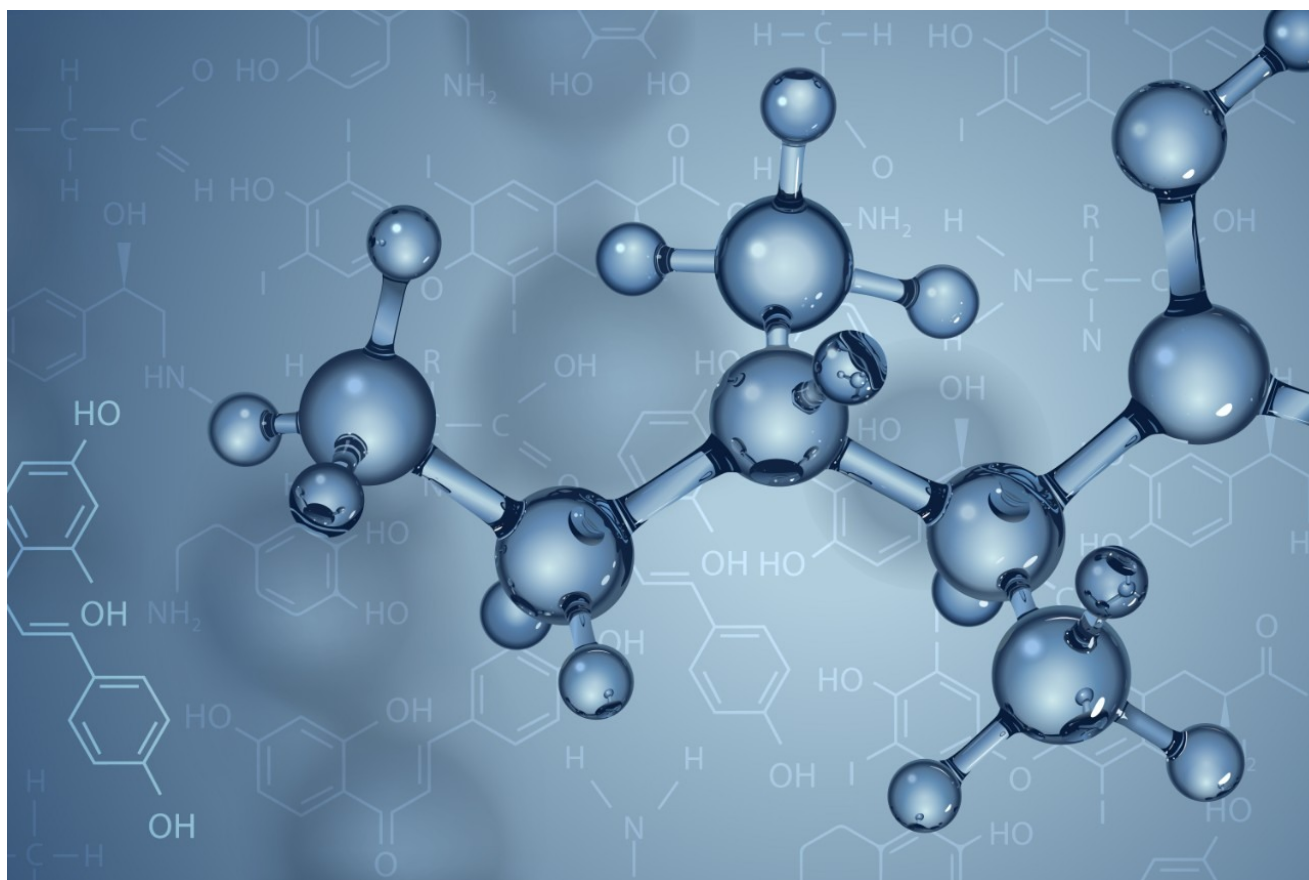


Parallel Dual Column GC-MS with the AutoSpec Ultima NT

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Abstract

This application brief describes an analytical method developed for the analysis of dioxins, furans, and PCBs.

Introduction

The Waters Micromass AutoSpec Ultima NT incorporates as standard a dual GC interface allowing the installation and use of two GC columns simultaneously. The high capacity pumping configuration of the AutoSpec Ultima NT deals with the elevated carrier gas flows encountered during dual column operation. An additional benefit is that a GC column can be removed and changed without venting the instrument, reducing instrument downtime and increasing productivity.

The dual column capability of the AutoSpec Ultima NT enables the development of methods that can significantly increase productivity.

This application brief describes an analytical method developed for the analysis of dioxins, furans, and PCBs.¹ Polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and dioxin-like polychlorinated Biphenyls (DLPCBs) have been determined (as identified by the World Health Organisation (WHO)) in one analytical run on two columns. This method reduces the analysis time from 2 hours to approximately 40 minutes.

Experimental

Configuration

For dual column operation the GC is fitted with two injectors and two GC columns. In combination with autosamplers this gives the possibility of using two columns of different phases or dimensions (if required) for:

- (1) Automated injection with choice between two columns
- (2) Automated simultaneous injections on two columns
- (3) Automated staggered injections on two columns

The MassLynx Software for AutoSpec Ultima NT incorporates integrated GC (Agilent 6890) and autosampler (Agilent 7683, CTC-GCPal and CombiPal) control. The GC can be configured with two injectors, two Agilent 7683 autosampler turrets and one sample tray; these can be controlled simultaneously allowing operation as described in (1) and (2). The alternative is use of the CTC-GCPal with two injectors, this will allow operation as in (1) and (3),

and for example giving the option of staggered injection.



Waters Micromass AutoSpec Ultima NT.

Results and Discussion

The GC was configured as previously described and the standard sensitivity specification test was performed and achieved under standard conditions on each injector. The standard specification is signal-to-noise >125:1 for 100 fg 2,3,7,8-TCDD co-injected with 1pg $^{13}\text{C}_{12}$ -2,3,7,8-TCDD monitoring two 'native' and two 'labelled' channels, plus lock-mass and lock-mass check, at 10,000 resolution using a 30m, 0.18 mm id, 180 μm DB5 column.

This was then repeated and the specification (signal-to-noise >125:1) was achieved separately with two different columns (40 m, 0.20 mm id, 180 μm and 20m, 0.18 mm id, 180 μm). These columns were chosen due to their suitability for the analysis of dioxins, furans and PCBs in one analytical run (described later).

To compare single column and dual column performance both columns were then interfaced into the instrument ion source. The standard specification was then repeated injecting simultaneously onto both columns.

In this dual column mode, a variety of flow rates were set to investigate the effect of carrier gas flow on sensitivity.

Figures 1 and 2 show the results of the sensitivity measurements in the dual column mode i.e. injecting onto the 20 m and 40 m columns simultaneously. Figure 1 is with 1 mL/min and Figure 2 with 1.8 mL/min total helium flow respectively. It can be seen that in each case the signal-to-noise values obtained are comparable and easily meet the specification criteria. The ability of the AutoSpec Ultima NT to operate at high carrier gas flow rates without

compromising performance is due to its high pumping capacity, giving flexibility for dual column method development.

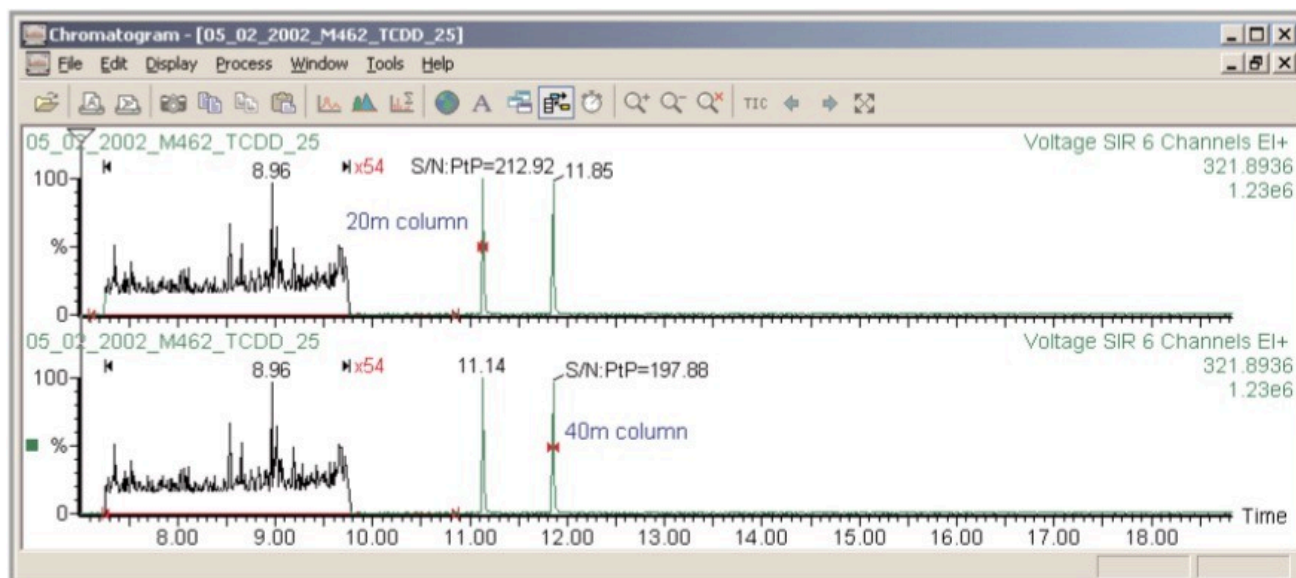


Figure 1. Dual column sensitivity at 1 mL/min He flow.

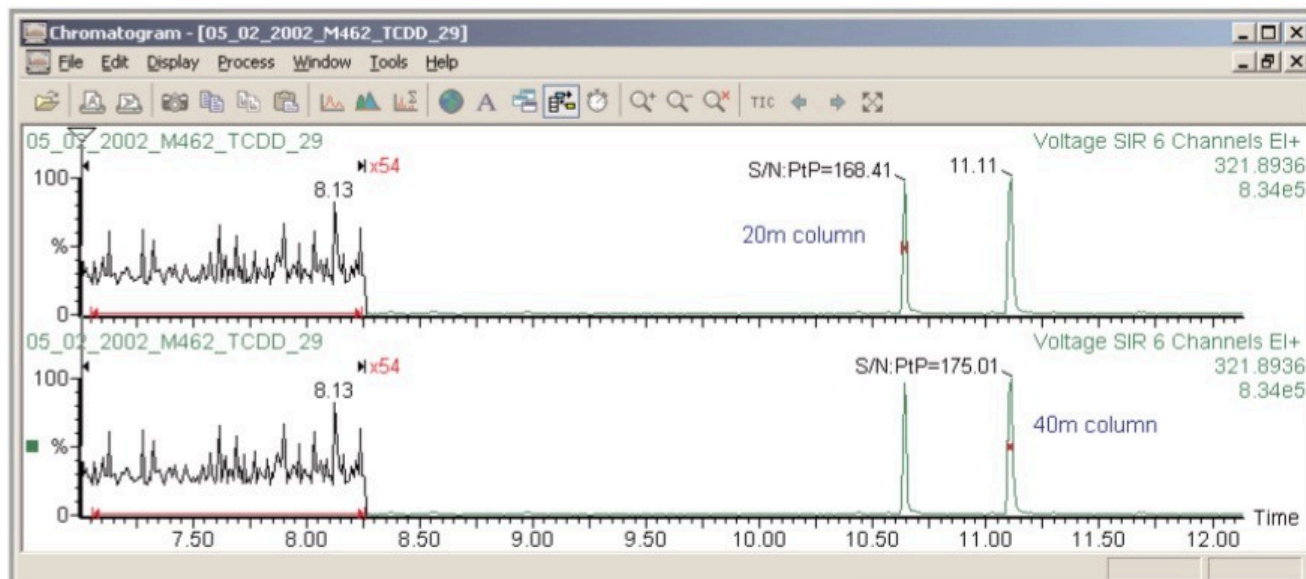


Figure 2. Dual column sensitivity at 1.8 mL/min He flow.

Dual Column Performance: Quantification

The quantitative performance of the instrument in dual column mode was evaluated by running standard calibration curves for 16 dioxins and furans and 12 PCBs simultaneously in one analytical run on two columns. The results show excellent quantitative linearity; calibration curves for 2,3,7,8-TCDD and PCB118

from 0.25 to 100 pg/g and 0.5-200 pg/g respectively are shown in Figures 3 and 4.

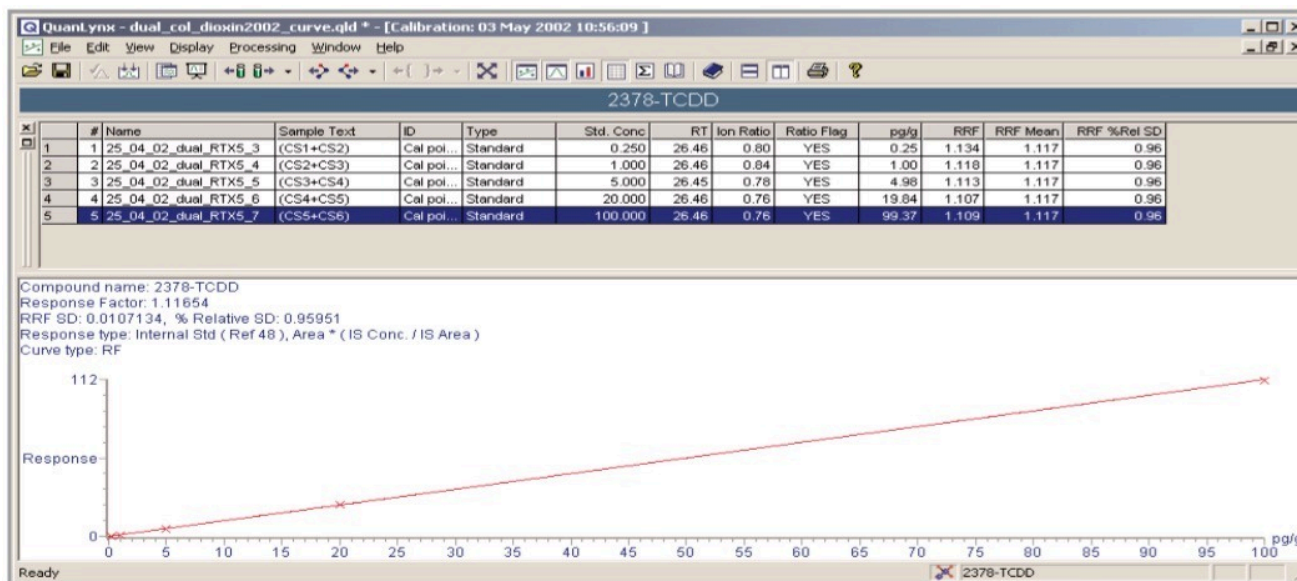


Figure 3. Dual column calibration curve for 2,3,7,8-TCDD.

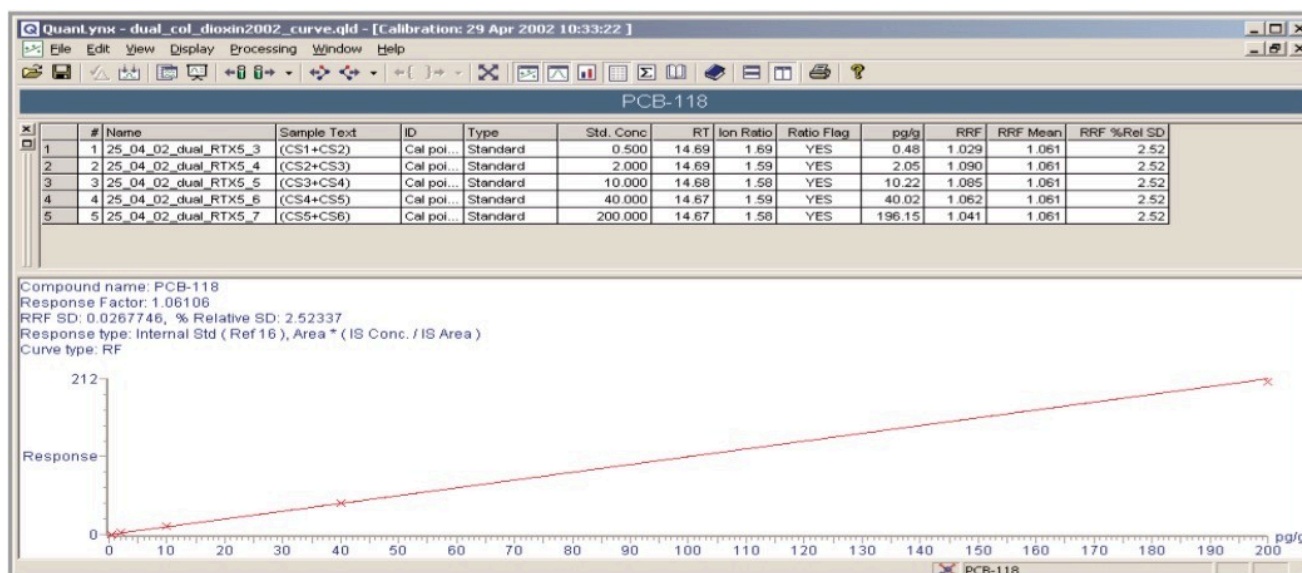


Figure 4. Dual column calibration curve for PCB 118.

Further work has been done on dioxins, furans and PCBs and is available as poster reprint, "Method for the Simultaneous Analysis of PCDD/Fs and DLPCBs Using Dual Microbore Column GC-HRMS in Sinterash Samples, Allowing the Determination of 'Totals' Group Concentrations", (WMP 240) from www.waters.com <
<http://www.waters.com>>.

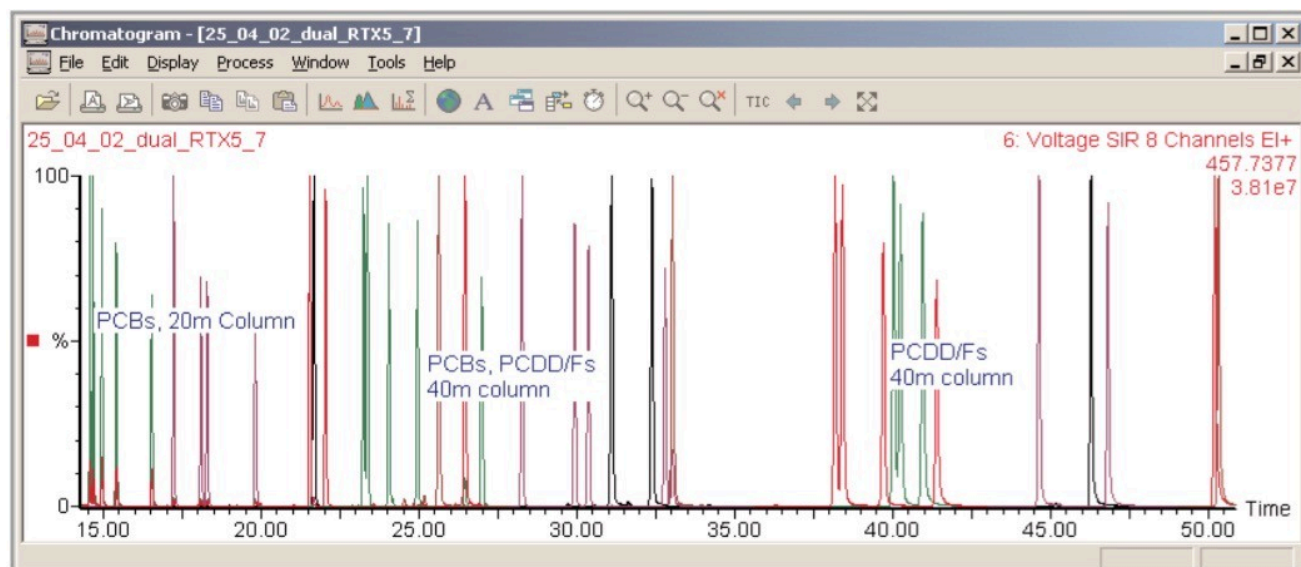


Figure 5. Dual column analysis of Dioxins, Furans, and PCBs.

Conclusion

The dual column GC interface and high pumping capacity of the AutoSpec Ultima NT offers the opportunity to develop methods to increase productivity by combining analyses which would normally require separate lengthy single runs. Operating with two GC columns installed at normal operating carrier gas flows does not compromise the sensitivity or quantitative linearity of the AutoSpec Ultima NT.

References

1. K.A.MacPherson, E.J.Reiner and T.M.Kolic, Dual Microbore Column GC-HRMS Analysis of Polychlorinated Dibenzo-Pdioxins (PCDDs), Polychlorinated Dibenzofurans (PCDFs) and Dioxin-Like Polychlorinated Biphenyls (DLPCBs). *Dioxin* 2001.

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