Development of a Gas Chromatograph to Analyze Chemical Heating Packs

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Background

Self-heating hot pots contain a chemical packet to boil a container of water when placed inside. While the complete composition of the packet is unknown, labs in China have identified calcium oxide, sodium bicarbonate, and aluminum powder as common key compounds. These packs generate heat via the exothermic reactions of these compounds in water. The main reactions are as follows:

$$CaO + H_2O = Ca(OH)_2$$
$$2Al + 6H_2O = 2Al(OH)_3 + 3H_2$$
$$2Al + Ca(OH)_2 + 2H_2O = Ca(AlO_2)_2 + 3H_2$$
$$2NaHCO_3 + Ca(OH)_2 = CaCO_3 + 2NaOH + H_2O + CO_2$$
$$2Al + 2NaOH + 2H_2O = 2NaAlO_2 + 3H_2$$

Eq. 1. Chemical reactions.

Hydrogen gas, (H2) a clear, odorless, non toxic gas, is a predicted product in this reaction, while carbon monoxide (CO), an extremely toxic gas, is not. Still, many caution against using self-heating hot pots due to carbon monoxide risk. People cite multiple incidents of triggered CO detectors (including one on campus here at Deerfield) as evidence, however most commercial CO detectors cross-react with H2, making these cases insufficient to prove significant CO production.

In this project we detail and develop methods to for building a low-cost gas chromatograph (GC). We isolate chemical samples from 自嗨锅煲仔饭 (Zi Hai Guo Self-Heating Claypot Rice) and try to identify the reaction products via gas chromatography.

Methods and Materials

A gas chromatograph was constructed using materials available in a high school laboratory or via online retailers. The GC apparatus consists of an air compressor, injection port, column, oven chamber, and detector. A comprehensive list of materials can be found in the appendix.

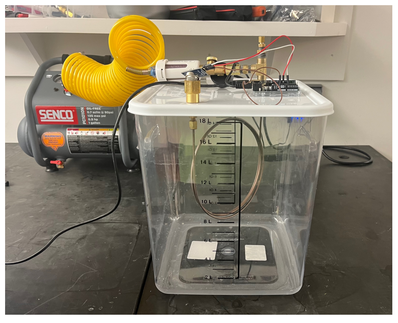

Figure 1. Assembled GC apparatus.

An air compressor controls the flow rate of air, our carrier gas. A packed stainless steel column separates gas as they travel through the stationary phase material. The plastic container holds the apparatus upright and can easily be turned into an oven. Heating decreases retention time but is not strictly necessary for separation, so we left the oven empty for this experiment.

An MOS (metal oxide semiconductor) gas sensor at the end of the column varies voltage input based on gas concentration. The specific model used is MQ-5, though other MQ sensors were tested.

The sensor was connected to the computer and allowed to heat up for a few minutes before data collection. The gaseous products were then collected via syringe and manually injected into the GC septum while carrier gas flowed through at a pressure of 25 psi. Directly after injection, the pressure was then increased to 75 psi to expedite data collection time. This pressure was maintained for the remainder of the data collection period.

Before running samples, we ran the GC with air to see how air flow affected the signal. At t=10s, 5ml of air was injected and the pressure was turned up from 25 psi to 75 psi. The pressure change caused a drop in signal that quickly stabilized. There was no visible sign of the 5ml injection, so extra air in samples are not expected to interfere with or be noticeable in results.

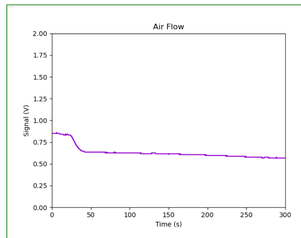

Figure 2. Chromatogram of carrier gas (air) with changing pressure.

Results

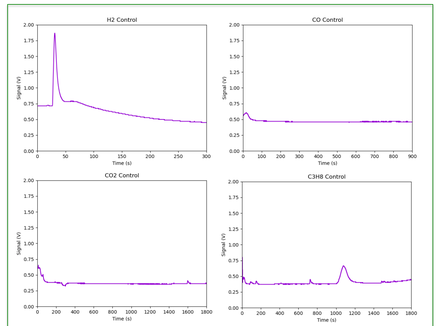

Figure 3. Chromatograms of various control gasses.

Control samples of individual gasses were done to determine sensor sensitivity and later aid in matching unknown peaks via time. H2 and C3H8 caused high signals and were easy to detect chromatographically. CO and CO2 produced low signals and were indetectable after passing through the GC.

A mixture of gasses was used to demonstrate the separative ability of the GC. The gas sensor could only actually detect 2 of the 4 gasses in this mixture, so we expected a maximum of 2 peaks in a successful graph, one for H2 and one for C3H8. We indeed see 2 peaks corresponding to H2 and C3H8 and appearing at appropriate times based on control sample data.

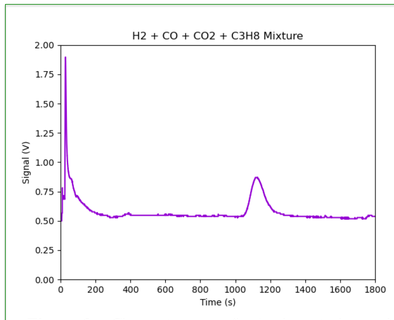

Figure 3. Chromatogram of mixed control sample showing successful separation and identification of H2 and C3H8.

With a more sensitive detector we would expect to see a CO2 peak directly in front of or overlapping with the C3H8 peak, and a CO peak about halfway between H2 and C3H8.

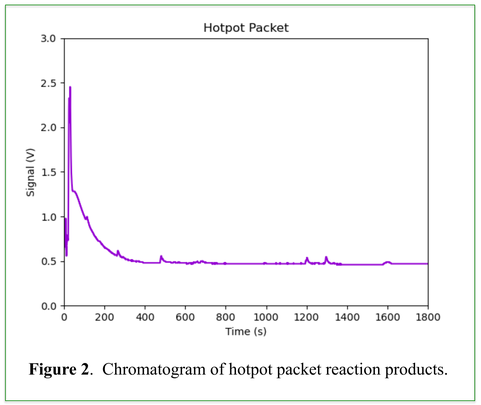

Figure 2. Chromatogram of hotpot packet reaction products.

The only identifiable product of the self-heating packet was H2. An abundance of H2 is stoichiometrically expected and clearly shown here. CO2 is also predicted but was not detected in this graph. CO was not expected nor detected. These missing peaks do not prove the absence of these gasses.

Based on our hypothesis for the contents of this packet and subsequent chemical reactions, we expected to see a CO2 peak following the H2 peak, and an otherwise empty chromatogram. Due to our limitations in sensing ability, we could not fully produce this.

Limitations

Of the MQ sensors, the MQ-9 and MQ-7 are most sensitive to CO, however they require cycling voltage and data reading which leaves gaps in chromatograms. The MQ-2, MQ-5 and MQ-135 operate on constant voltage but can only detect CO levels above several hundred ppm. Our control CO supply of 100ppm was thus indetectable by the MQ-5 sensors we used. Our control CO2 concentration was also likely below the detection threshold.

The missing peaks in our control and experimental chromatograms can likely be attributed to below-sensing-threshold levels or other sensor faults. It is still possible, however, that the GC could not transport or separate certain gasses. Different sensing equipment and definitive sensing of gasses pre-GC would be required to confirm the efficacy of the rest of the GC apparatus. In the future we hope to improve our detection method to be able to show CO2 and CO presence. At present, our results are inconclusive in determining whether or not self-heating hotpot packets produce CO.

References and Appendices

References

Appendices

Full Paper