Anatune Ltd
Unit 4, Wellbrook Court, Girton Road, Cambridge, CB3 0NA, UK
Tel: +44 (0) 1223279210 Fax: +44 (0) 1223279253
Email: info@anatune.co.uk Internet: www.anatune.co.uk

Separation of Complex Aroma Compounds using Selectable 1D/2D GC-MS with Simultaneous Olfactory and Element Specific Detection

Kathy Ridgway, Applications Chemist, Anatune Ltd, Girton, Cambridgeshire (UK).

First Published: 2015

Introduction

In complex flavour mixtures, it can be difficult to separate closely eluting compounds and fully define those that are most aroma active. The use of human assessors via an olfactory detector may assist in identifying the region of interest, but where peaks are closely eluting accurate descriptors can be difficult and specific compounds may be difficult to identify. The use of extended chromatographic run times, when performing GC-O is also not recommended, as assessors sniffing at the odour port will become tired and potentially less sensitive to a change in odour.

The patented GERSTEL Selectable 1D/2D-GC/MS System can run analysis just through one column (1D mode), but also enables sections of the chromatogram to be cut and separated on a second-dimension column (2D), using so called 'heart cutting'. As the system is based on a single standard GC/MS system, all the data are acquired in one chromatogram. The system is controlled by the integrated software and a simple change in method parameters, meaning a series of methods can be run in a sequence using only one dimension, or making a series of cuts to the second dimension – without any change in the configuration.

The system configuration also enables a choice of column dimensions and phases, enabling two 30M orthogonal columns to be used where appropriate. The use of a Cryo trap can also provide enrichment using sequential heart cuts.

Within this application note, we show how the GERSTEL selectable 1D/2D can be used to help separate and identify coeluting compounds and enable more accurate organoleptic assessment of those most aroma active.
Examples given are one large peak, with several components 'hidden' in the first dimension and 2 closely eluting peaks with different sensory descriptors, separated to enable better olfactory detection. In this example, we also demonstrate the benefits of using an element specific detector (NPD) for certain compounds.

Figure 1 – Instrument set-up

Instrumentation

GERSTEL MPS 2
Agilent GC 7890A with LTM module and deans/splitter
Agilent 5977 MSD with NPD and GERSTEL ODP 3
Maestro software integrated

The system is configured as shown in Figure 1 and illustrated in Figure 2.

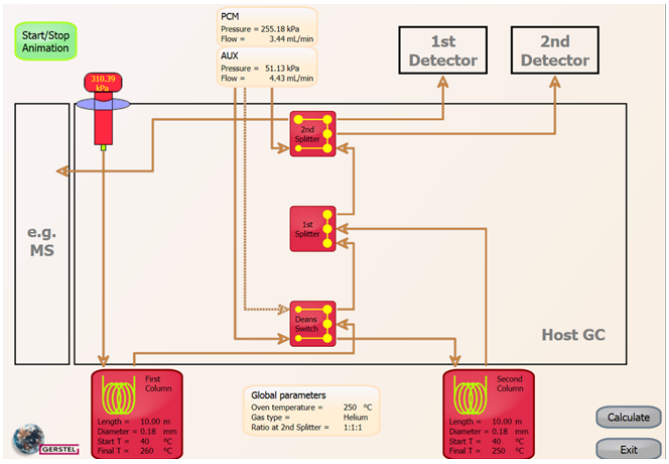

Figure 2 – System configuration

Method

Direct split injection of extracts were performed. The system was set up to split to an MSD, Olfactory detector and NPD (2:1:1).

Injector 10:1 Split at 200C
LTM column 1 DB-5 10M x 180x 0.18 µm,
LTM column 2: DB-wax 10m x 180x 0.18 µm

Method parameters:
To enable a heart cut at a specific region of interest, timed events are used that divert the flow at that point to the second column. This portion can be sent directly to the detector (along with the remaining portion from the first dimension) or trapped until the first dimension analysis is complete. Alternatively, using backflush immediately after the heartcut results in only the analytes from the second dimension reaching the detector after heartcut. Backflush is achieved through a ramped pressure program.

Results

Figure 3 shows example chromatograms from 1D and 2D analysis with the heartcut at 9 minutes that reveals peaks hidden underneath. Separation is achieved without the need for a long run time.

Figure 4 shows the chromatogram from analysis of a complex flavour mix. Using ODP on the 1D chromatogram it was difficult to separate the aromas for Creosol (described as spice, clove, vanilla and phenolic) and Estragole (described as sweet, phenolic, anise). Following heartcut the peaks were better separated.

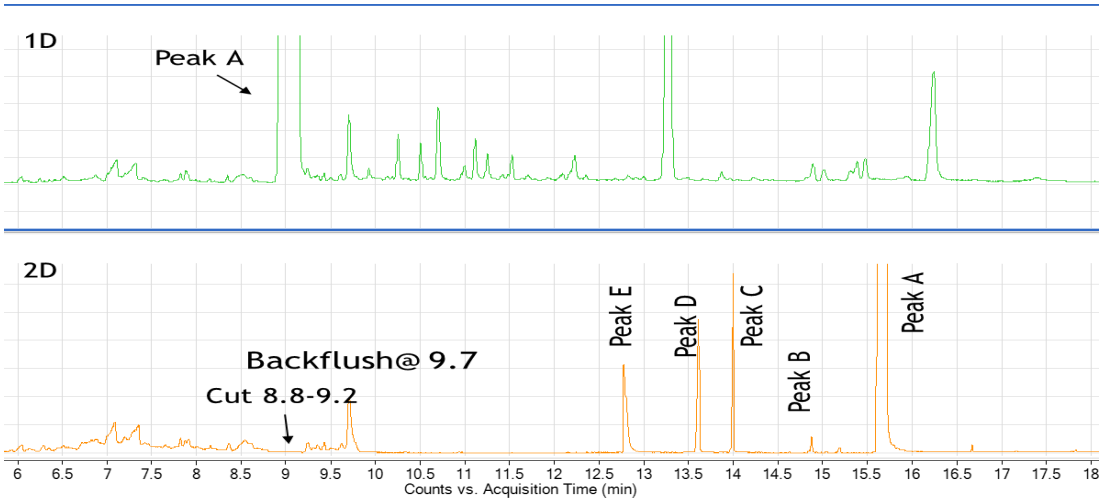

Figure 3 – Heartcut of Peak A, reveals peaks B, C, D and E beneath

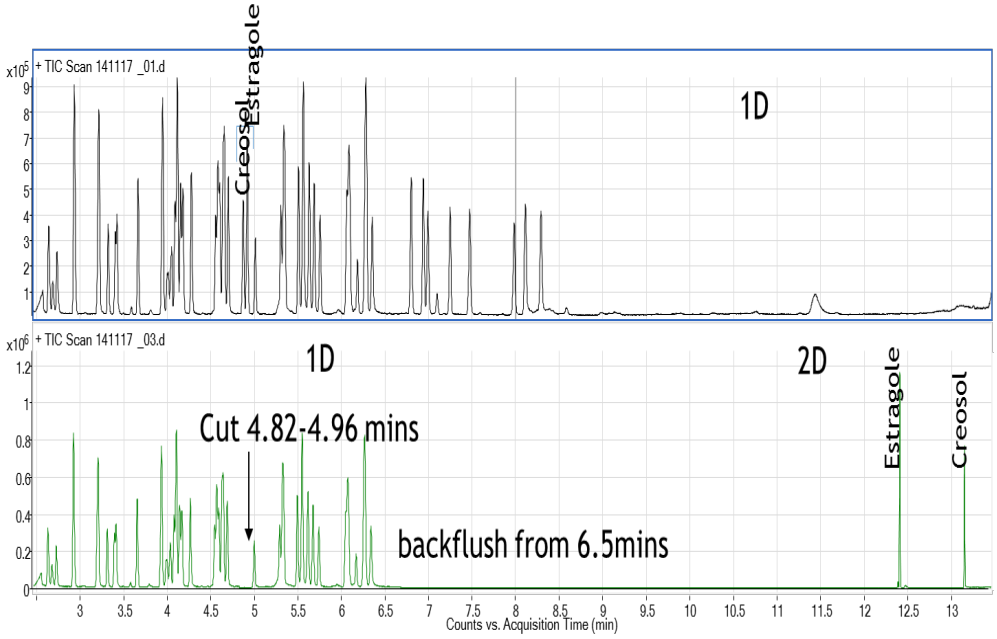

Figure 4 – Heartcutting peaks from a crowded area, enables better Olfactory detection

Figure 5 shows the benefit of using an element specific detector. The increased sensitivity allows better detection and can be valuable, due to the low odour thresholds for many flavour compounds. In this instance the NPD was used to give better detection of nitrogen containing aroma compounds, such as pyrazines and indole. Some are identified in Figure 5. As the backflush occurs after the 2,3-Diethyl-5-methylpyrazine peak, this is still eluted in the first dimension, whereas the peaks between 4.9 and 5.2 minutes are cut to the second dimension and elute later following separation on the LTM2 DB-wax column. The peaks observed in 1D after the backflush time (such as indole) are no longer observed in the chromatogram.

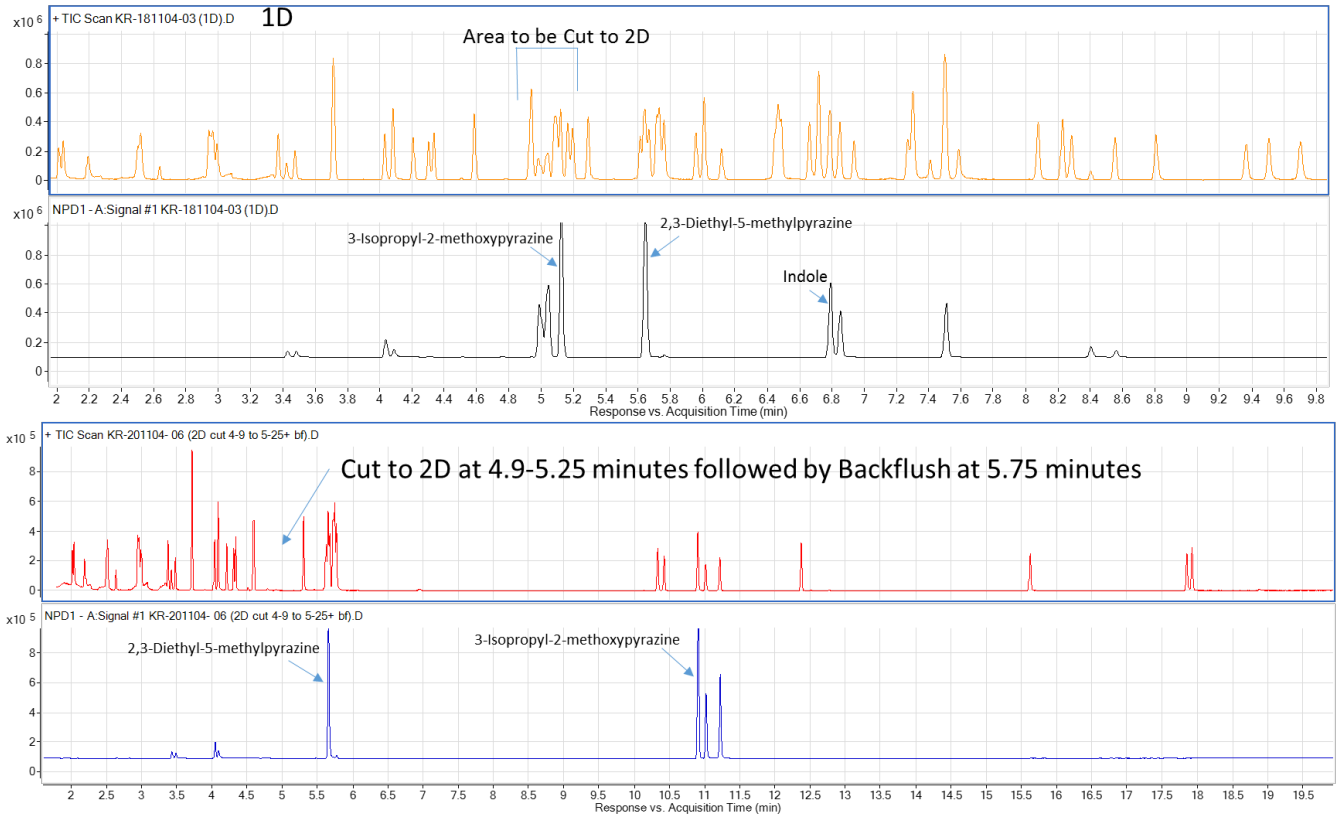

Figure 5 – The benefit of an element specific detector alongside the MSD

Discussion

This application note shows how in combination with specific detectors, heartcut 1D/2D can be used to characterize aroma active compounds and provide increased separation, without the need for extended chromatographic runs. This was also illustrated in a recent paper by Kikuo Sasamoto and Nobuo Ochiai[1].

The benefit of the GERSTEL 1D/2D system is that one chromatogram is obtained containing compounds from both the first and, (if used), second dimension separations.

References

1. J Chrom A 1217 (2010) 2903-2910