

A Trace Analysis of Suspected Wastewater

by

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Senior Honors Thesis

May 4, 1990

A handwritten signature in cursive script that reads "James P. Rybarczyk". The signature is written in dark ink and is positioned below the typed name of the mentor.

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INTRODUCTION AND THEORY:

This project involved the testing of water for organic pollutants. The water being tested was under a great deal of suspicion; it was a creek (Mud Creek) which passed directly through a landfill. Mud Creek, which converges with the White River, is part of the Muncie water supply. The landfill, located just outside of Muncie, was thought (by local residents) to be using careless dumping techniques. In addition to this, people who had wells in the area complained of being sick more than usual. Past research (1) had also shown conclusively that the landfill contaminated the creek with several toxic inorganic trace metals. There seemed to be ample reason to conduct an analysis of organic pollutants. PH²

The most effective method of testing for organic compounds in a liquid sample was gas chromatography. Gas chromatography is a qualitative (tells what it is) and quantitative (tells how much) technique which can detect compounds as low as 1-10 mg/L. Gas chromatography is a fairly simple technique. The liquid is injected with a syringe into a port, where it is picked up by an inert carrier gas. This gas flows continuously through the column. Inside the column are solid particles, known as the stationary phase, which interact with the compound. The column is heated to a temperature which will put the compound(s) into a gaseous state. The carrier gas and compound, known as the mobile phase, flow through the column at a constant rate. When different compounds are present, they interact with the stationary phase differently. Some compounds are very reactive

(with the stationary phase) while others are not. In this way, the different components of a mixture can be separated. Compounds which are less reactive with the packing will come out of the column earlier than more reactive compounds. The time required for a substance to completely go through the column is known as the retention time. (see diagram I)

The detector used with this column is a thermal conductivity detector. This apparatus basically consists of a heated metal wire, which carries a current. This current then passes through a series of resistors, known as a Wheatstone Bridge. The carrier gas alone gives a constant current while passing over the wire. Organic compounds do not conduct electricity as well as the carrier gas, and the change in current is picked up by the resistors as the compound passes over the wire. The detector is hooked up to a strip chart recorder, which prints out the changes as peaks. Each peak corresponds to a single compound. (see diagram II)

Diagram I: GOW Mac 150 Gas Chromatograph

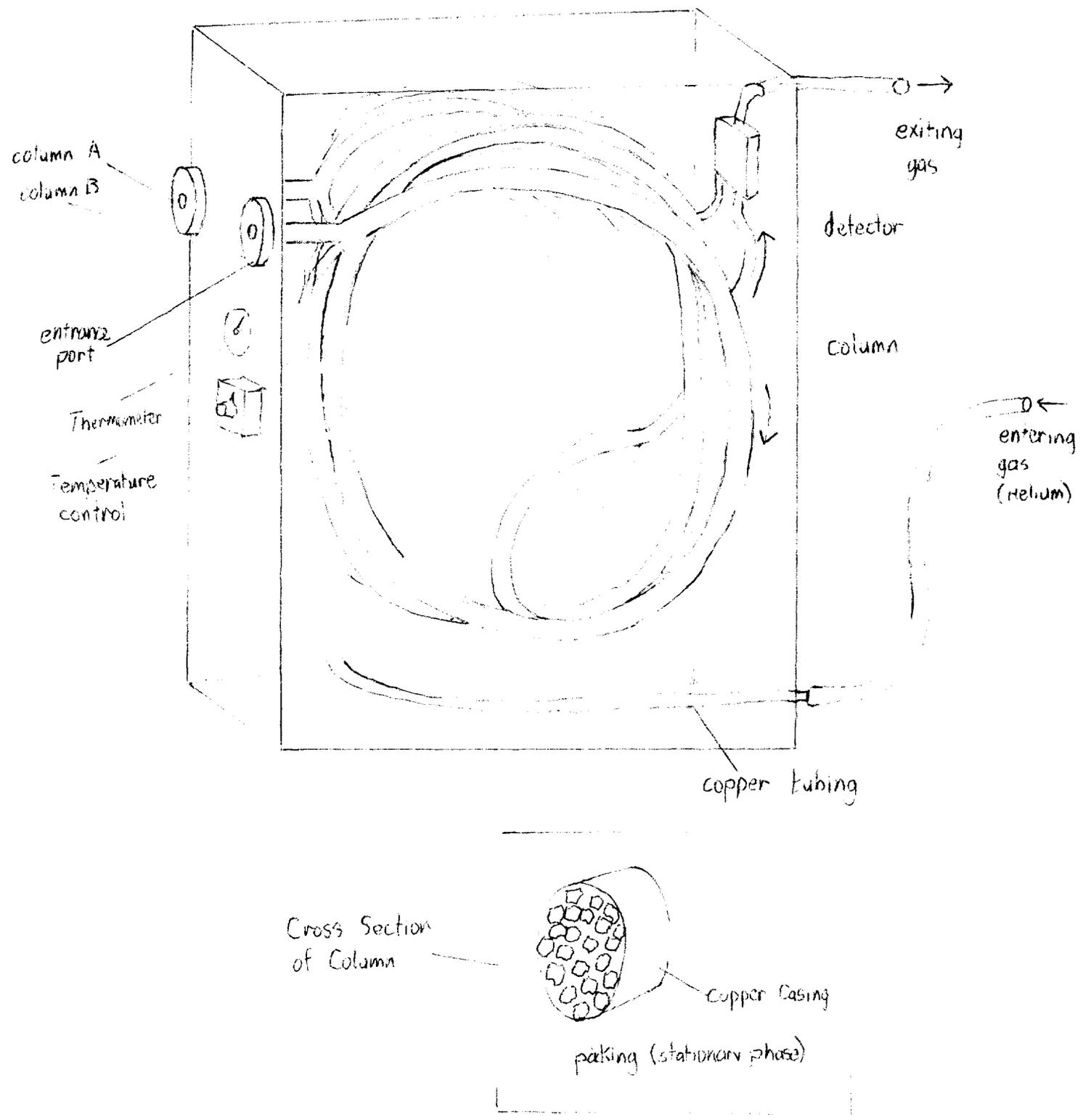
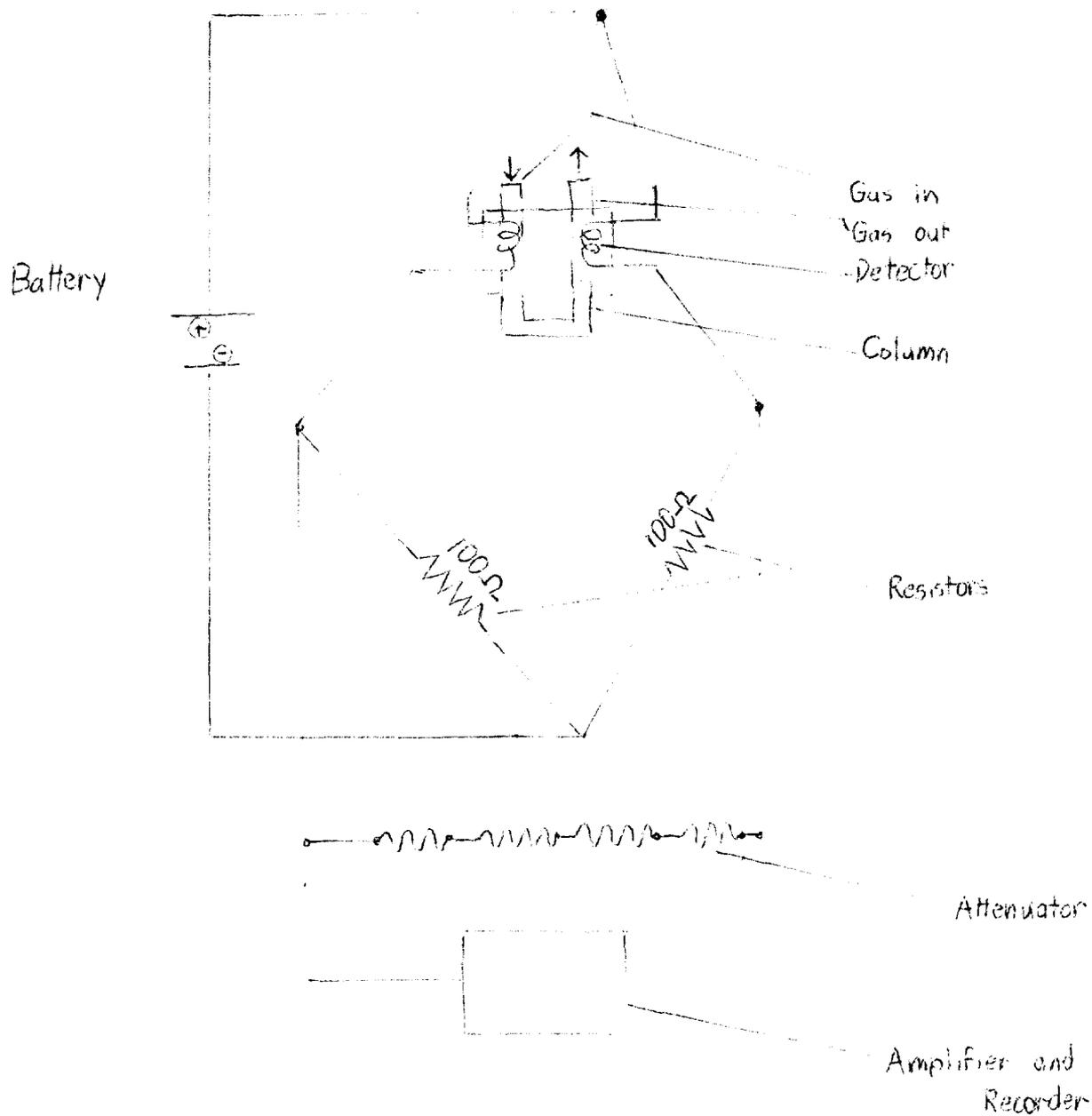


Diagram II: Schematic view of Detector



EXPERIMENTAL:

Water samples were taken from several sites. These samples were taken both upstream and downstream from the landfill. The difference, assuming there was one, would be due to the landfill. Two ditches, which drained from the landfill (west and east of it) were also examined. The samples were sealed and refrigerated immediately after collection, to prevent volatile organics from evaporating.

The experimental procedure was very straightforward. The seemingly polluted samples (such as Mud Creek downstream) were the main focus of investigation. Each sample was run "as collected" (not concentrated). However, only one peak was registered by the detector in each case. This peak, of course, was water. Any other compounds should have shown up as separate peaks. This procedure was repeated many times. Several different variables were incorporated into the procedure. For example, the samples were shaken before analysis (in case the organics were more dense than the water, and were near the bottom). The samples were also taken from different places in the creek, and at different water levels. The results of these tests were the same; only one compound (water) was seen.

The next technique used was extraction. A small amount of organic solvent (5ml, compared to 50ml of water) was used to solvate any organic compound present. The rationale behind this was that if the compound would go into solution, it would be at a relatively higher concentration than in the water. Several extractions were taken each time, and then combined. A variety of solvents were used, including polar and nonpolar,

as well as volatile and nonvolatile. Polar molecules contain an electronegative or positive element(s), such as oxygen (O), and react differently than nonpolar organics. It should also be noted here that the gas chromatograph contains two columns: one which reacts with polar and one that reacts with nonpolar compounds. In each stage of the testing, both columns were used. The two nonpolar (and poorly volatile) solvents used were toluene and xylene. The two polar, volatile solvents used were dichloromethane and ether (universal organic solvents; almost everything is soluble in them). The result of each test run, however, was the same: one peak, one compound.

Two additional techniques were then employed. The flow rate was varied, as was the temperature. Different flow rates make compounds come out at different times, and can increase or decrease resolution. This proved futile, as no additional peaks "appeared." The temperature variation also gave negative results. It was hoped that increasing the column temperature might vaporize a nonvolatile organic which previously could not be seen. It did not, however, and lowering the temperature (to see a volatile compound) gave a similarly negative effect.

Finally, combinations of all of the previously listed techniques were tried. Nonvolatile extractions were run at high column temperatures, for example (and vice versa). One other technique used in the search for organics (specifically nonvolatile) was the "boiling off" of water. More specifically, water was distilled away, in hopes that the less volatile compound would remain behind. As more of the water was boiled off, the amount of organic compound left behind would supposedly become

greater. The small amount of remaining water could then be extracted with a solvent, and run through the G.C. This method was very time consuming, and gave no positive results.

RESULTS:

(1) There were no detectable, significant amounts of organic pollutants in the water immediately surrounding the landfill.

(2) These results were confirmed by a mass spectrometer analysis performed on a Mud Creek (downstream) sample. Actually, very minute amounts of naphthalene (a component of diesel fuel) were found. Its concentration, however, was so low (5-10 ppb, after condensing) that it could not have been detected by the G.C. used. Mass spectrometers are extremely expensive, and I did not have access to one.

DISCUSSION:

The results of this experiment, although not very exciting, were definitely positive. Muncie's drinking water was (and hopefully is) not being contaminated by organic compounds from the landfill. The experience was rewarding, though it did not initially seem to be. A good working knowledge of the gas chromatograph was obtained, and many techniques were learned. All possibilities for finding compounds were seemingly exhausted. A great deal of time was spent on this "search" for contaminants, and none were found. The confirmation by mass spectrometer analysis was an assurance that a thorough job had been done. The knowledge and experience obtained in this research is extremely valuable. These techniques could be applied to trace analysis of almost anything (especially organic compounds), or mixtures of different compounds (as would be utilized by food and drug analysis). The research done in this lab was both functional and educational.

REFERENCE:

Bye, Sheryl. "The Migration of Heavy Metals from a Landfill to Surface Waters" (unpublished).