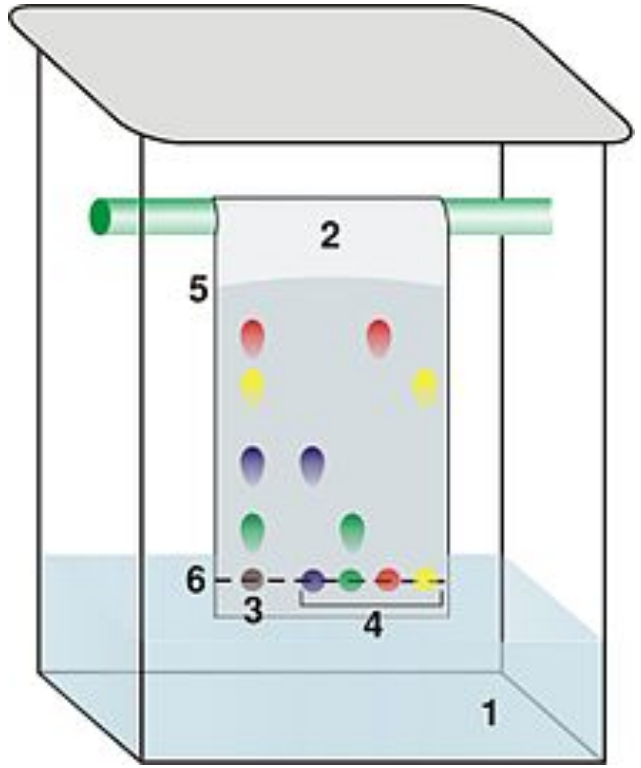
Gas Chromatography

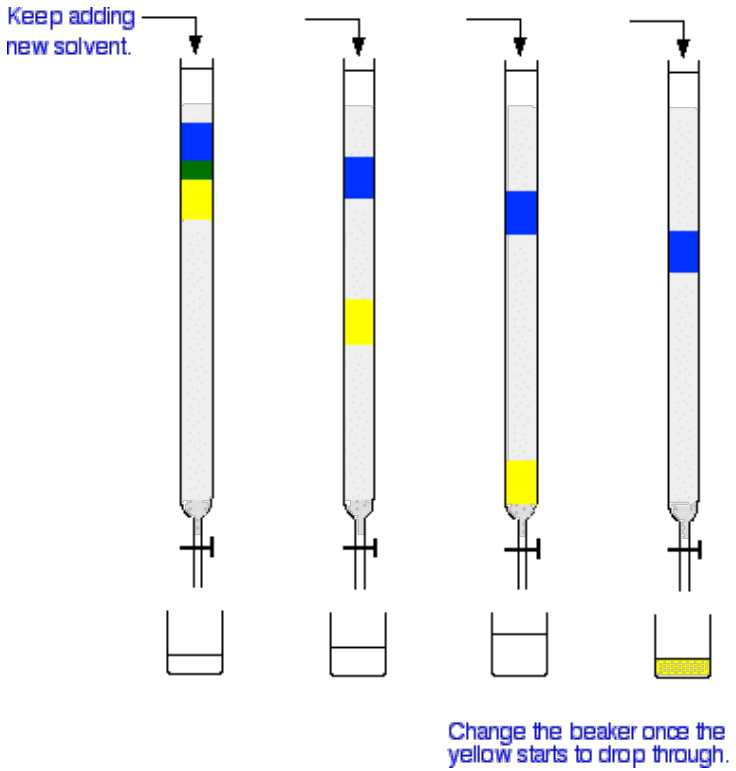
Several Types of Chromatography

- Chromatography can separate a complex mixture into individual components
  - **Qualitative analysis** Which components are present in the sample?
  - **Quantitative analysis** How much of each compound is present?
- 3 main types in teaching labs
  - Thin Layer Chromatography - simple qualitative analysis of solutions
  - Column Chromatography - crude TLC with larger volume
  - Gas Chromatography – useful for liquids with low boiling points



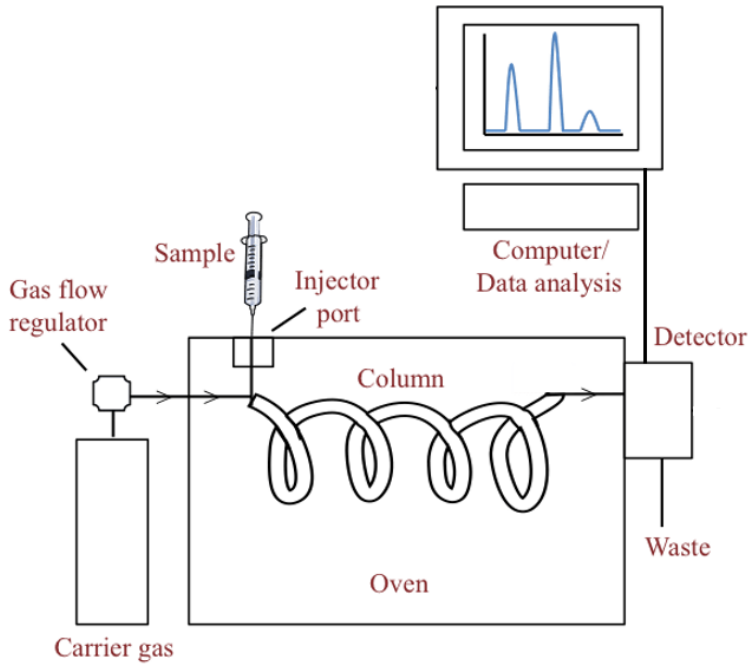
Thin Layer Chromatography

- Often the first type of chromatography introduced in organic chemistry
- Useful for colored or UV-absorptive compounds
- Students routinely use TLC throughout the semester
- Allows qualitative analysis based on differences in polarity



Column Chromatography

- TLC on a larger scale
- Frequently used in research labs
- Teaching labs usually use small (microgram) samples
- Theoretically can be used for qualitative and quantitative analysis
  - Difficult in practice, especially for a teaching lab
- Not very useful in teaching labs
  - Limited by time constraints and the range of student abilities



Gas Chromatography

- Allows analysis of solutions with volatile components
- Can be used for both qualitative and quantitative analysis
- In lab, one machine is operated by the TA
  - Instrument is delicate and very expensive
  - Requires use of microliter syringes
- The usefulness of the data is limited by the skill of the TA
- Research instruments use auto-samplers

The Ideal Teaching Instrument

Facilitates guided yet independent learning

Demonstrates proper use of the instrument and resulting data

Is accurate with minimal maintenance and under adverse conditions

Is durable and cost-effective

A New Gas Chromatograph

Facilitates guided yet independent learning

- Supports autoinjection for repeatable results
- Does not require expensive and fragile microliter syringes
- Is affordable enough to be in every student's workstation

Demonstrates proper use of the instrument and resulting data

- Browser based data management and digital lab manuals

Is accurate with minimal maintenance and under adverse conditions

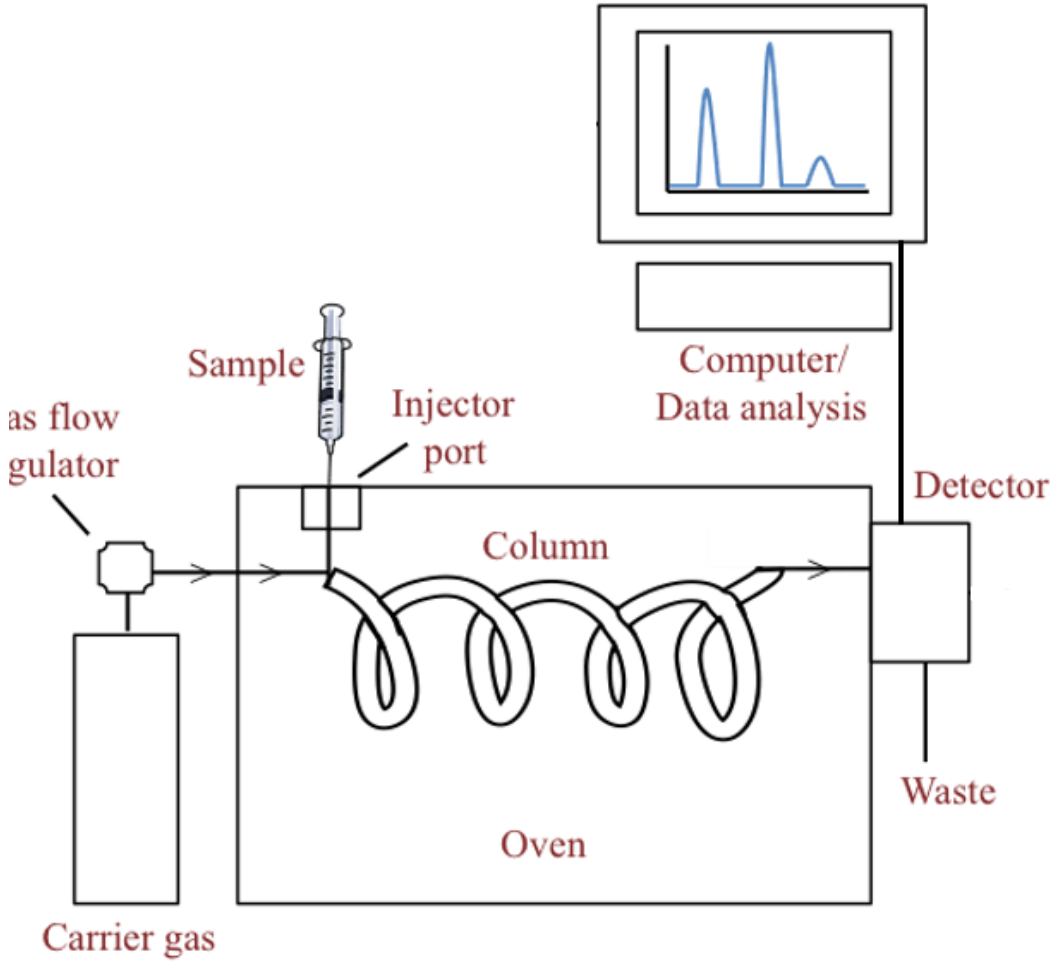
- MIL-PRF rated small signal diodes are hermetically sealed and operational up to 200C

Is durable and cost-effective

- Modular design makes it trivial to replace both reference and sensing diodes

Inside a Gas Chromatograph

- Three major systems
  - Sample injectors
  - Column
  - Detector
- Scope of proposed project
  - Venturi autoinjection
  - Diode detectors
  - Web GUI and data collection terminal



Brief Comparison of Designs

Proposed Project

- Small signal diode
  - Hermetically sealed package is extremely durable
  - Not tied to inert gasses
- Automatic temp control
  - Prevents “overshooting”
  - Protect components
- Venturi autoinjection
  - Makes use of microcapillary tubes
  - Rugged and cheap

Gow-Mac Series 350 TCD

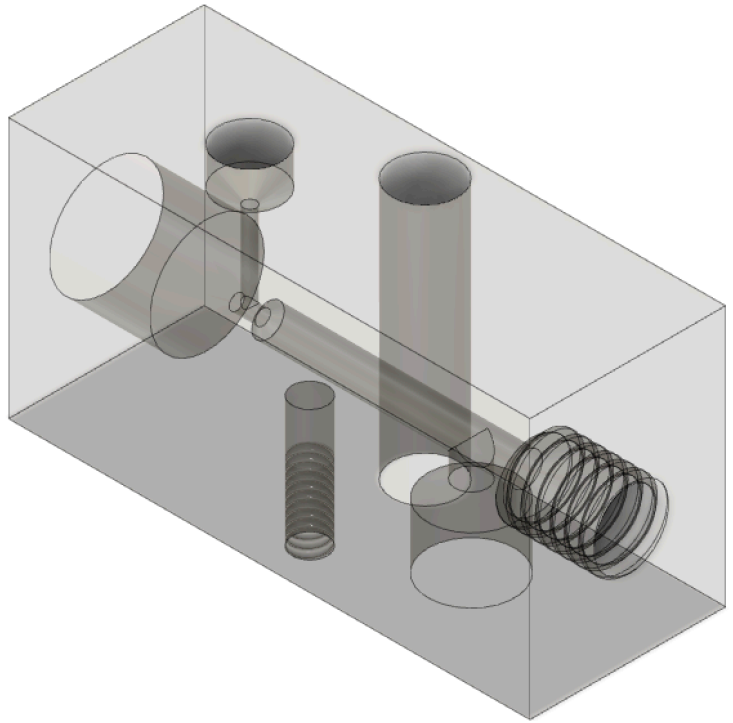
- Tungsten thin-filament RTD
  - Requires inert carrier gas
  - Intolerant of misuse
  - Very expensive and fragile
- Manual TRIAC temp control
  - Cannot “set and forget”
  - Poor repeatability
- Manual injection
  - Poor repeatability
  - Requires fragile syringes

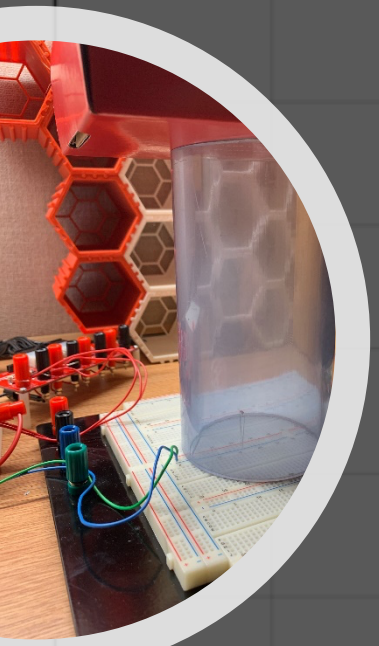
A Student-Friendly Autoinjector

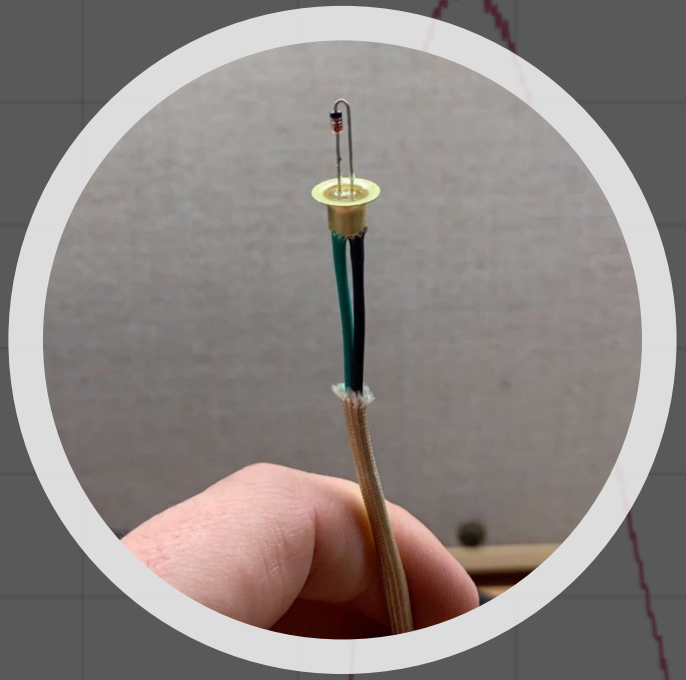
Venturi Autoinjector

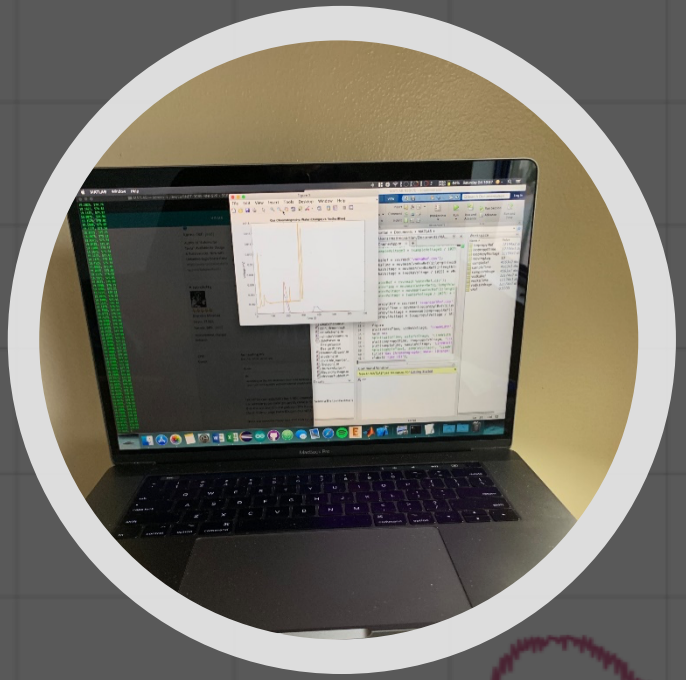
- Take advantage of the steady-state flow in the column
- Requires use of air as a carrier
- Designed to be manufacturable
  - All angles can be put in by standard drill bits
  - Linear internal passageways can be directly drilled

Heated Injection Block









Current Progress

- Functional diode detector
- Instrumentation amplifier
- Total cost of prototype: $200 (column excluded)
- Gow-Mac Series 350 list price (new): $9000

Design Methodology

Subsystems and circuits

- Two diodes: sensor and reference
  - Zero-adjust potentiometer on reference channel
- Adjustable current source (0.1 – 100 mA)
- Instrumental (differential) amplifier
  - Gain of approximately 10,000
- Signal filtering
  - 2^{nd} order active RC filter on each input

Data recording

- Noise profiling with oscilloscope
- Graph obtained with Vernier voltage probe

Problems to Solve

Amplifier performance

- Voltage drift over time (need to zero before every test injection)
- High gain (~10,000) is difficult to stabilize

Noise suppression

- Microvolt noise is difficult to suppress

Diode sensitivity

- Diode is relatively large
- Glass package is a good insulator
  - Poor slew rate

Difficulty detecting samples without helium

- Very faint in nitrogen
- Undetectable in air

Venturi Autoinjector

- Manufacturing and materials
- Optimal venturi dimensions

Final Project Goals

Develop	the diode detector gas chromatograph into a affordable and flexible instrument
Implement	a TRIAC based programmable temperature controller
Test and fabricate	a usable venturi autoinjector to support microcapillary sampling
Design and implement	a web based GUI to manage data and display digital lab manuals
Collaborate	with the Dr. Robert Senter and the LMU Department of Chemistry to set up each lab station with this instrument