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Fast GC Oven Cool-Down for the Determination of Residual Solvents

Technical Note 002

The most popular GC application in the pharmaceutical field is USP 467, the determination of residual solvents. Typically run on a GC-FID with a headspace sampler, this analysis examines drug products for the presence of harmful compounds that may have been introduced during the manufacturing process. Speed of analysis is important because there are so many samples that must be routinely analyzed. Many labs, however, do not wish to deviate from established chromatographic conditions for fear of compromising test results. Indeed, any changes to column dimensions, temperature programs and carrier gas flow rates would necessitate a new method validation exercise which itself requires considerable effort.

One way to reduce the time of analysis and increase sample throughput is to shorten the cool-down step of the GC cycle. This approach has many advantages. First and foremost is that the method does not have to be re-validated because SOP conditions remain intact. In addition, method development is not needed and technicians do not have to be re-trained.

Fast Oven Cool-Down

Cool-Down times depend on the ventilation conditions and the heat capacity of the ventilation medium. Conventional GCs rely on the internal oven fan to ventilate the oven between runs. Liquid CO_2 is not widely accepted because it is expensive and labor intensive. A tank farm must be managed and individual tanks must be changed frequently. On the other hand, simply improving the ventilation performance of the GC oven can speed up the cool-down process considerably. Many "real world" factors can impact cool-down times. Sometimes a GC is placed in an area with restricted air movement around the intake and exhaust vents. Hot exhaust air can mix with the incoming air intended for cooling the oven. Often a duct is used to direct the hot air away from the oven intake but this restricts exhaust flow and puts more load on the oven fan. All of these conditions result in slower cool-down rates.

The GC Chaser is a Fast GC Accessory that improves oven ventilation through the use of an auxiliary blower. It significantly speeds up the cooling cycle under optimal conditions and also overcomes limitations imposed by vent restrictions. It does not use any consumables such as liquid CO_2, therefore, it is cost effective and labor free. The GC Chaser is controlled by a circuit that monitors the electrical current applied to the GC oven during the temperature program. At the end of the run the circuit activates a centrifugal blower. Once the start temperature is reached it automatically turns the blower off. Installation is quick and easy. The current sensor, shown in Figure 1, simply snaps around one of the oven heater wires. The blower is ducted to the oven intake by a flexible aluminum duct and a flow adapter slides into the intake as shown in Figure 2.

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Figure 1: Current Sensor.

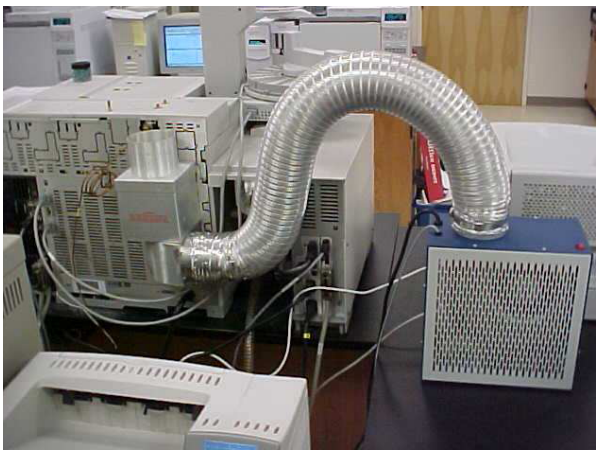

Figure 2: GC Chaser Ducted to the Oven Intake.

Experimental

An evaluation was performed at Schering Plough, Union NJ to determine the GC Chaser impact on routine residual solvent analysis. It was installed on an Agilent 6890 GC with an FID and a LEAP Technologies CTC CombiPAL headspace sampler. Sample sets were analyzed strictly according to SOP. No changes other than the addition of the GC Chaser were made. The start temperature for the run was 30₀C.

Results

The GC Chaser reduced the cool-down time by 2 minutes resulting in an immediate gain of 8% in sample throughput. This lead to an additional 5 sample runs between system suitability checks. Oven stability at the start temperature is also important. Fluctuations directly impact the retention times of highly volatile compounds such as methanol. The retention times were compared for runs made with and without the GC Chaser. Using helium carrier, RT drift was not observed as shown in Figure 3. Separate trials were performed with hydrogen carrier gas with the same result, i.e. no RT drift was observed.

Conclusion

Greater productivity can be achieved with fast oven cool-down accessories. This approach is especially attractive because separation parameters are not changed and methods do not have to be re-validated. The GC Chaser provides a low cost, simple design that is fully automated. Initial capital cost is quickly recovered through higher sample throughput.

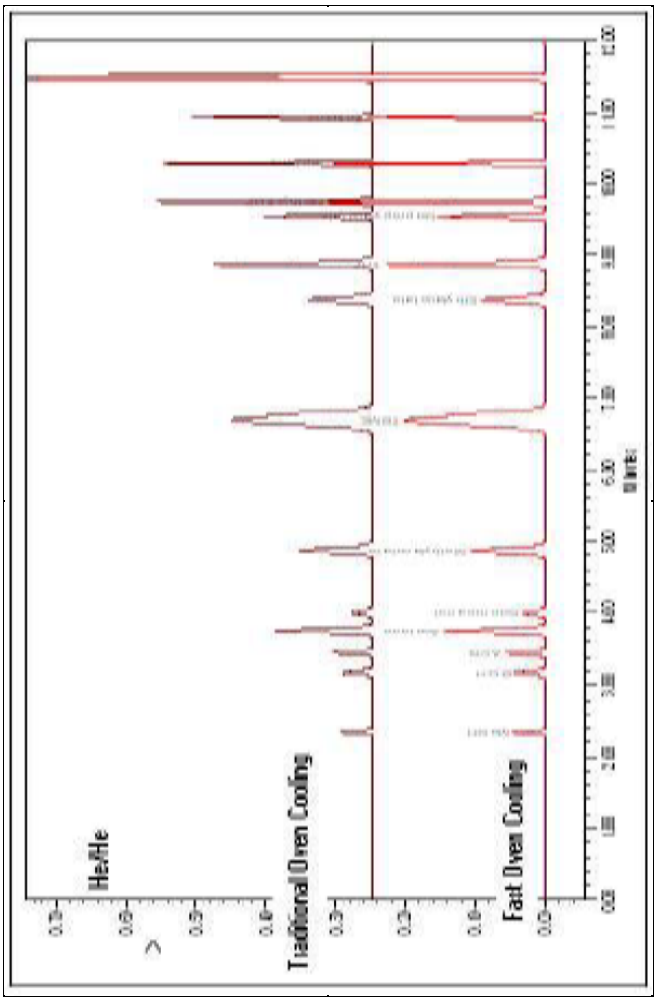

Figure 3: Comparison of Two Oven Cooling Methods.

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