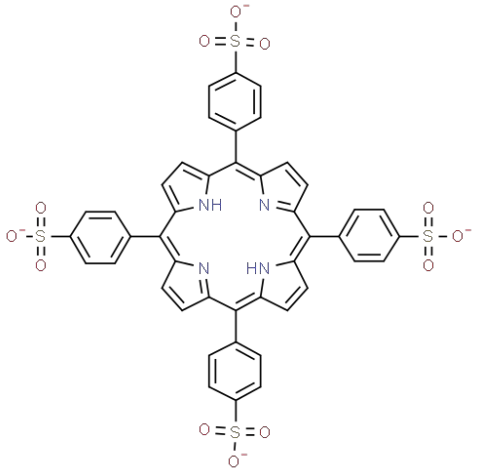
K. Rakow, N. & Suslick. A colorimetric sensor array for odour visualization. Nature, 406:710–713, August 2000.

Progress: Colorimetric Array Detector

- **Innovation**: Sulfonate the metalloporphyrin compounds so they can be dissolved in water.
  - Avoids chloroform, dimethyl sulphoxide (DMSO), and dichloromethane (DCM)

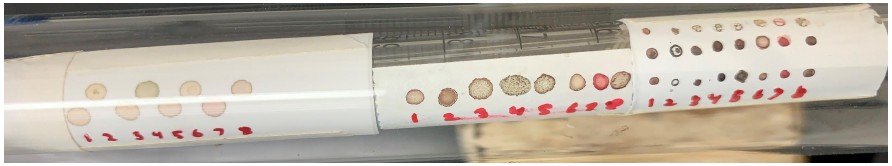






5,10,15,20-tetrakis(4-sulfonatophenyl)porphyrin
(Source: ChemSpider)

Testing the Colorimetric Array Detector



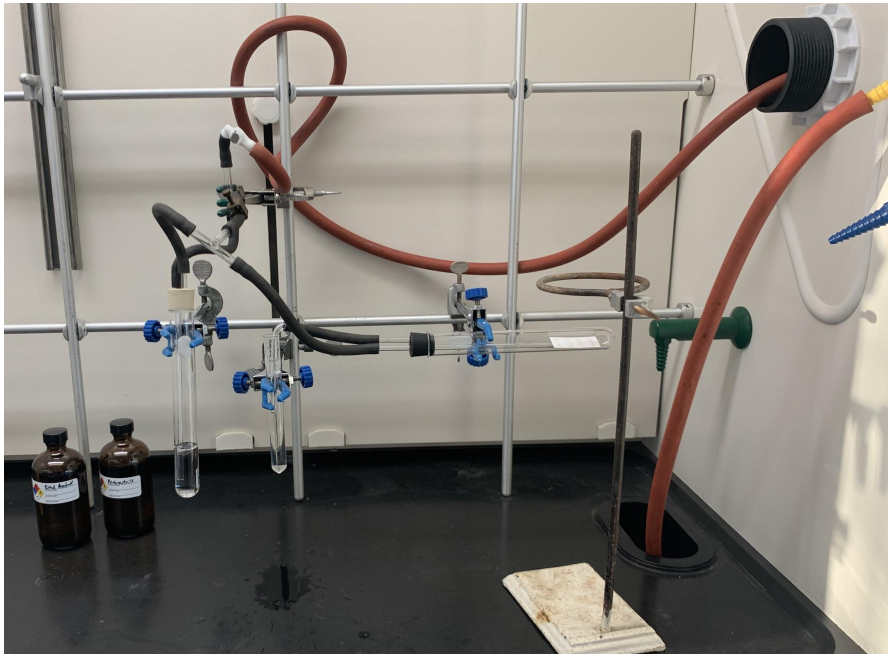


Figure 1: TSPP(Co^{2+}) initial

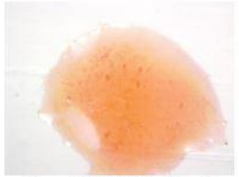


Figure 2: TSPP(Co^{2+}) w/ acetonitrile

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

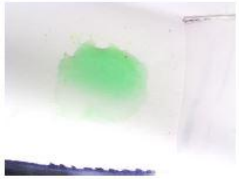


Figure 7: TSPP(Ni^{2+}) initial

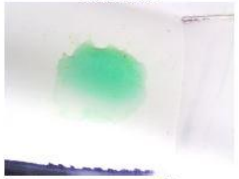
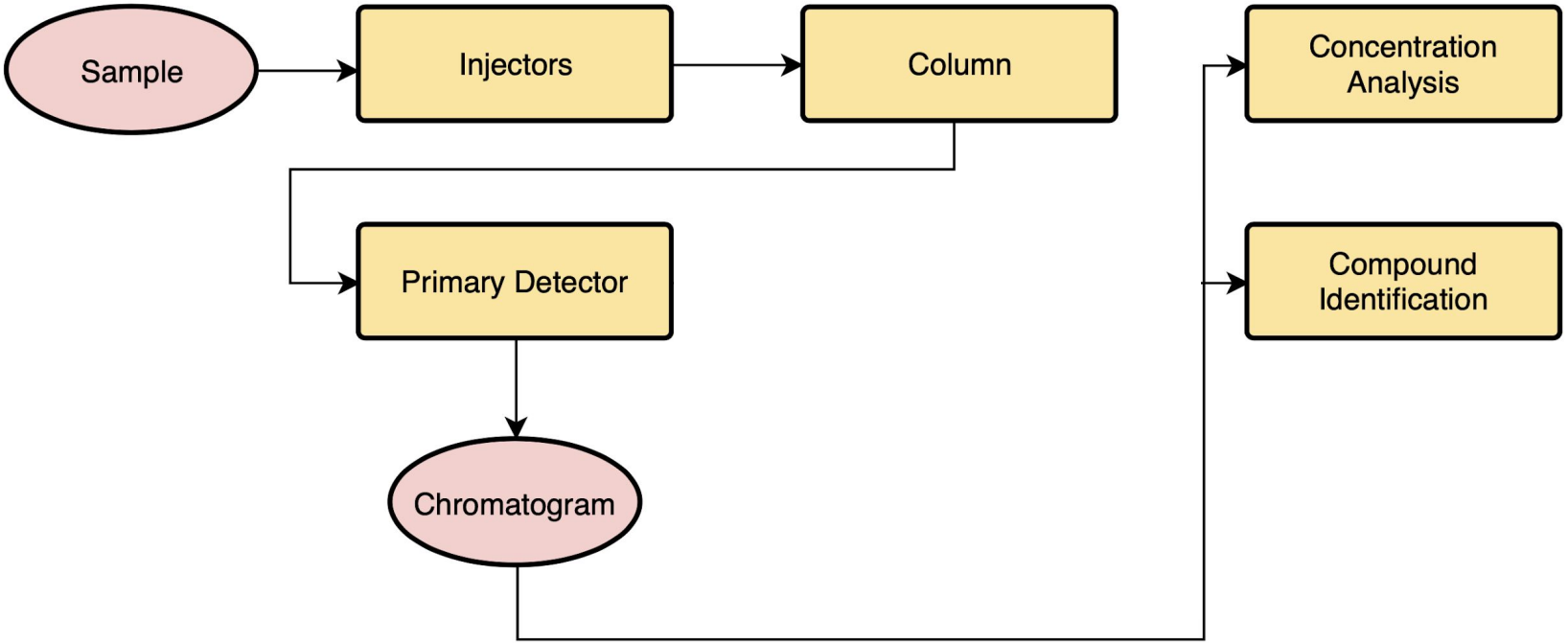


Figure 8: TSPP(Ni^{2+}) w/ acetonitrile

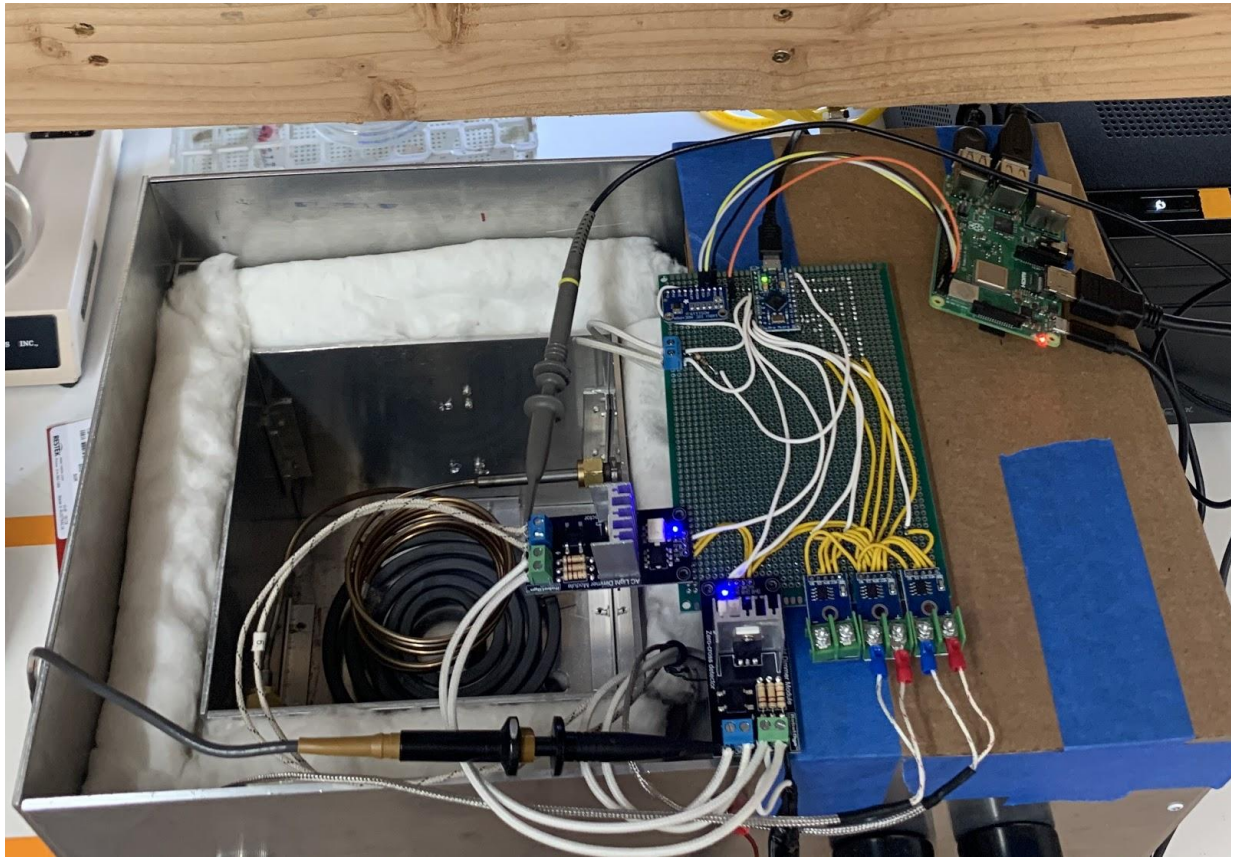
Figure 9: TSPP(Ni^{2+}) change

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

System and Components



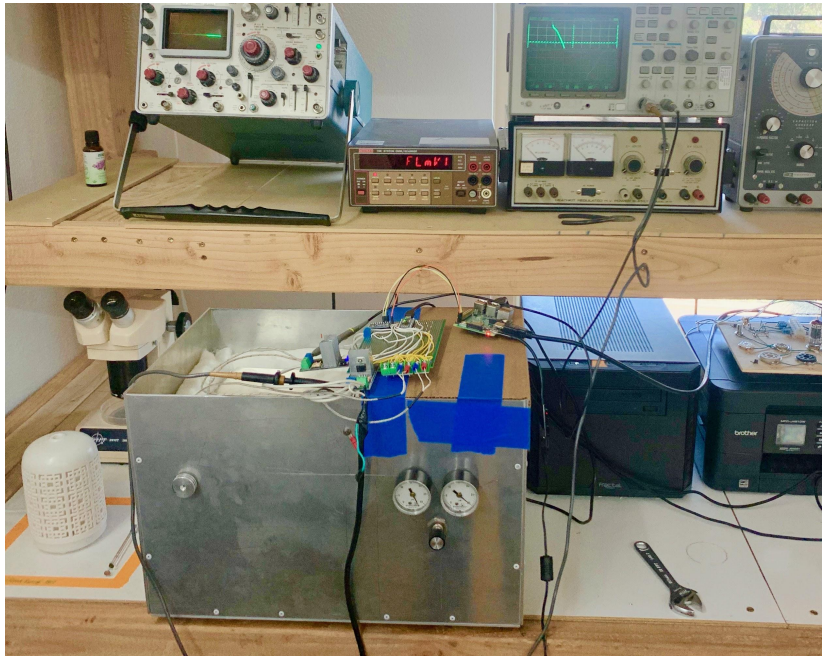
Current Prototype



Review of Hardware Features

Single Diode and Compressed Air

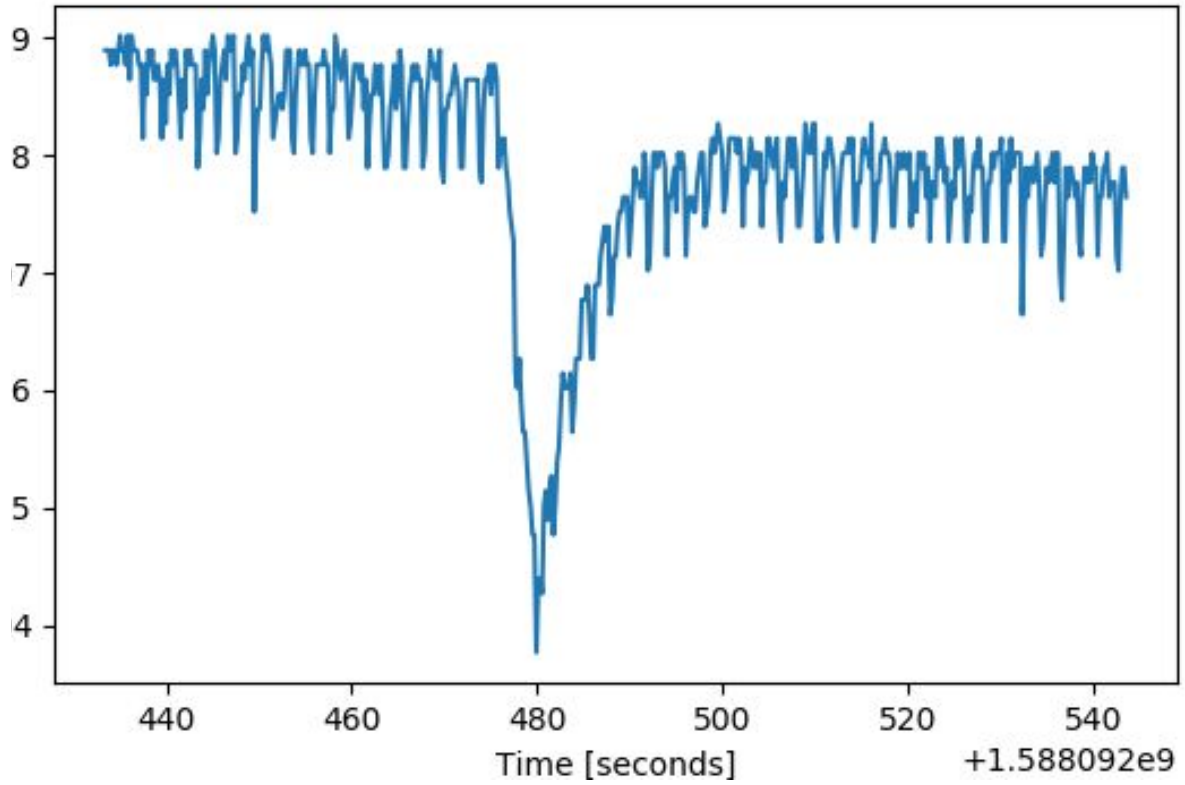
- TRIAC controlled temperature
  - Stability
  - Overtemperature protection
  - “Set and forget”
- Diode detector
  - Tested to work in helium
  - Compressed air is not as good
    - Low thermal conductivity difference between air and sample
- Carrier flow control
  - Regulated in two places
  - Adjustable on instrument
  - Column head pressure gauge
  - Gas inlet pressure guage



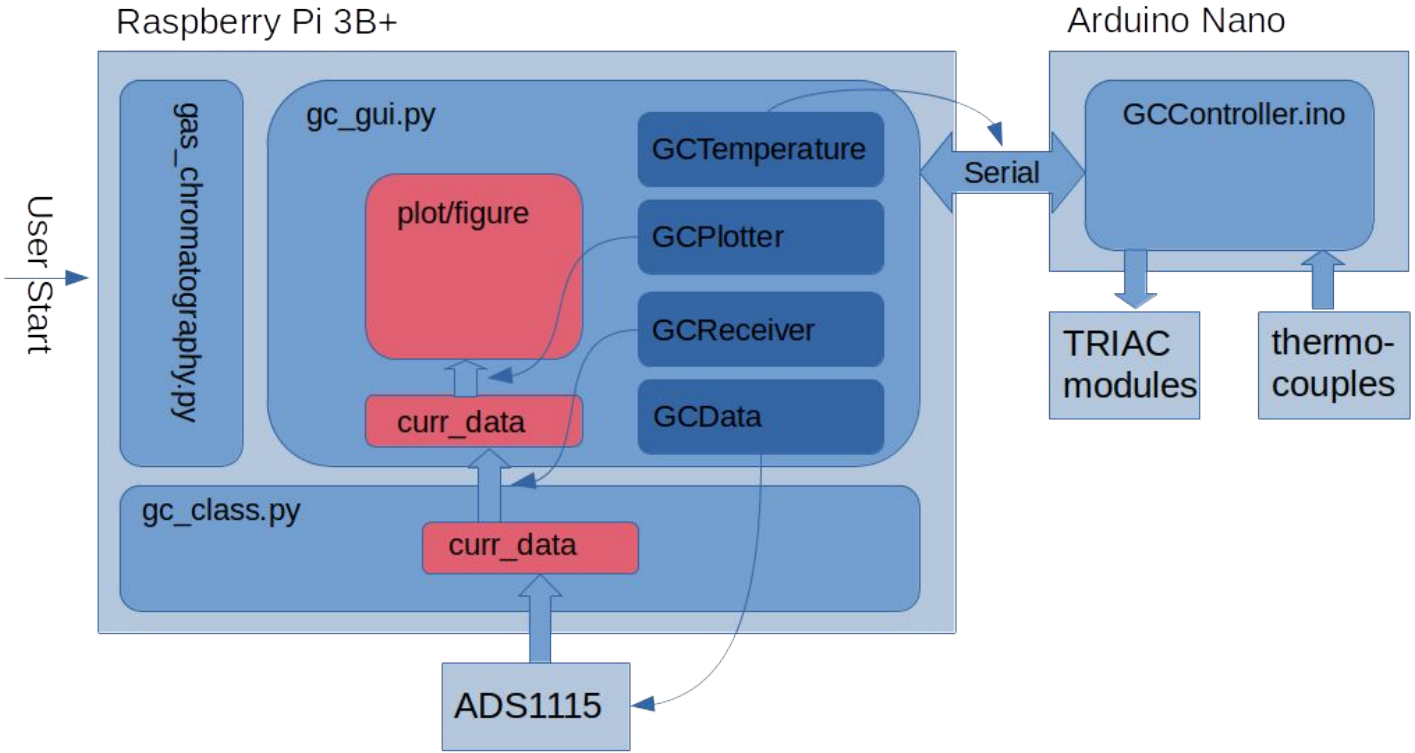
Results / Current Chromatograms

Results / Current Chromatograms

Exported as PNG from software utility

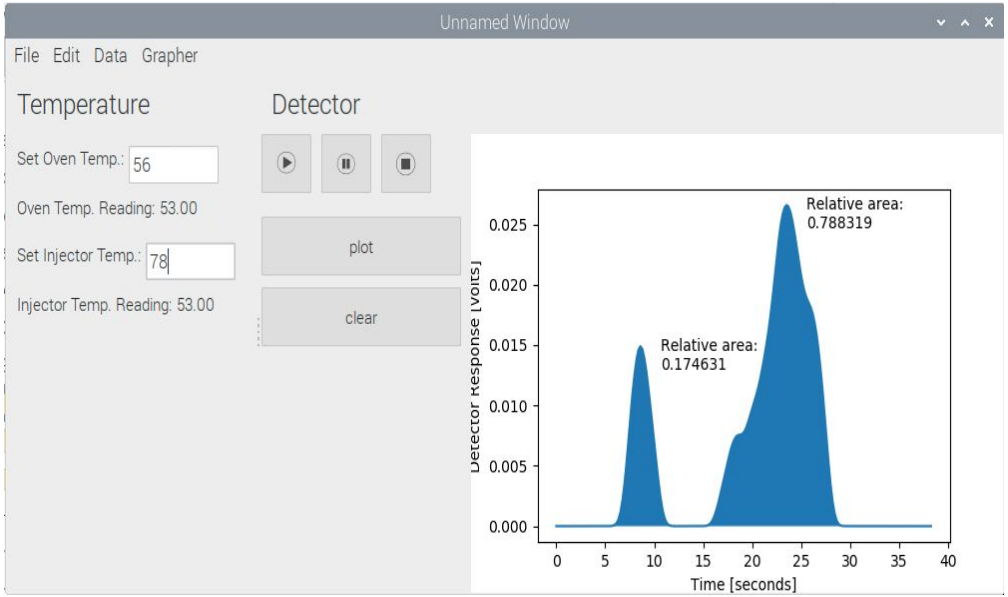


User Interface and GUI Design



GUI Example

- Separate YAML configuration file for default settings.
- Threaded data collection and live plotting.
- Math operations
  - Integrate data (voltage)
  - Normalize data (voltage)
  - Clean the time axis
  - Apply a low pass filter
  - Calculate relative area of peaks
- Fill under data on plot.
- Threaded temperature feedback text box updating.
- Ability to set oven and injector temperatures.
- Save current session as ".gc". file type (JSON data structure)
- Save current plot as JPEG or PNG image.
- Open previous session files (".gc") into the current window.



Full Stack

Fresh Raspberry Pi

Install and Configure scripts

Reboot and Autorun on startup

```
echo "Install script to set up a new Raspberry Pi to collect gc data and run the gc suite upon startup."
echo "Install instructions given at https://github.com/cgreen18/Gas-Chromatography/tree/master/Installation"
echo "Tested on Raspberry Pi Model 3B+ w/ Raspbian 10 Buster"
echo "20 February 2020 Conor Green"
```

```
jam@jamonicy: ~/Documents/Capstone/Gas-Chromatography/Installation
File  Edit  View  Search  Terminal  Help
./install.sh: line 53: `echo "Building wxPython. Will take a long time (~1-2 hrs)"'
jam@jamonicy:~/Documents/Capstone/Gas-Chromatography/Installation$ sudo nano ./install.sh
jam@jamonicy:~/Documents/Capstone/Gas-Chromatography/Installation$ sudo ./install.sh
Install script to set up a new Raspberry Pi to collect gc data and run the gc suite upon startup.
Install instructions given at https://github.com/cgreen18/Gas-Chromatography/tree/master/Installation
Tested on Raspberry Pi Model 3B+ w/ Raspbian 10 Buster
20 February 2020 Conor Green

Updating repos
Hit:1 https://download.virtualbox.org/virtualbox/debian bionic InRelease
Hit:2 https://packages.microsoft.com/repos/vscode stable InRelease
Hit:4 http://security.ubuntu.com/ubuntu bionic-security InRelease
Hit:5 http://ppa.launchpad.net/gezakovacs/ppa/ubuntu bionic InRelease
Hit:6 http://us.archive.ubuntu.com/ubuntu bionic InRelease
Hit:7 http://us.archive.ubuntu.com/ubuntu bionic-updates InRelease
Hit:8 http://archive.canonical.com/ubuntu bionic InRelease
Hit:9 http://us.archive.ubuntu.com/ubuntu bionic-backports InRelease
Hit:3 https://packagecloud.io/AtomEditor/atom/any any InRelease
Hit:10 http://us.archive.ubuntu.com/ubuntu bionic-proposed InRelease
Ign:11 http://ppa.launchpad.net/wxformbuilder/release/ubuntu bionic InRelease
Ign:12 http://ppa.launchpad.net/wxformbuilder/wxwidgets/ubuntu bionic InRelease
Err:13 http://ppa.launchpad.net/wxformbuilder/release/ubuntu bionic Release
  404  Not Found [IP: 91.189.95.83 80]
Err:14 http://ppa.launchpad.net/wxformbuilder/wxwidgets/ubuntu bionic Release
```

```
pip install -U pip

# Install dependencies

apt-get update
pip install -U six wheel setuptools
apt-get install build-essential tk-dev libncurses5-dev libncursesw5-dev libreadline6-dev libdb5.3-dev libgdbm-dev libsqlite3-dev
apt-get install dpkg-dev build-essential libjpeg-dev libtiff-dev libsdl1.2-dev libgstreamer-plugins-base0.10-dev libnotify-dev
```

User Interface and GUI

		*Scores are out of 5																	
		C				**Java**				**Python**									
Design Criteria	**Weight (%)**	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

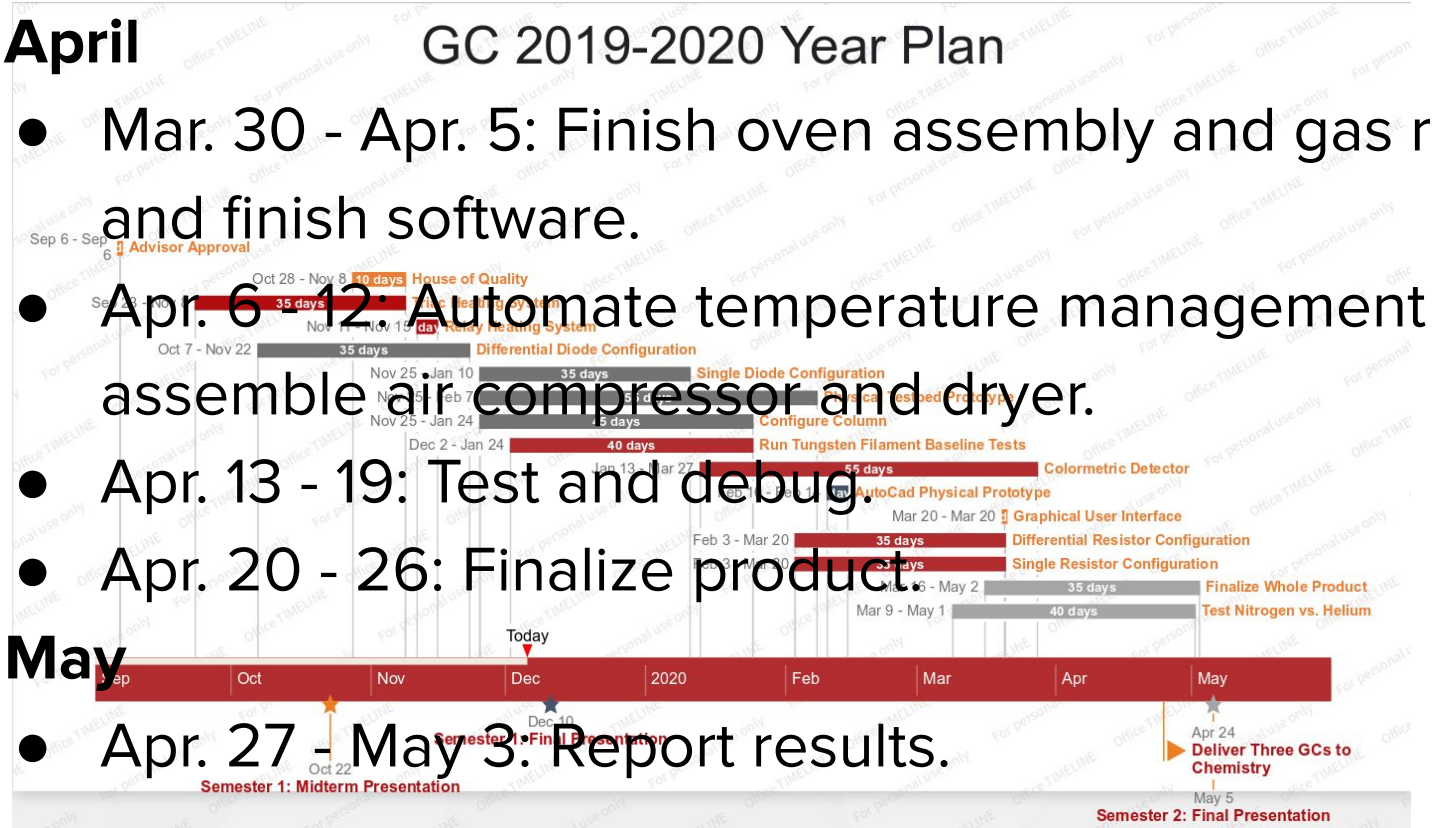
Development Plan

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.



Looking Forward

Hardware

- Weld together walls of instrument shell.
- Better gas flow rate control.
- Variety of carrier gases to determine cheapest that yields sufficient signal-to-noise ratio.
- Place electronics inside the shell for portability and application to educational setting.

Software

- Ability for user to modify settings such as graph color and markers. This functionality was began with the *GCConfigPopup* class but was left unconnected in the final product (version 4).
- Cutting/removing sections of data via a GUI tool.
- GUI tool for zooming and moving around the plot.
- Window/menu functionality to modify settings pertaining to the hardware (e.g. gas flow rate).
- Proper error handling through custom exception classes: the code should throw informational exceptions for the programmer/user during new code testing or hardware set up. For example, currently nothing is done when no open serial ports are found (most likely unconnected cord) and this causes errors further down execution.
- Printing to printer.

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 ≥ 5% of substance
- **Carrier gas**: Air > nitrogen > helium
- **Safe**
- **Durable and serviceable**: ≥ 2 weeks uptime / service
- **Economic**: ≤ $500/instrument
- **Documentation**