Headspace Sampling Device

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Introduction

Volatile Organic Compounds(**VOCs**) are present in a diverse array of sources, encompassing both naturally occurring organic materials and synthetic, inorganic products, the analysis of these compounds is a key component in most industries, some of which include the monitoring of VOCs in beverages[1], food[2] or the analysis of certain chemicals[3].

VOCs are analyzed by vaporizing an analyte and siphoning a respective aliquot of vapor; **headspace** to a mass spectrometer for analysis, my headspace sampler aims to provide a cost effective alternative to current market auto samplers.

Main Objectives:

- Distribute analyte from liquid phase to its gaseous state using heating and agitation methods such as stirring.
- Distribute aliquot of headspace to mass spectrometer for analysis.
- Develop a unified user interface that integrates and optimizes the system's various parameters for enhanced efficiency.

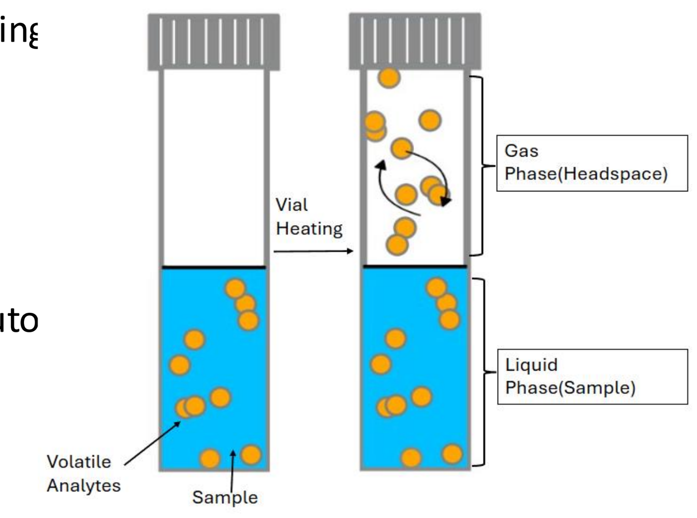

Fig1. Analyte Distribution from liquid phase to gas phase.

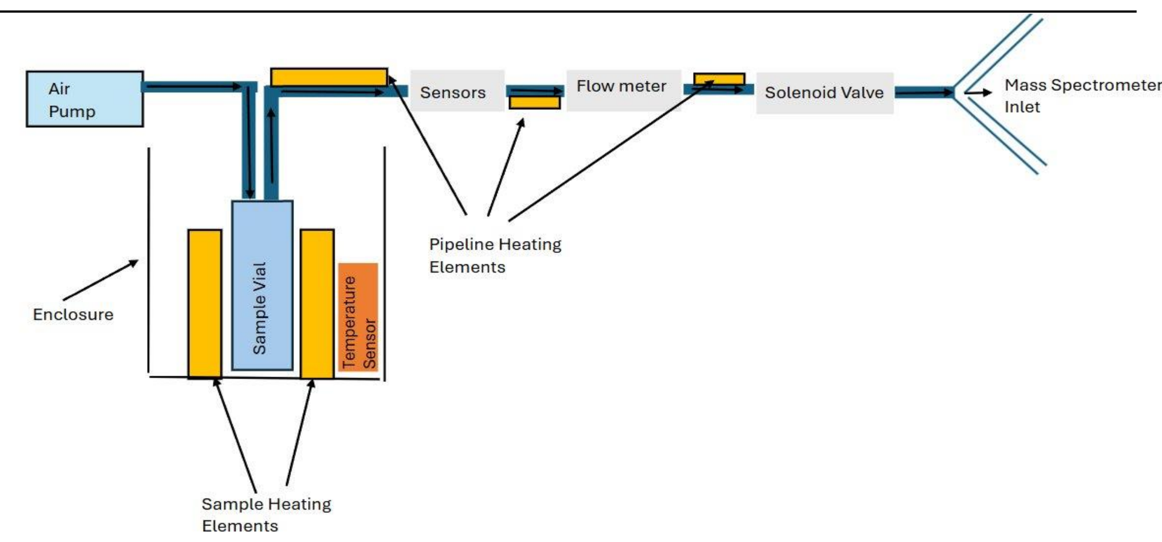

Fig2. Simplified System Plan: Air-flow pushes the desired headspace to the mass spectrometer when the headspace has reached equilibrium.

Methodology Analyte Vaporization & Distribution

Hardware:

1. Heating
2. Airflow Distribution

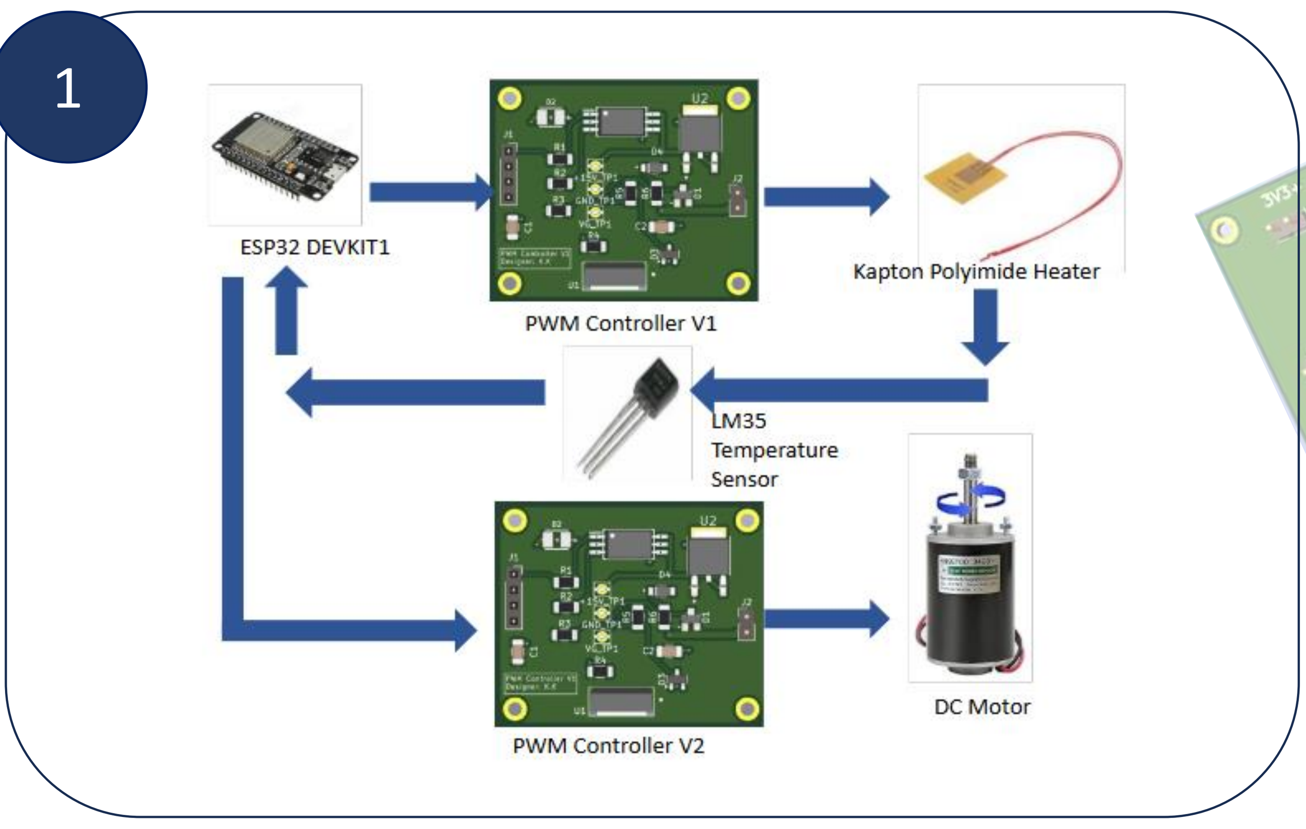

Fig3. Analyte heating architecture illustrating how the ESP32 MCU and the various peripherals; LM35 Temperature sensor, Kapton Polyimide Heater and DC Motor coupled with a neodymium magnet facilitates the conversion of an analyte from liquid to gas phase.

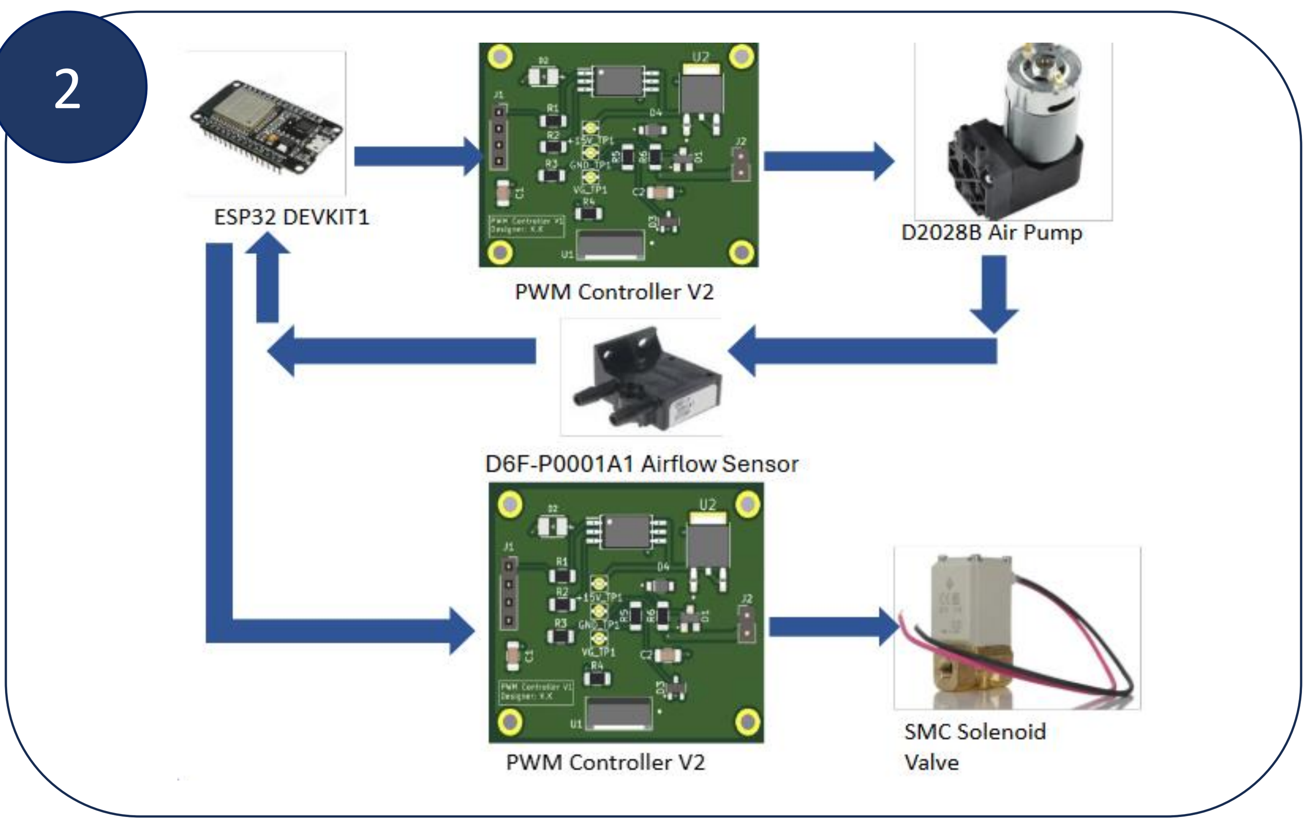

Fig4. Airflow distribution architecture illustrating how the ESP32 MCU and the various peripherals; V1 and V2 controller boards, D2028B Air Pump, D6F-P0001A1 Air Flow Sensor and SMC Solenoid Valve collaborate to manage the airflow distribution.

•**ESP32 DEVKIT1; Wi-Fi** integrated **MCU,** enabling robust web functionality and seamless internet communication allowing the web user to access system peripherals wirelessly, powered by a specially designed interposer board, capable of delivering necessary voltage to system peripherals.

•**PWM Controller V1**: The initial Version of the PWM controller for high-side switching , engineered for rapid precise and secure **PWM** operation.

•**LM35**: An integrated temperature sensor featuring a **one-second** thermal response time, which ensures accurate temperature measurement.

•**High Torque DC Motor**: When paired with a neodymium magnet and a corresponding magnet in the analyte this motor fascinates a **faster equilibrium** for the liquid.

•**PWM controller V2:** The second version of my custom designed PCB for low-side switching, it offers enhanced protection against inductive loads and an improved interface for connecting other peripherals.

•**SMC Solenoid Valve:** 12V valve that facilitates in the distribution of the headspace.

•**D6F-POOO1A1 Airflow Sensor:** facilitates the distribution of the headspace, enabling the user to select desired aliquot of headspace.

•**D2028B Air Pump**: 12V air pump that enables variable airflow into the system.

Software

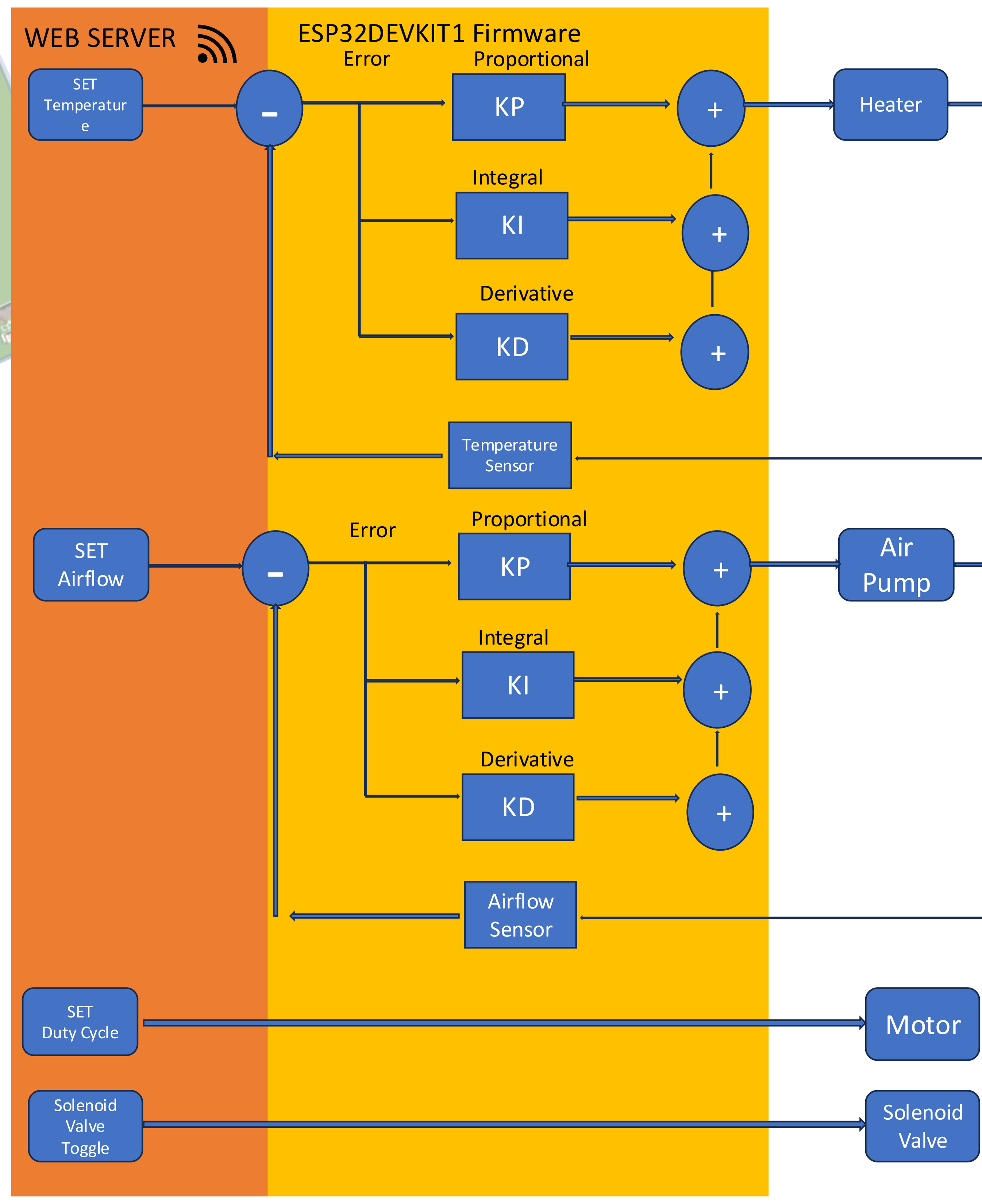

Fig5. Overall software design, showcasing Webserver and Firmware communication to operate system Peripherals.

Software functionality:
Comprised of three elements:

- Arduino sketch
- HTML file
- JavaScript file

1. HTML file defines the structure and layout of the user interface.

2. The JavaScript file handles the dynamic aspects of the interface by, fetching data periodically and graphing respective data, the script ensures real time updates where the graph is continuously updated as soon as new data is received.

3. The Arduino sketch handles sensor data acquisition, the PID algorithm and hardware control.

Together , these files create a complete system where the ESP32 performs sensor monitoring and hardware control, the Web Server built with HTML and JavaScript provides real time feedback and control over the connected devices.

System Overview

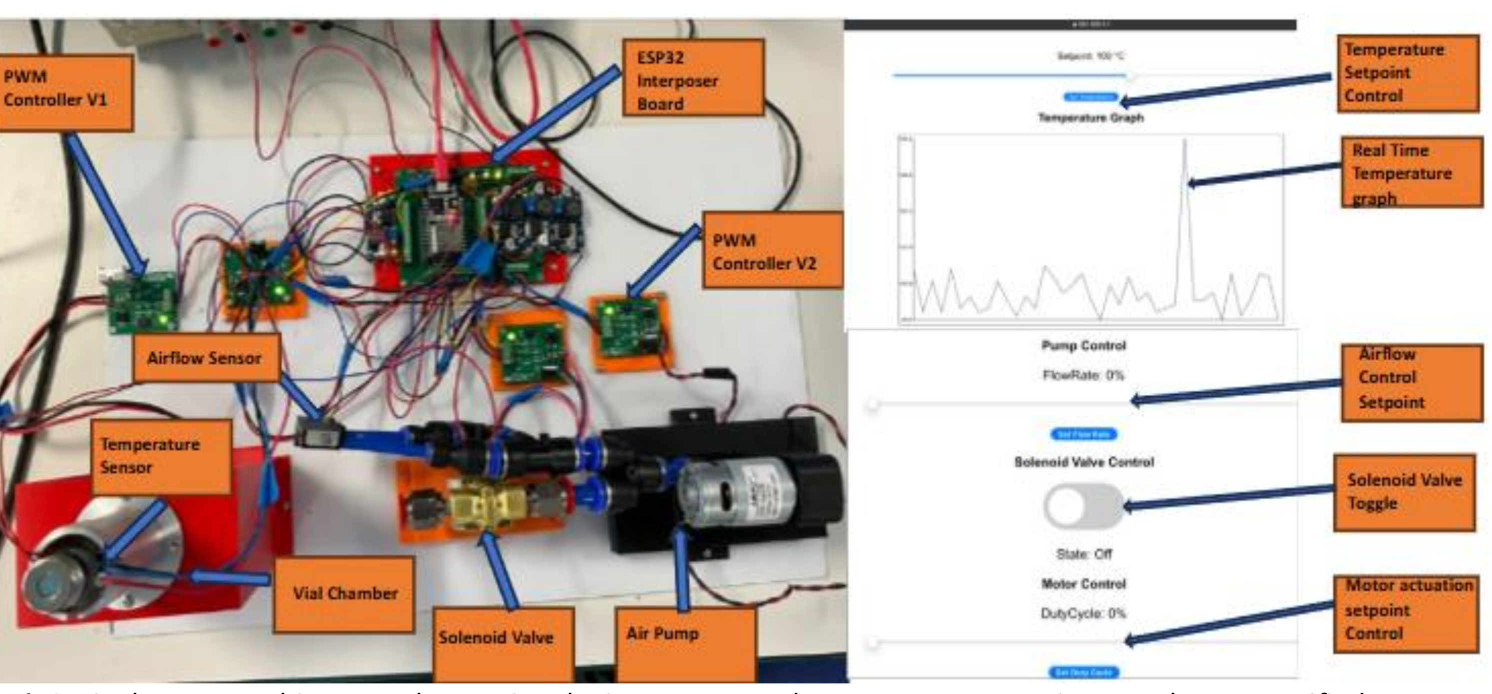

Fig6. Final system architecture showcasing the interconnected components operating together as a unified system.

The system immediately adapts to parameter changes hosted on the web server, ensuring that the vial temperature reaches its setpoint in minimal time. The airflow setting allows for adjustable airflow delivery, solenoid actuation occurs without delay, and motor control lets its user stir or disturb the vial contents as needed. Overall, the system functions as intended with minimal to no faults.

Results

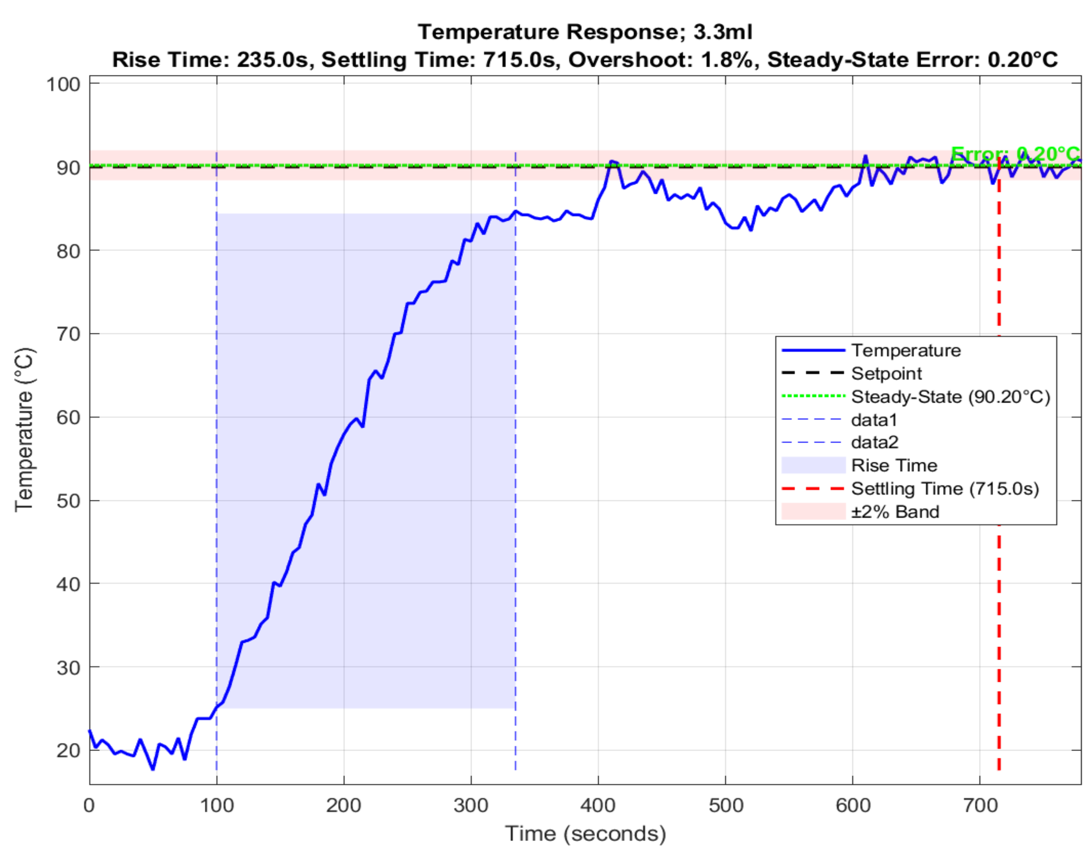

Fig7. PID Temperature response, with vial volume at 3.3ml.

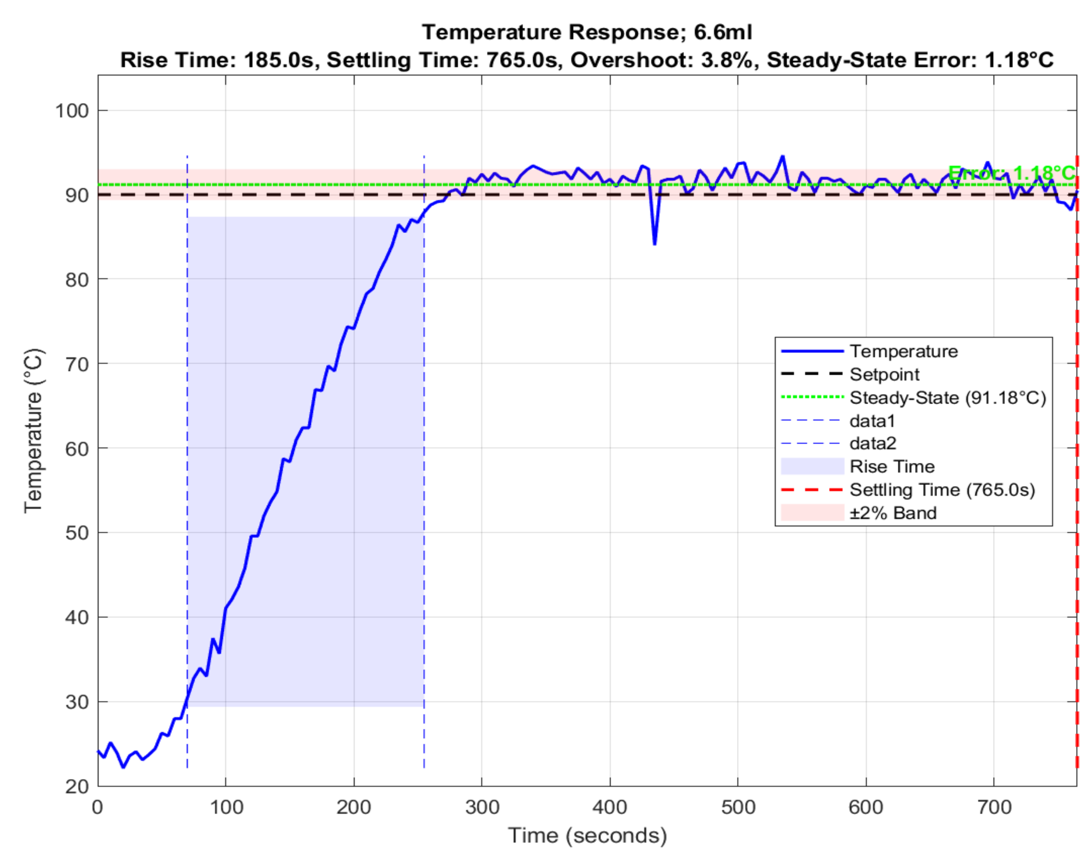

Fig8. PID Temperature response, with vial volume at 6.6ml.

Conclusion

- The temperature of the vial gets to the desired temperature with 0.69°C error and an average rise time of 3 minutes and 30 seconds.
- The graphical user interface enables enhanced accessibility, allowing for easy access to the different parameters of the system increasing sampling efficiency.
- Variable airflow allows a desired aliquot of headspace to be delivered, when needed.

Further Developmemt

- Reduced electrical noise.
- Aluminum pipes and brass fitting to enhance quality of sample profile.
- Enclosure to protect exposed circuit elements.
- Additional sensors; Humidity and Pipe Temperature Sensor for further system control.

References

[1]*An-flavor-compounds-BEER-8697-headspace-8890-gc- ...* Available at: https://www.agilent.com/cs/library/applications/an-flavor-compounds-beer-8697-headspace-8890-gc-5994-4292en-agilent.pdf

[2]. Perez-Hurtado *et al.*, "Direct analysis of volatile organic compounds in foods by headspace extraction atmospheric pressure chemical ionisation mass spectrometry," *Rapid Communications in Mass Spectrometry*, vol. 31, no. 22, pp. 1947–1956, Oct. 2017, doi: https://doi.org/10.1002/rcm.7975.

[3]Author links open overlay panelN. NicDaéid and AbstractThis article provides a very brief introduction to the methods used to collect (2013) *Analysis of fire debris*, *Encyclopedia of Forensic Sciences*. Available at: https://www.sciencedirect.com/science/article/abs/pii/B9780123821652001 01X?fr=RR-2&ref=pdf_download&rr=8d215110899463f4