Low-cost Gas Chromatography – Column and Oven

Angelo Carlo E. Arceta
IV BS Ch-ACS

Mentors: Dr. Armando Guidote
Engr. Tristan Calasanz
Mr. Cally Enaje
Introduction: Gas Chromatography

• Technique that is used to separate, qualify and/or quantify components in a sample
• Widely used in fields of research, medicine, education and others

• Basic components: injection port, a flow regulator, a column, an oven and a detector
Introduction: Gas Chromatographs

- Many different types of GC’s with variations in the types of detectors used (FID, TCD, ECD, NPD, etc.)
- GC’s vary in sensitivity and selectivity
- VERY EXPENSIVE
- Brand new educational TCD GC: $4999 or P204,459.10 (GenTechScientific)
### Prices of GC’s Today

#### Facts

<table>
<thead>
<tr>
<th>Type</th>
<th>Price Range in USD</th>
<th>Price Range in Php</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brand New</td>
<td>8,495-19,095</td>
<td>339,800-763,800</td>
</tr>
<tr>
<td>Brand New</td>
<td>3,900-7,500</td>
<td>116,000-300,000</td>
</tr>
</tbody>
</table>

Philippine context…

• Not all universities have a GC

• Most lack funding for teachers and equipment

• CHED requirement: In 3 years, all schools that offer a Chemistry degree must have a GC
Significance/Motivation

- Education

- Social Responsibility

- Pioneering Analytical Chemistry Instrument Manufacturing Industry
Objectives

- To clean and pack stainless steel columns
- To test the efficiency of powdered detergents as packing material
- To design a low-cost version of a GC oven
- To create a database of local suppliers of electronic parts that will be used in the fabrication
- To share the findings of this study with the different schools offering a BS Chemistry degree
Limitations

- Stainless-steel columns have no specific way of determining whether the packing performed is already satisfactory
- Detergents today have an aroma
- The designed oven is only isothermal
- For qualitative analysis only

Methodology

**PROTOTYPE**
Design and Construction of Oven heater circuit

**COLUMNS**
Cleaning & Self-packing of used stainless steel column
Connection to injector port and detector

**FABRICATION**
Complete Final circuit design for PCB etching and ASSEMBLY of a new GC Casing

**DETECTOR**
Design and Construction of Detector c/o Mr. Cabrera

**RUNS**
Calibration of oven temperature and Optimization of parameters to obtain qualitative analysis

Purchasing of materials
Methodology

Column Cleaning and Packing

Washings
Acetone
Toluene
Detergent

Funnel
Column
Vacuum Set-up
Methodology

Column Cleaning
## Table 1.0 - Column Dimensions and Wt. of SP

<table>
<thead>
<tr>
<th>Column</th>
<th>Column Length</th>
<th>Inside Diameter (mm)</th>
<th>Obtained weight of SP (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>20.0 inches</td>
<td>2.3 0.3</td>
<td>2.1367 0.0001</td>
</tr>
<tr>
<td>B</td>
<td>173.0 cm</td>
<td>2.0 0.3</td>
<td>4.6003 0.0001</td>
</tr>
</tbody>
</table>
Methodology

Oven Prototype Circuit
Results

Oven Circuit Implemented
Oven Circuit Etching for PCB
Methodology

Instruments Used for the Casing

1. Drill Press
2. Band Saw
3. Metal Sheet Cutter
4. Bending Machine
5. Milling Machine
6. Lathe Machine
7. Electric Drilling Machine
8. Grinding Machine
Results
Table 2.0 – Database of Spare Parts for GC Fabrication

<table>
<thead>
<tr>
<th>GC Part</th>
<th>Specific Description</th>
<th>Store</th>
<th>Location</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oven circuit</td>
<td>Heater</td>
<td>Deeco</td>
<td>Raon, Quiapo</td>
<td>90.00</td>
</tr>
<tr>
<td></td>
<td>Zero-Crossing Triac</td>
<td>Alexan</td>
<td>SM North</td>
<td>196.50</td>
</tr>
<tr>
<td></td>
<td>Triac</td>
<td>Alexan</td>
<td>SM North</td>
<td>170.75</td>
</tr>
<tr>
<td></td>
<td>Resistors, capacitors, fuse</td>
<td>Alexan</td>
<td>SM North</td>
<td>62.75</td>
</tr>
<tr>
<td></td>
<td>Transformer, ICs and wires</td>
<td>Alexan</td>
<td>SM North</td>
<td>197.00</td>
</tr>
<tr>
<td></td>
<td>Fiberglass wires</td>
<td>Unison Commercial</td>
<td>Avenida, Sta. Cruz</td>
<td>150.00</td>
</tr>
<tr>
<td></td>
<td>Thermostat and Thermometer</td>
<td>Tyson</td>
<td>T. Mapua, Sta. Cruz</td>
<td>1,500.00</td>
</tr>
<tr>
<td>Column</td>
<td>Tide powdered detergent</td>
<td>SM Hypermarket</td>
<td>SM North</td>
<td>91.75</td>
</tr>
<tr>
<td>Component</td>
<td>Supplier</td>
<td>Address</td>
<td>Price</td>
<td></td>
</tr>
<tr>
<td>---------------------------------</td>
<td>-------------------------</td>
<td>--------------------------</td>
<td>---------</td>
<td></td>
</tr>
<tr>
<td>Injector Port</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Plastic Syringes</td>
<td>CJ Medical Supply</td>
<td>Bambang, Sta. Cruz</td>
<td>10.00</td>
<td></td>
</tr>
<tr>
<td>Septum</td>
<td>CJ Medical Supply</td>
<td>Bambang, Sta. Cruz</td>
<td>30.00</td>
<td></td>
</tr>
<tr>
<td>Casing</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Aluminum flat sheet</td>
<td>Novarise East</td>
<td>T. Morato, Quezon City</td>
<td>2,754.00</td>
<td></td>
</tr>
<tr>
<td>Ceramic fiber blanket</td>
<td>Unison Commercial</td>
<td>Avenida, Sta. Cruz</td>
<td>1,080.00</td>
<td></td>
</tr>
<tr>
<td>Fiberglass tubings</td>
<td>Unison Commercial</td>
<td>Avenida, Sta. Cruz</td>
<td>55.00</td>
<td></td>
</tr>
<tr>
<td>Nuts, bolts, handle, hinges</td>
<td>Tee To Suy</td>
<td>Recto, Sta. Cruz</td>
<td>470.00</td>
<td></td>
</tr>
<tr>
<td>Flare nuts and adapters</td>
<td>Manheng</td>
<td>T. Mapua, Sta. Cruz</td>
<td>610.00</td>
<td></td>
</tr>
<tr>
<td>Brass coupling</td>
<td>Manheng</td>
<td>T. Mapua, Sta. Cruz</td>
<td>90.00</td>
<td></td>
</tr>
<tr>
<td>Primer Spray paints</td>
<td>Chun Muk Screw</td>
<td>T. Mapua, Sta. Cruz</td>
<td>85.00</td>
<td></td>
</tr>
<tr>
<td>High-temp Spray paints</td>
<td>Tee To Suy</td>
<td>Recto, Sta. Cruz</td>
<td>165.00</td>
<td></td>
</tr>
<tr>
<td><strong>TOTAL EXPENSES</strong></td>
<td></td>
<td></td>
<td><strong>7,807.75</strong></td>
<td></td>
</tr>
</tbody>
</table>
### Results

#### Table 3.0 – Labor and Equipment Use Costing

<table>
<thead>
<tr>
<th>GC Part</th>
<th>Specific Description</th>
<th>c/o</th>
<th>Particulars</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>Casing</td>
<td>Labor Cost</td>
<td>Mr. Enaje</td>
<td>52.15 hrs</td>
<td>7,913.00</td>
</tr>
<tr>
<td></td>
<td>Machine Operation Cost</td>
<td>Mr. Enaje</td>
<td>All the equipment</td>
<td>112.50</td>
</tr>
</tbody>
</table>

**TOTAL EXPENSES for LABOR and USE of Equipment** 8,025.74

**TOTAL EXPENSES (GC components w/o a detector)** Php15,833.49
### Results

#### Table 4.0 – Time to Reach Set Temperature

<table>
<thead>
<tr>
<th>Set Temp (°C)</th>
<th>Ave Time reached (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>43.46</td>
</tr>
<tr>
<td>100</td>
<td>184.31</td>
</tr>
<tr>
<td>150</td>
<td>561.97</td>
</tr>
<tr>
<td>200</td>
<td>1,615.85</td>
</tr>
</tbody>
</table>
## Results

### Table 5.0 – Calibration of Oven Temp

<table>
<thead>
<tr>
<th>Set Temp in Thermostat (°C)</th>
<th>Thermocouple Thermometer Reading (°C)</th>
<th>Oven Thermometer Reading (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Lowest</td>
<td>Highest</td>
</tr>
<tr>
<td>50</td>
<td>38.6</td>
<td>70.1</td>
</tr>
<tr>
<td>100</td>
<td>78.5</td>
<td>114.8</td>
</tr>
<tr>
<td>150</td>
<td>116.6</td>
<td>148.4</td>
</tr>
<tr>
<td>200</td>
<td>156.7</td>
<td>185.7</td>
</tr>
</tbody>
</table>
Temp Range: 78.5-114.8 ºC

Results

Temperature Range Over Time
Set Temp of 100 ºC
### Results

#### Comparison of Baselines (50 °C)

Two-sample t test with unequal variances

<table>
<thead>
<tr>
<th>Variable</th>
<th>Obs</th>
<th>Mean</th>
<th>Std. Err.</th>
<th>Std. Dev.</th>
<th>[95% Conf. Interval]</th>
</tr>
</thead>
<tbody>
<tr>
<td>tid~t50c</td>
<td>9220</td>
<td>146.3526</td>
<td>0.1364818</td>
<td>13.10509</td>
<td>146.0851 146.6201</td>
</tr>
<tr>
<td>ov2~t50c</td>
<td>9220</td>
<td>178.7134</td>
<td>0.1356247</td>
<td>13.0228</td>
<td>178.4476 178.9793</td>
</tr>
<tr>
<td>combined</td>
<td>18440</td>
<td>162.533</td>
<td>0.1531448</td>
<td>20.79614</td>
<td>162.2328 162.8332</td>
</tr>
</tbody>
</table>

- **diff** = mean(tideat50c) - mean(ov225at50c)
  - 
  - $t = -1.7 	imes 10^2$
- Satterthwaite's degrees of freedom = 18437.3

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Ho: diff = 0</td>
<td>Ha: diff &lt; 0</td>
</tr>
<tr>
<td>Pr(T &lt; t) = 0.0000</td>
<td>Pr(</td>
</tr>
<tr>
<td>Pr(T &gt; t) = 1.0000</td>
<td></td>
</tr>
</tbody>
</table>

Statistical Computations were performed using the software StataSE9
Results

Comparison of Baselines (100 °C)

<table>
<thead>
<tr>
<th>Variable</th>
<th>Obs</th>
<th>Mean</th>
<th>Std. Err.</th>
<th>Std. Dev.</th>
<th>[95% Conf. Interval]</th>
</tr>
</thead>
<tbody>
<tr>
<td>tid~100c</td>
<td>564</td>
<td>154.5922</td>
<td>.5429716</td>
<td>12.89486</td>
<td>153.5257  155.6587</td>
</tr>
<tr>
<td>ov2~100c</td>
<td>564</td>
<td>215.8688</td>
<td>.4167392</td>
<td>9.897009</td>
<td>215.0502  216.6873</td>
</tr>
<tr>
<td>combined</td>
<td>1128</td>
<td>185.2305</td>
<td>.9746499</td>
<td>32.73431</td>
<td>183.3182  187.1428</td>
</tr>
<tr>
<td>diff</td>
<td>1128</td>
<td>-61.2766</td>
<td>.6844631</td>
<td></td>
<td>-62.61966 -59.93353</td>
</tr>
</tbody>
</table>

\[ \text{diff} = \text{mean(tideat100c)} - \text{mean(ov225at100c)} \]

\[ \text{t} = -89.5250 \]

\[ \text{Satterthwaite's degrees of freedom} = 1055.43 \]

\[ \text{Pr}(T < t) = 0.0000 \quad \text{Pr}(|T| > |t|) = 0.0000 \quad \text{Pr}(T > t) = 1.0000 \]

Statistical Computations were performed using the software StataSE9
## Results

### Comparison of Baselines (150 °C)

<table>
<thead>
<tr>
<th>Variable</th>
<th>Obs</th>
<th>Mean</th>
<th>Std. Err.</th>
<th>Std. Dev.</th>
<th>[95% Conf. Interval]</th>
</tr>
</thead>
<tbody>
<tr>
<td>tid~150c</td>
<td>9220</td>
<td>223.3751</td>
<td>0.0904846</td>
<td>8.688403</td>
<td>223.1977 223.5524</td>
</tr>
<tr>
<td>ov2~150c</td>
<td>13953</td>
<td>225.7967</td>
<td>0.0772886</td>
<td>9.129553</td>
<td>225.6453 225.9482</td>
</tr>
<tr>
<td>combined</td>
<td>23173</td>
<td>224.8332</td>
<td>0.0593492</td>
<td>9.034541</td>
<td>224.7169 224.9495</td>
</tr>
</tbody>
</table>

\[ \text{diff} = \text{mean(tideat150c)} - \text{mean(ov225at150c)} \]
\[ t = -20.3504 \]
\[ \text{Ho: diff} = 0 \]
\[ \text{Satterthwaite's degrees of freedom} = 20402.5 \]

- Ho: diff < 0 \[ \Pr(T < t) = 0.0000 \]
- Ha: diff > 0 \[ \Pr(T > t) = 1.0000 \]
- Ha: diff != 0 \[ \Pr(|T| > |t|) = 0.0000 \]

Statistical Computations were performed using the software StataSE9
Conclusions

- Powdered detergents cannot be used as packing materials
- Parts for GC fabrication can be bought here in the country
- The electro-mechanical thermostat does not fully meet the required performance in maintaining an isothermal temperature
Recommendations

- Refine the temperature controller part of the oven circuit to achieve optimum isothermal condition
- Possibility of temperature programming
- Fabricate own injector port and oven cooling device
- Run pesticide samples such as Malathion
- Come up with a manual
<table>
<thead>
<tr>
<th>stats</th>
<th>tid~t50c</th>
<th>ov2~t50c</th>
<th>tid~100c</th>
<th>ov2~100c</th>
<th>tid~150c</th>
<th>ov2~150c</th>
</tr>
</thead>
<tbody>
<tr>
<td>mean</td>
<td>146.3526</td>
<td>178.7134</td>
<td>154.5922</td>
<td>215.8688</td>
<td>223.3751</td>
<td>225.7967</td>
</tr>
<tr>
<td>N</td>
<td>9220</td>
<td>9220</td>
<td>564</td>
<td>564</td>
<td>9220</td>
<td>13953</td>
</tr>
<tr>
<td>max</td>
<td>192</td>
<td>247</td>
<td>192</td>
<td>240</td>
<td>247</td>
<td>247</td>
</tr>
<tr>
<td>min</td>
<td>115</td>
<td>119</td>
<td>119</td>
<td>183</td>
<td>183</td>
<td>183</td>
</tr>
</tbody>
</table>
For highly inductive loads (power factor < 0.5), change this value to 360 ohms.

Figure 13. Hot-Line Switching Application Circuit (MOC3041M, MOC3042M, MOC3043M)
Hysteresis

- History dependence of the thermostat
- The furnace is either off or on, with nothing in between.
  - The thermostat is a system
  - The input is the temperature
  - The output is the furnace state.
Solid state relays

- Functions also as an electronic switch
- Faster switching time
- Decreased electrical noise when switching
- More expensive
Van E

\[ H = \frac{B}{u} + C_s u + C_m u + A \]

- **Plate Height**
- **Longitudinal diffusion term**
- **Stationary-phase mass transfer term**
- **Mobile-phase mass transfer term**

Multiple Paths
The Development of a Low Cost Gas Chromatograph: Detector and Computer Interface

By: Mitchell Angelo J. Cabrera
Mentors: Dr. Guidote
Dr. Libatique
Engr. Calasanz
Objectives

A. Create a Low Cost GC: Detector

B. Provide a computer interface for the GC

C. Successfully integrate detector with other parts of GC (Mr. Arceta’s thesis) and with the computer interface

D. Create a manual for a “Do It Yourself” GC
Limitations

- Cost should be significantly lower than commercial GC’s ($5,000 or P200,000)

- Analog to Digital Converter can only handle 8 bits (maximum $2^8 - 1$ or 255 points)

- Selectivity of samples dependent on the column (OV225)
Limitations

• Only compatible with computers that have parallel ports

• Program only works on Windows 2000 or higher with Dev C++ libraries

• Thermistors are not as sensitive as the sensors of commercial GC’s
Finding the right detector…

1. Webcams
   - Too programming intensive
   - Low feasibility

2. IR Detectors
   - Could successfully detect the presence of gases
   - Sensor used was made to be TTL compatible (only gave “high” or “low” signals)
Finding the right detector…

3. Car lamps
   - Worked as a “thermistor”
   - Easily oxidized
     (Tungsten filaments)

4. Glow plugs
   - Based on the study of R.E. Herbener (Hillsdale College, Michigan U.S.A.)
   - Requires a large current
Finding the right detector…

5. Thermal Conductivity Detector

- Design readily available
- Basis for previous working educational GC’s
- Inexpensive
How it works: TCD

Separated sample enters detector

Comparison of thermal conductivities of sample and reference gas

Amplification of signal

Production of an electrical signal

Recorder output

Detection Block

Reference Cell

Sample Cell

Imbalance!
Final TCD Circuit Design
Data Acquisition Circuit
Computer Interface
Detector/Output Fabrication

- Designed and Implemented TCD Circuit on Breadboard
- Testing and Finalization of Circuit Design
  - Fabricated TCD Circuit on a PCB (UP IRC Lab)
  - Installed Thermistors in Detector Block
  - Integrated Detector and Computer Interface with GC
- Explored possible detector outputs
  - (microcontrollers, printers etc.)
  - Found and appropriated existing data acquisition circuit (care of Mr. Dennis Duran)
How it works: GC Analysis

1. Power up the GC
2. Turn on power supply of data acquisition circuit
3. Run data analysis program
4. Press S to save data (before injection) and S again to stop recording (after injection)
5. Press Q to stop the program
6. Open the .txt file of the data and copy onto Excel
7. Plot data
Cost

Thermistors: P 17.00 X 2 = P 34.00

LF353 Dual Operational Amplifier: P 13.00

Detector Block/Casing: P 500 – P 1000.00 (Estimation)

UP PCB Fabrication: P 1500.00

4N25 Optoisolators: P 20.00 X 8 = P 160.00

Parallel Port Cable: P 100.00

Other Electronics Components: ~ P 100.00

Total Cost: P 2907.00
Total Cost of our Gas Chromatograph:
P 15,833.49 (Mr. Arceta’s part) + P2,907.00 = P18,740.49

Doing the math:
(P204,459 - P 18,740.49)/P204,459 X 100%

= 90.83% DECREASE IN PRICE
Results and Discussion

Baseline of “Chromatogram”
Results and Discussion

• Preliminary testing: LOD is around 0.2mL (Water Vapor)

• Tests with 0.2mL, 0.5mL, 1.0mL Acetone are inconclusive

• Comparison of signals with Tide packed columns and the OV225 show that the GC can detect the fragrance of Tide
Conclusion

• It is feasible to create a Low Cost Gas Chromatograph with locally bought materials (low sensitivity)

• The selectivity of the detector is dependent on the column

• The detector can only do significant analysis at temperatures below 150 degrees Celsius

• The GC can detect the fragrance of Tide (T-Test)

• Results are still inconclusive for other samples (trials ongoing)

• Upon further study, Glow plugs, Webcams and IR detectors may be used as sensors for the GC
Recommendations

• Optimize parameters

• Use more sensitive thermistors

• Find other places for fabrication of PCB

• Design and fabricate a new detector block

• Make a window at the back of the GC for easier detector maintenance
Recommendations

• Make use of Parallel port-USB connectors

• Make program for computer interface more appropriate for GC analysis (Fix baseline problem etc.)

• Upon enhancing the performance of the GC, create the DIY GC Manual

• Appropriate GC for Palm Pilots

• Continue study on Glow plugs, Webcams and IR detectors as possible GC sensors
Acknowledgements

- **GOD**, for giving us the strength to push through
- Family
- Dr. Armando Guidote
- Engr. Tris Calasanz
- Dr. Nathaniel Libatique
- Sir Cali
- Mr. Dennis Duran (5th year BS ECE)
- Salustiana Dy Foundation Inc. Professorial Chair, for funding our study
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- UP IRC Laboratory
- Dr. Erwin Enriquez
- Mr. Ian Ken Dimzon, Ms. Jaclyn Santos, Ms. Helen Cativo
- Mr. Toto Oppus
- The great Chemistry and ECCE laboratory staff
- Chemistry Faculty
- Blockmates – Aral na!
- All the Chem Peepz 😊
Thank You! 😊