www.sge.com

TECHNICAL ARTICLE

A NOVEL FOCUSSING TECHNIQUE FOR GC

The AirSharp™ is a peak-focussing GC accessory that uses compressed air as a coolant to cool a very small area of the capillary column rather than the standard liquid carbon dioxide (CO2) or liquid nitrogen (N2) that are used in many of the currently available cold traps. The AirSharp has been designed to focus individual peaks just prior to the compounds eluting from the column. This focussing increases the signal-to-noise ratio and sensitivity of individual peaks thereby lowering detection limits and making low-level analyses easier without increasing the amount of contamination through excess large volume injection.

Features and uses

AirSharp is simple to install and use and once installed can be left in the GC oven when not in use. The AirSharp can be used with most GC instruments and requires a GC with software that has external event capabilities.

Unlike the liquid CO^2 and N^2 cold traps that require large amounts of bottled gases, the AirSharp can use air from a cylinder or by air compressor. The AirSharp only uses small quantities of compressed air at a time to cool sections of the capillary column making it cheap to use.

The AirSharp is designed for use at the end of a capillary column to sharpen up individual peaks before they hit the detector. The AirSharp can also be used at the beginning of the column like a standard cold trap to compress the initial sample band if the initial temperature is high enough. Another use for the AirSharp is at the beginning of the 2nd column in multidimensional systems to simulate an injection.

How does it work?

AirSharp works by cooling a small section of the capillary column that is passed through the cooling zone (Figure 1) with ambient compressed air. The compressed air enters through the top of the air transfer line and into the cooling zone as shown in **Figure 1**. The airflow is controlled by the external event function as part of the gas chromatography software. The AirSharp can be turned on and off at set times to suit the elution of individual peaks. This gives the user a great deal of control over when and for how long to apply the compressed air to the capillary column and these settings can be saved to the method for future use.

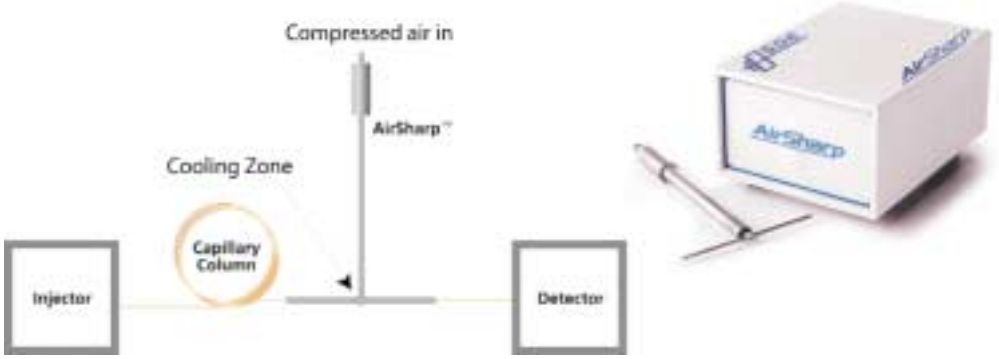

Figure 1. Picture of the cooling apparatus of the AirSharp and note its positioning near the end of the capillary column and before the detector. The capillary column passes through the tubing at the bottom and the compressed air is blown in from the top cooling the outside of the capillary column. Also a picture of the AirSharp control module.

The AirSharp is very effective at increasing the signal-to-noise ratio and therefore sensitivity of late high boiling point compounds. High boiling point compounds require high oven temperatures greater than 150°C before elution from the capillary column. These compounds are usually subject to certain degrees of inlet mass discrimination thereby lowering the amount of compound on-column and therefore are often more difficult to detect. These compounds also spend longer in the column so the sample band will be smaller and broad making detection more difficult. By applying AirSharp to these high boiling compounds on an individual peak basis, the peak can be sharpened and the signal-to-noise ratio increased thereby lowering detection limits. The ambient air that AirSharp uses as its coolant requires an oven temperature of 150°C or higher to be most effective. The ambient air-cools the capillary column by about 80-100°C, thereby slowing the leading edge of the sample band so that eventually the trailing edge will catch it. This process causes the sample band to become very narrow. When the cooling is turned off, the narrowed sample peak is released and travels the short distance through the column to the detector. This results in a very sharp peak shape with a higher signal-to-noise ratio and greater sensitivity (**Figure 2**).

Figure 2 is an example of the AirSharp being applied to a late eluting peak as part of a Polynuclear Aromatic Hydrocarbon (PAH) mixture. As can be seen from the overlaid chromatogram of the standard chromatogram with no AirSharp applied (1) and the chromatogram with the AirSharp applied (2), there is a huge difference in peak heights and peak shape for this chromatogram. The oven temperature when these compounds eluted was 290°C.

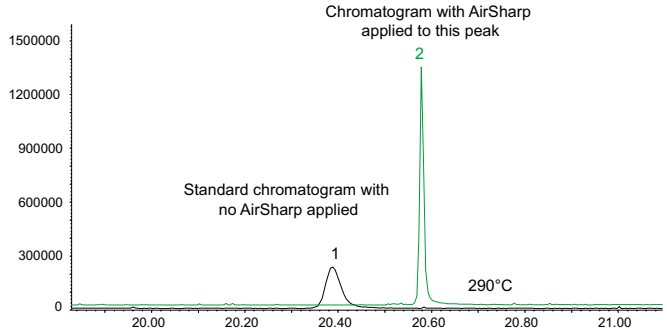

Figure 2. Overlaid chromatogram of a high boiling point compound that has not been subjected to AirSharp (1) and the same peak after AirSharp was applied (2).

How long before the peaks do you apply AirSharp?

The AirSharp should be activated 12-15 seconds before the front edge of the peak is eluted. This allows enough time to cool the section of the column sufficiently to slow the progress of the sample band so the trailing edge can 'catch up' to the front of the sample band. An example of when to apply AirSharp to a peak is shown in **Figure 3**. Taking the chromatogram of the PAH sample shown in Figure 2, the AirSharp is turned on at 20.10 minutes. This is approximately 12-15 seconds before the leading edge of peak 1 would normally elute. The AirSharp is then left on until just after peak 1 would normally have finished eluting from the column, which in this example is 20.45 minutes. The AirSharp was only applied for about 20 seconds to the capillary column and as can be seen from Figure 2, the resultant peak (peak 2) elutes at 20.60 minutes. The peak is response now is much higher than peak 1 making detection and quantitation easier.

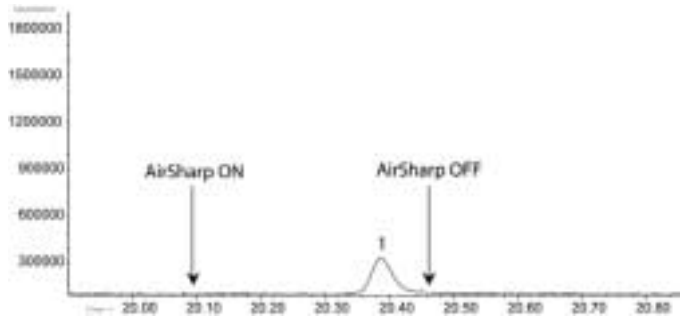

Figure 3. Chromatogram of when to turn the AirSharp on and off with respect to the elution of the targeted peak.

Applications

Applying the AirSharp to a number of peaks in a chromatogram can increase the detection limits of high boiling point compounds that may be present in low levels. **Figure 4** shows the analysis of a commonly screened drug mixture in racehorses. Low level analysis is essential for detecting traces of any potential drugs that the horse may have been injected with. As can be seen from this chromatogram, Diphenoxylate (9) is very difficult to detect at 0.5ng on-column due to background noise/bleed at low levels. This makes reproducible quantitation and detection difficult.

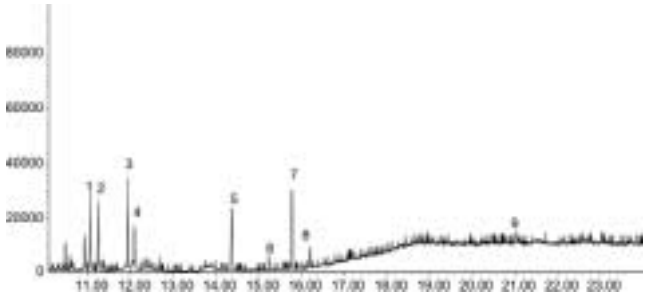

Figure 4. Chromatogram of the horse racing mixture at 0.5ng on column. Notice the low response of Dilantin (6), Nordiazepam (8) and Diphenoxylate (9).

Using the AirSharp to focus peaks 6, 7, 8 and 9 can enhance the signal-to-noise ratio 3-4 times resulting in much easier detection of compounds present in trace levels. **Figure 5** shows the overlaid chromatogram of the standard chromatogram as seen in Figure 4 with the chromatogram with AirSharp applied to peaks Dilantin (6), Diazepam (7), Nordiazepam (8) and Diphenoxylate (9). As can be seen in Figure 5, the responses of the peaks in question after AirSharp has been applied are vastly improved making detection simpler. Diphenoxylate (9) at 0.5ng on column is now easily distinguished from the baseline noise as are Dilantin (6) and Nordiazepam (8). This gives more reproducible quantitation and greater sensitivity resulting in lower detection limits.

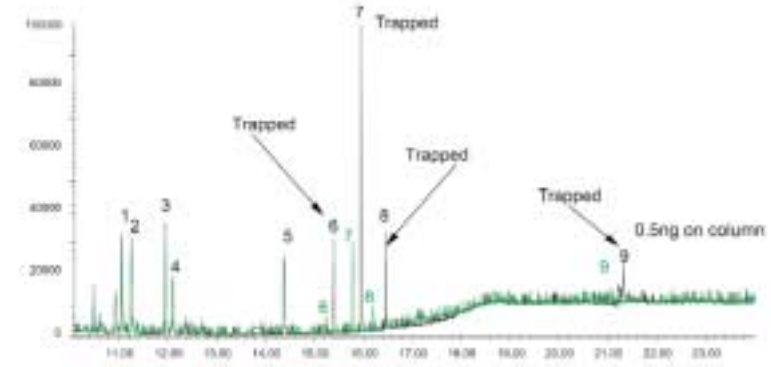

Figure 5. Overlaid chromatograms of the standard chromatogram of the horse racing mixture (in green) and the AirSharp focused chromatogram (in black) at 0.5 ng on column. Note the improved signal to noise ratio of the peaks subjected to AirSharp focusing and the distinguishing the peaks of interest from that of the baseline noise.

Conclusion

The AirSharp is a simple to use peak focussing accessory to a GC that is ideal for peak sharpening late-eluting high boiling compounds. Increased sensitivity of individual peaks avoids the need for large volume injections to detect trace level compounds. Improved peak shape and increased signal-to-noise ratio make quantitation at low levels more reproducible.