



Thermal Extraction of VOCs using a Multi-ramp Method with a CDS Pyroprobe 6150

Application Note

Fragrance, Food & Flavor

Abstract

This application note demonstrates a quick temperature ramp technique to improve the focusing of VOCs without a need for trapping.

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Introduction

The CDS Pyroprobe proportional–integral–derivative (PID) controller is an analog circuit designed to quickly ramp sample temperatures above 600°C for rapid pyrolysis without temperature overshoot. A new feature in the 6000 Pyroprobe institutes a multi-ramp tool, which allows for up to 3 temperature ramps per method. This can be used to reach a fast equilibration time at low temperatures, which helps in the analysis of VOCs without sorbent trapping.

In this application, the multi-ramp feature was used to create an ultra-quick heating for thermal extraction of VOCs. Compounds with retention times under 10 minutes are traditionally collected onto an a sorbent trap for refocusing. Due to a quick ramp technique used with the Pyroprobe multi-ramp feature, in conjunction with the Pyroprobe's low dead volume, under 30 μL , early eluting compounds can be extracted directly to the GC-MS while retaining a sharp peak shape.

Experimental

Ethyl Butyrate was dissolved in methanol at a concentration of 24 $\mu\text{g}/\mu\text{L}$, and 3 μL of the solution was added to a Drop-In-Sample Chamber (DISC) tube. This was analyzed using a quick ramp technique, and compared to a single ramp using a 6150 Pyroprobe connected to a GC-MS.

Single Ramp

Pyroprobe
DISC Chamber:
Final 1: 80°C 60s
Final 2: none

Interface: 300°C
Transfer Line: 325°C
Valve Oven: 300°C

Multi-Ramp

Pyroprobe
DISC Chamber:
Final 1: 1300°C 1.6s
Final 2: 80°C 60s

Interface: 300°C
Transfer Line: 325°C
Valve Oven: 300°C

GC-MS

Column: 5% phenyl (30m x 0.25mm)
Carrier: Helium 1.25mL/min
50:1 split
Injector: 360°C
Oven: 40°C for 2 minutes
12°C/min to 320°C
hold 15min
Ion Source: 230°C
Mass Range: 35-600amu

Results & Discussion

Ethyl butyrate, which is used as a fruity flavor in some foods, has a boiling point of 121°C and elutes between 6 and 7 minutes on a 5% phenyl column. This compound was used to study a quick ramp technique for thermal extraction. Figure 1 shows ethyl butyrate performed with a single ramp extraction at 80°C for 60 seconds directly to the GC. Here the peak width at half-maximum height (FWHM) is 0.23 minutes.

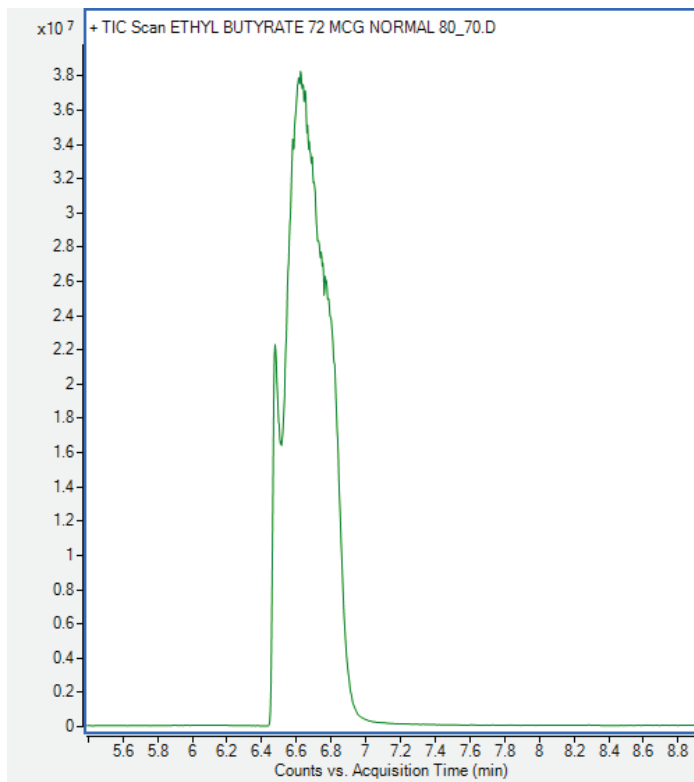


Figure 1. Ethyl Butyrate at 80°C using a single ramp.

The new feature of the Pyroprobe 6000 has the capability of performing up to 3 final temperature setpoints per method. Figure 2 shows the Pyroprobe Screen in the software demonstrating the capability of adding two additional ramps.

The proper combination of two temperature setpoints can result in an accelerated ramp to 80°C. By using 1300°C with a time of 1.6 seconds for the first setpoint, the sample will be heated at the most rapid rate to reach 80°C. A second setpoint of 80°C for 60 seconds allows the thermal extraction to complete. Figure 3 shows ethyl butyrate thermally extracted at 80°C using this multi-ramp technique. The FWHM decreased to 0.06 minutes, a nearly four-fold improvement of peak resolution. The quick temperature ramp and low dead volume of the Pyroprobe allowed for the immediate release of the volatile compound to be correctly synchronized for GC-MS analysis without the need for a sorbent trap.

Other applications also benefit from multiple heating steps per method. A “puff” of a cigarette or e-cigarette goes through a series of heating steps¹. Plus certain regulatory methods may

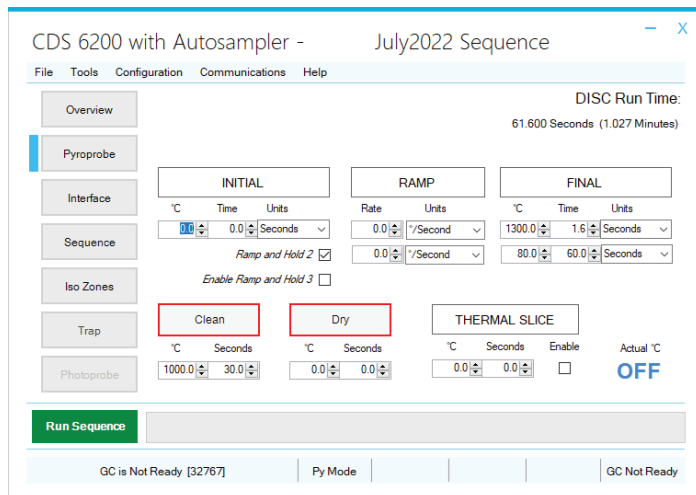


Figure 2. Pyroprobe software showing the multiple ramp capability.

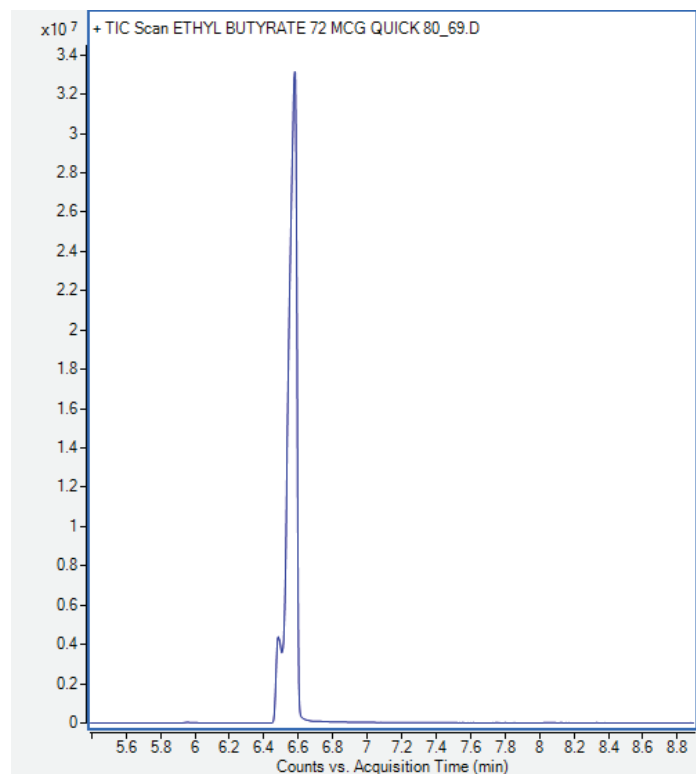


Figure 3. Ethyl Butyrate at 80°C using a multi-ramp.

require multiple heating ramps; IEC method 62321-8 requires 2 heating ramps for the extraction of phthalates in electronics²

In addition, by controlling the GC ready and GC start in each method, the Pyroprobe can also “stitch” multiple methods together to run in one GC run³. Combining these two features could provide the analyst with up to 30 heating ramps for one single GC run.

Conclusion

The Pyroprobe 6000 Series can perform up to 3 temperature ramps per method using the new multi-ramp feature. This flexibility,

provides an accelerated heat ramp for extraction of volatiles. The combination of two temperature setpoints, 1300°C for 1.6 seconds, followed by 80°C for 60 seconds, results in a direct thermal extraction of early eluting compounds, retaining peak shape without the need for a sorbent trap for re-focusing.

References

1. B Eitzinger, B., Pirker, S., "Numerical Simulation of a Cigarette during Smoking", January 2014, Contributions to Tobacco Research 21(7) pp. 403-416.
2. IEC 62321-8 Determination of certain substances in electrotechnical products –Part 8: Phthalates in polymers by gas chromatography-mass spectrometry (GC-MS), gas chromatography-mass spectrometry using a pyrolyzer/thermal desorption accessory (Py/TD-GC-MS).
3. K. Sam, Phthalate Calibration and Reproducibility with a Reference Polymer Material using an IEC Standard Method, CDS App Note #253.