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Modification of a Gas Chromatograph for Use at an Undergraduate Laboratory

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Introduction

Gas chromatography provides chemists with a powerful method of separating and quantitating chemicals in the gas phase. Our lab recently received two PerkinElmer Arnel AutoSystem XL Gas Chromatographs from MillerCoors Brewing Company, which were designed to sample gas directly from a pressurized source. One of the gas chromatographs (GC) uses a photoionization detector (PID), a flame ionization detector (FID) and an external sulfur chemiluminescence detector (SCD). This GC was modified so that samples could be introduced by means of a syringe and a split/split-less injector. The original gas lines were entirely reconfigured to enable dual detection using the PID and FID. Work is underway to employ the SCD for the detection of sulfur compounds using headspace sampling. The overall goal of this project was to configure a GC that would be useful to undergraduate laboratories.

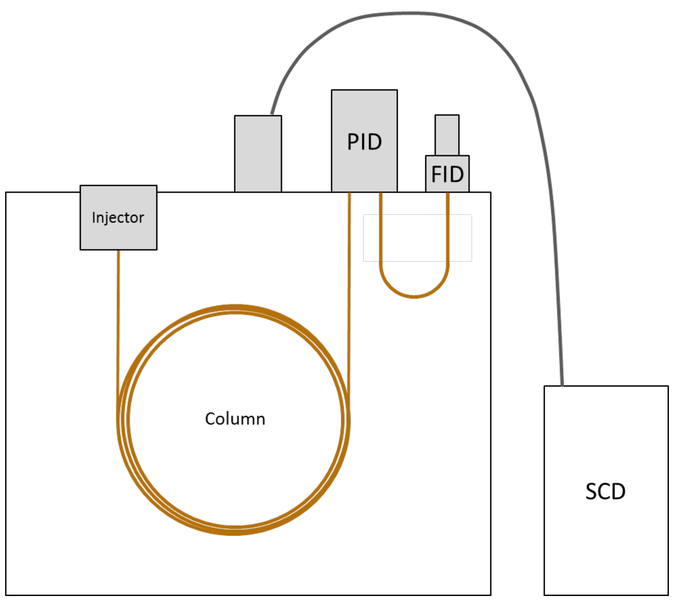

Figure 1. Diagram of the reconfigured GC.

Experimental

Instrument Set-Up. The valves in the originally in the GC, along with the gas lines connected to them, were removed. An split-splitless injector was installed for splitless functionality and connected to two flow controllers via the input port and the septum purge. A SupelcoWax™-10 column (60 M x 0.32 mm, I.D.: 0.25 µm) was attached from the injector to the PID. The PID was coupled to the FID. GC flow rates were established as follows: column, 1.5 mL/min; septum purge, 1.75 mL/min; FID hydrogen, 45 mL/min; FID air, 450 mL/min; PID make-up gas, 10 mL/min. The GC analog output was digitized and transferred to a computer using ChromQuest software.

Sampling. The PID intensity was set at medium-low. The oven, injector, PID and FID were set at 250°C. 3-Heptanone (25 µL) was injected into the GC and data was collected for 5 min.

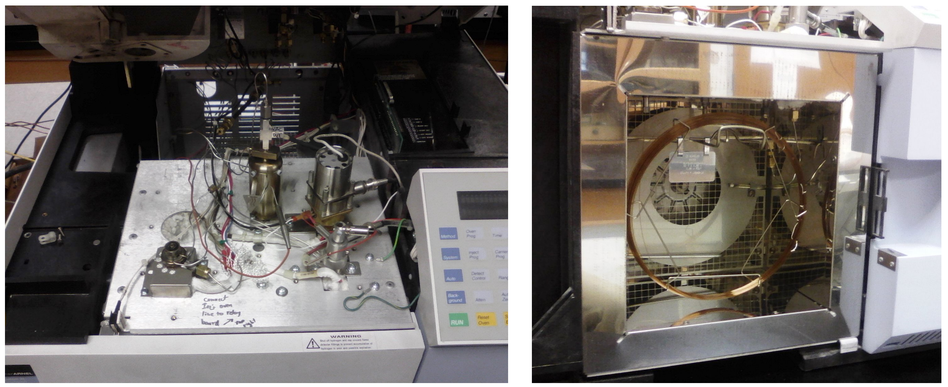

Figure 2. The reconfigured injector and detectors under the hood of the GC (left) and the GC oven with the capillary column (right).

Results and Discussion

A number of challenges were overcome during this project. The injector purge line broke free of its weld and was secured using high temperature epoxy. A complex arrangement of third-party valves, that were part of the original configuration, were removed in order to permit the new plumbing configuration (Fig. 2). The analysis of 3-heptanone can be seen in Figure 3.The FID signal yielded a narrow and fairly symmetrical peak with very little tailing. The PID signal is atypical for a chromatogram and not usable for quantitative studies. Reasons for this are unclear. Other injections produced quantifiable PID chromatograms, so clearly more optimization of the two detectors is needed.

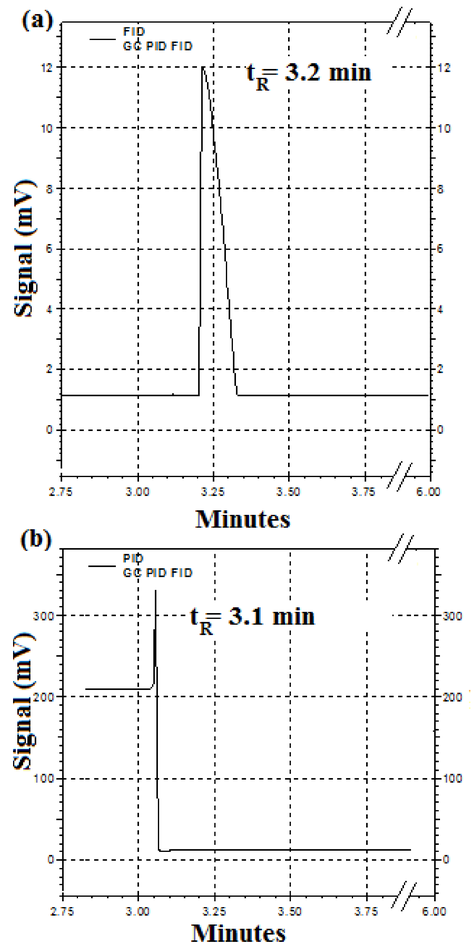

Figure 3. Chromatogram of 3-heptanone from the (a) FID signal and (b) PID signal.

Future Work

The analysis of sulfur compounds in the headspace of a beverage sample was in progress during the GC reconfiguration and was awaiting the successful test of the SCD. Upon exposure to 400-nm light this beverage would produce a thiol species that was intended to be analyzed via GC-MS and GC-FID. Finally, the split function of the injector will be implemented in order to improve the chromatographic peak shape.

References