Volatile Sampling Device Prototype II

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Abstract— For Gas Chromatographer there are currently many sampling devices that are used in conjunction with Gas Chromatographer to improve its detecting and sampling capability. Cryo-Focusing, Headspace sampling, Headspace Trap Sampling, Solid Phase Micro extraction, and many other techniques are used. There is also a technique known as Volatile sampling for which previous work for volatile sampling device prototype has been constructed. In overcoming the short comings of the previous device is being explored.

Index Terms— Gas Chromatographer, Volatile Sampling Device, Cartridge Heater, Compressor Refrigeration, Adsorption, Elution, Seperation.

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1. GAS CHROMATOGRAPHY

There are currently many diverse techniques that can be used for the separating solutes from mixtures. These are used in the analysis of constitutes of a mixture organic or inorganic in nature. Chromatograph is a chemical analytical instrument used for extraction of various constituents from a complex entity. The chromatograph uses a packed narrow column, in this column the complex entity which is to be extracted is entered from one end. Then due to adsorption and desorption phenomenon the various constituents of the entity travel at different velocities through the column. The constituents are then separated and differently collected.

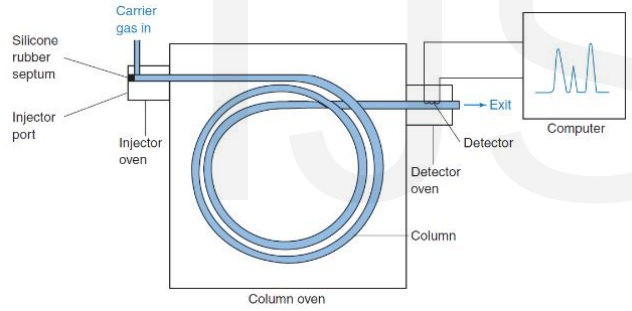

Figure 1 Gas Chromatographer Block Diagram

In gas chromatography, the components of a sample are dissolved in a solvent and vaporized in order to separate the analytes by distributing the sample between two phases: a stationary phase and a mobile phase. The mobile phase is a chemically inert gas that serves to carry the molecules of the analyte through the heated column. To separate the compounds a solution sample that contains organic compounds of interest is injected into the sample port where it will be vaporized. The vaporized samples that are injected are then carried by an inert gas, which is often used by helium or nitrogen. This inert gas goes through a glass column packed with silica that is coated with a liquid. Materials that are less soluble in the liquid will increase the result faster than the material with greater solubility. The purpose of this module is to provide a better understanding on its separation and measurement techniques and its application.

2. VOLATILE SAMPLING DEVICE

Volatiles in the current context are molecules with boiling point up to 200 0C. Conventional methods for separating volatiles are biased towards one of the physical or chemical properties of the molecule and hence are not complete. So volatiles separated from the mother matrix will contain fewer molecules and even the most precise instruments can resolve only fewer molecules. On the other hand, a better sampling device utilising thermal desorption, kinetic desorption, solubility in steam, desorption by microwave, desorption by magnetic induction or laser can separate

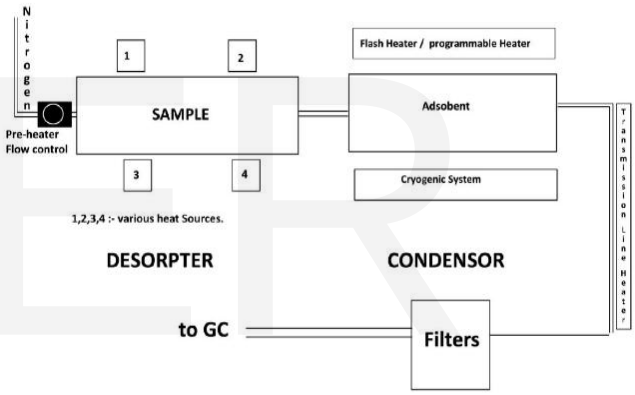

Figure 2 Volatile Sampling Device Block Diagram

larger number of molecules and hence can enhance the efficiency of instruments like MS.

A sampler using thermo-kinetic desorption was fabricated in house and comparative study was conducted against conventional techniques. A 25% increase in peaks was observed when analysed with GC. The possibility of thermal decomposition was ruled out by conducting GC- MS studies. The aim of the current study is to develop a highly precise sampling device with global standards and has wide applications in the field of aromatic, analytical, biological and medical fields.

2.1 Volatile Separating Device CFD Analysis

This Report describes a new design approach that came from a volatile separation technology. The conventional methods, separated volatiles from materials from Organic matters are biased to any single physio-chemical properties of the volatile and are efficient to separate low boiling fractions only. The goal of this new design approach is to provide better heating and cooling modes, Equipment, maintaining of inert atmosphere in envelope, well stabilized desorption-adsorption process for volatiles from the provided organic

matter. Volatile is a substance which can change state from a solid or liquid to vapour. Gas chromatography (GC) is the instrument used to study volatiles and it can analyse molecules whose boiling points are as high as 250OC or more. But it is necessary to make sure that Non-volatile material should not enter into the GC column. So there is a requirement of a safe sampling device for GC. Sampling device is separator unit, which will extract the molecules or volatiles from the given sample. The Prototype device for this is already manufactured, which is a POC (Proof of Concept). The goal of this report is to reduce the size of complete equipment, modular design. For the purpose of heating the envelope electrical heaters are used with sensor for establishment of feedback control system. Also for cooling the purpose electric cooling chip are used, which works on peltier effect. This report also analyses influence of the internal geometry design over flow path of nitrogen gas and turbulence created by pressurized nitrogen gas, with the help of CFD software.

2.2 Volatile Sampling Device Prototype-I

Figure 3 Volatile Sampling Device Prototype I

Compared to the Previous development where Dry Ice and Acetone was used for achieving Sub-Zero Temperature range. In Prototype-I Peltier IC was employed. Due to Change in shape of Desoptor to accommodate the CFD analysis theory, Cylindrical Heater were not feasible. Instead Cartridge heaters were used. Critical analysis was heating and cooling of the Condensor which is supposed to reach sub-zero temperature range. Multiple variations of heating and cooling have been experimented for optimum heating and cooling. A previous paper explores the same and a brief of the same has been mentioned below

2.3 Heating Curve

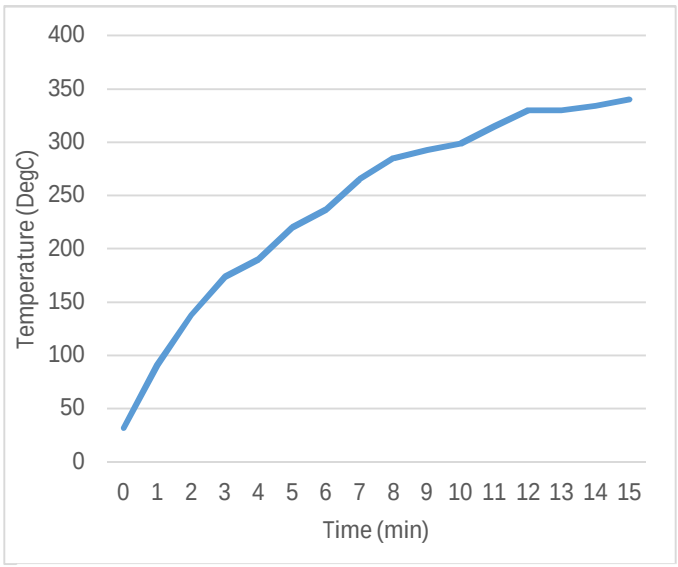

Figure 4 Condensor Heating Curve using 3 Heater

The Initial heating was done with 2 number 125 Watts heater and natural cooling was observed in the same. It was noted after multiple iteration that the heating up to 200 degrees for the internal cavity where the material is to be processed takes up to 10 min on an average. This would not work with the flash heating concept. Subsequently Heating with Higher wattage heater and incrementing the number of heater were implemented without much success as SS 316 is bad

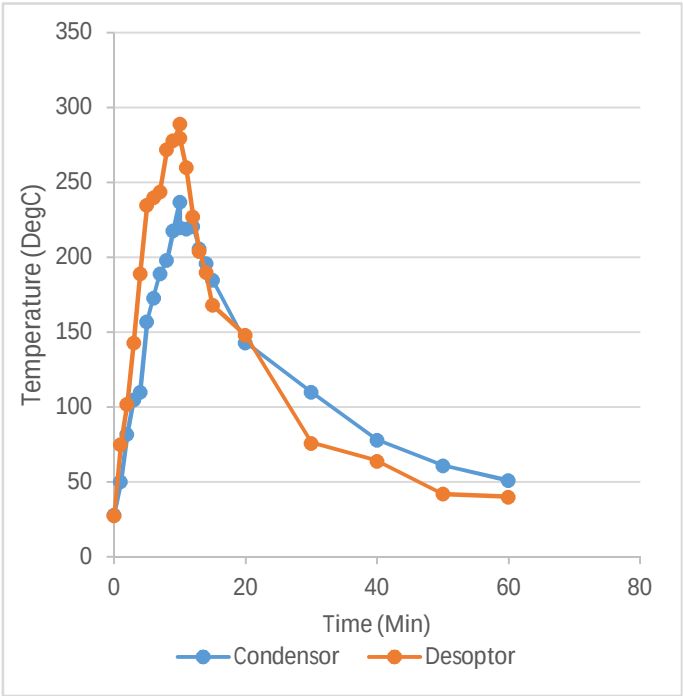

Figure 5 Heating and Natural Cooling Curve

conductor of heat, therefore. Heating and cooling took longer period than expected. Though operating point of operation was around 200 degrees even after further increments by using 175Wattage heater the average time of heating remains at 7 to 8 minutes. Making heating operation incompatible with flash heating. Additionally, Natural cooling curve remains its course, hence affecting further cooling curve.

2.4 Cooling Curve

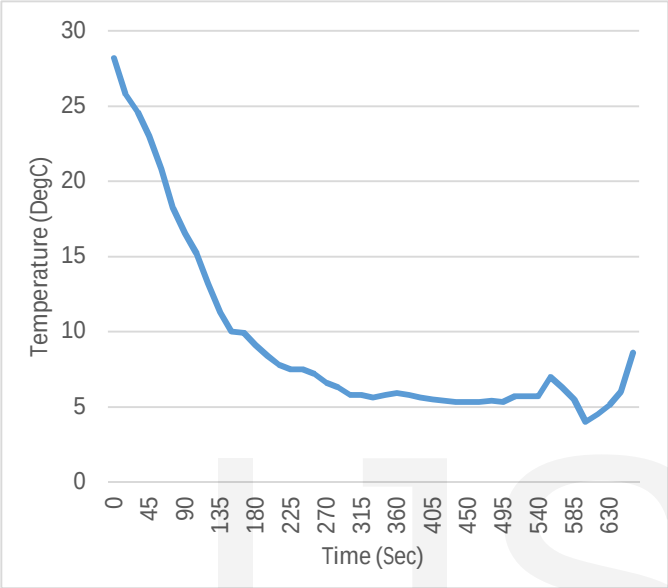

Figure 6 Condensor Cooling Curve

The cooling was condenser was implanted in various fashion. Using water block the Peltier plate hot side was cooled to room temperature. This would enable us generate a temperature difference of 20 to 30 degrees at the maximum the graph as shown explain the behaviour of condenser temperature during cooling. After sometime as the water heats up the effectiveness of Peltier cooling is lost and the same can be seen on the graph. Also roughly 3 min of effective cooling was achieved.

To further improve the cooling the water block were used with ice water creating a more effective but extremely bulky cooling solutions as the ice water would require continuous circulation. And the effective cooling time achieved was only 10 mins. After which the temperature stabilizes if the ice water allowed to circulate the ice eventually melts creating lack of cooling for further iteration. Thus, generating variable results in case to case basis

2.5 Limitations of SS316L during Heating

SS316L has a very poor thermal conductivity hence during heating and cooling process it takes up lots of time. Also during the same period if additional heat sink or any other cooling device is employed bringing the temperature down to room temperature takes a lot of time. Though the SS316L is compatible with any organic material but its inability for quick temperature turn around makes it unsuitable for volatile sampling device.

2.6 Limitations of Peltier during Cooling

During Peltier operation, it was observed that the temperature difference between the hot and cold side of was a maximum of 40Degrees. Also, the Peltier needs to dissipate the amount of heat it is absorbing otherwise the Peltier tends to overheat on both the sides. For various commercially available Peltier, it was observed that the maximum temperature range that the hot side can go up to is 150 ˚C.

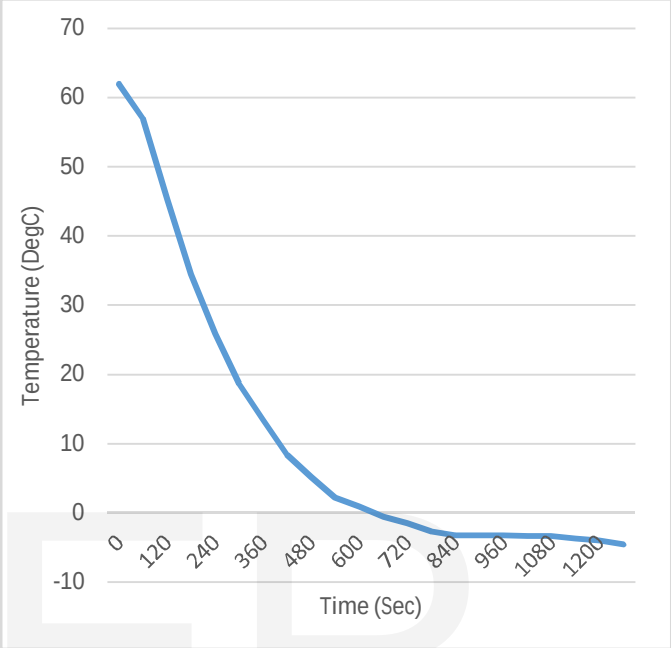

Figure 7 Condensor Cooling Curve

So, cooling process could not begin until the condenser is naturally air-cooled. Naturally air cooling drastically increases the time required for one cycle. Also when the Peltier is used only with a heat sink on the opposite side the lowest temperature achieved is significantly different from when Ice cooled water is supplied using water blocks. When the lowest temperature of -24 ˚C was to be achieved Ice water was used for the purpose. This in turn makes the system bulk. Also each Peltier draws a power of 120W on 12V, the Power supply requirement also increase drastically when using 3 Peltier IC, in parallel.

3. Further Modifications

Figure 8 Anodised Aluminium Desoptor Condensor

Considering the following limitations, it was further decided to use Anodized Aluminium and replace the Peltier IC with a cooling agent circulation technique. The reasoning being that Aluminium having a lower thermal resistance will offer

quick turnaround need for volatile sampling device. The results of the same are discussed further. As for the cooling agent, it was decided that when a pre-cooled media was prepared and circulated through the system the temperature

Figure 9 Compressor Refrigeration System

drop would indeed been driven by the thermal capacity of the cooling media. In the case of use of Aluminium for Desoptor and Condensor it would be a considerable advantage to have a cooling media of higher thermal capacity i.e. using of media other than water. An oil based mixture being the most stable under higher temperature and still in liquid state at sub-zero temperature.

4. Volatile Sampling Device Prototype-II

Considering the modification, it was duly decided to employ a compressor based thermal refrigeration system for cooling. An oil bath was thus prepared which would be pre-cooled to -5˚C for rapid turnaround of temperature. This cooling media was circulated only through the Condensor and Desoptor cooling not being critical in nature was air-cooled to conserve energy and reduce cost. The heating and cooling curve of same have been plotted below. For Manufacturing the Condensor and Desoptor Commercial Grade Aluminium was used. Both the components were White Anodized from inside as well as outside.

4.1 Heating Curve

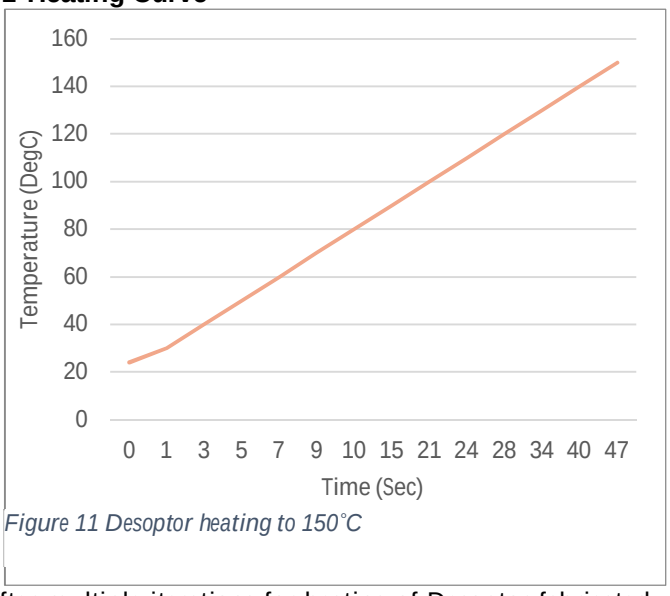

Figure 11 Desoptor heating to 150˚C

After multiple iterations for heating of Desoptor fabricated from Aluminium, the following average reading are presented in the graph. It can be seen with that 2-cartridge

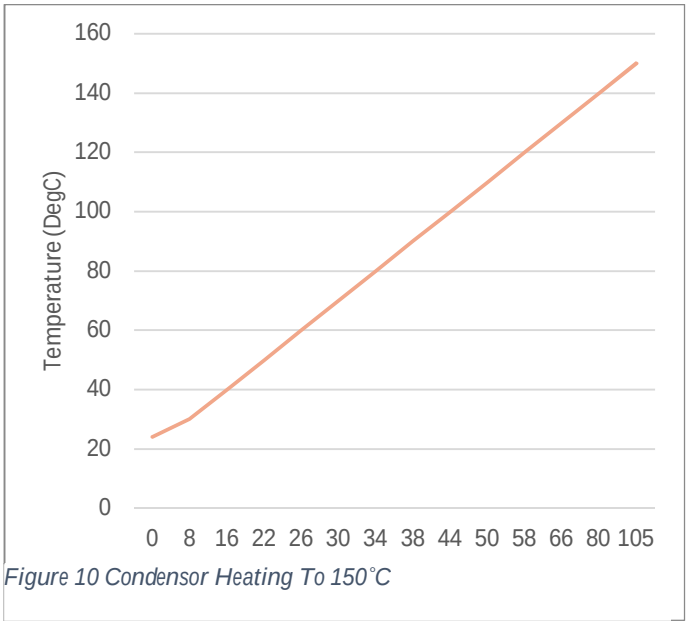

Figure 10 Condensor Heating To 150˚C

heaters of 175W it consistently possible to achieve 150˚C in under a minute. The control of the temperature is perfectly linear in nature. Any other temperature required above 150˚C is achieved with the same curve, data of the same can be extrapolated from the graph mentioned below. This give a fair bit advantage in heating temperature control of desoptor with regards to time taken to achieve a particular temperature.

Condensor fabricated from Aluminium was heated using 2-cartridge heaters of 175W. After multiple iteration and taking the average reading of the same plotted below is the heating graph for Condensor. Like Desoptor, even Condensor has linear heating curve. Heating below and above the mentioned temperature, the time required can be directly extrapolated from the curve shown below.

4.2 Cooling Curve

Cooling of Desoptor being predominantly by natural air

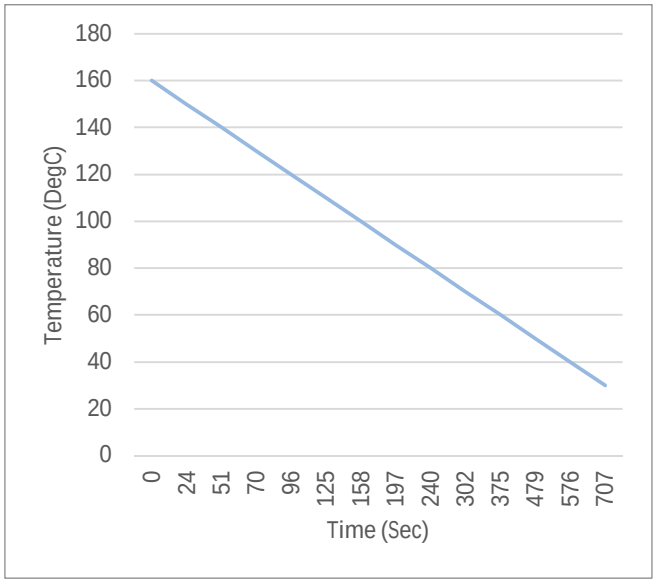

Figure 12 Desoptor Cooling From 150˚C

convention. Cooling of the same was effected by using an instrument cooling fan. Also, cooling times of the Desoptor not being critical in nature. The average of multiple iteration has been plotted in the graph below. It can be seen that unlike SS316L Condensor the maximum cooling time for Aluminium Condensor from 150˚C to room temperature is 10 minutes compared to 1 hours. This being a considerable advantage when reloading of samples were concerned.
Condensor cooling was employed using oil based cooling media agent. The oil was pre-cooled to -5˚C using compressor based refrigeration method. After the Condensor achieved a certain temperature the cooling media was pumped through the compressor under pressure, thus causing rapid fall in temperature. This fall in temperature can controlled with a delayed pumping operation. In the graph plotted below average values of multiple iterations where rapid pumping was always employed. It can be seen that the capacity selected is far excess in nature, as the temperature drops over 150˚C in 30 odd seconds. This method being a very effective cooling method

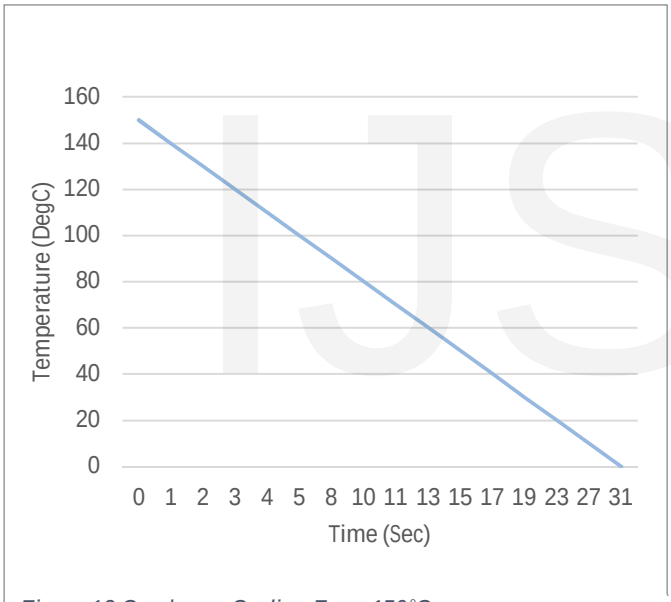

Figure 13 Condensor Cooling From 150˚C

5. FUTURE SCOPE

Cooling unit designed is very effective but bulky in nature. For effective operation of volatile sampling device, it would require a very power effective and size effective cooling methodology. In its current state a prototype of sampling can be generated and effective volatile sample can be verified.

6. CONCLUSION

The incapability of SS316L heating and cooling have been compensated by employing Anodized Aluminium, which in turn also maintains the compatibility of a food grade material. Being light weight and cheaper than SS316L, Aluminium also a commercially feasible option. Peltier continuously consumed power supply of 360 watts along with Ice water bath required to keep it operational to achieve the desired temperature range. The Compressor based refrigeration continuously consumes 1.5KW of energy but on the contrary generates results exceeding expectation. But the bulky nature of the compressor and its periphery components need to be worked on to improve it overall performance.

7. REFERENCES

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