PLOT PT GC Columns with Integral Particle Traps Separate Gases Without Particle Shedding

Application Note

Energy and Chemicals

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Abstract

A variety of fixed gases, from hydrogen to C_4 (butane), are separated under conditions likely to generate particle shedding using thermal and flow rate fluctuation. A comparison of Porous Layer Open Tubular (PLOT) columns with no particle trap, user-prepared glass wool traps, manufacturer-attached press-tight traps, and integrated particle traps is demonstrated. The integrated particle traps show no discernible spikes, indicating effective particle retention by the trapping ends. In addition, the permanently integrated traps have no potential for leaks and permit trimming of the ends if needed during maintenance.

Agilent Technologies

Introduction

PLOT columns have replaced their packed column counterparts in all aspects of fixed-gas separation [1]. Since their introduction, over 30 years ago, the number of phases available now allows for all separations to be achieved in less time and with tremendous efficiency [2]. However, PLOT column technology has had obstacles to overcome related to particle shedding of the various PLOT phases under various column oven conditions. These obstacles give rise to analytical system delays associated with inferior data quality, plugged switching valves, or even damage to rotary valve systems caused by abrasion of critical surfaces. Many end users have connected particle trapping devices as a simple section of capillary column with a polysiloxane phase coating thick enough to retain lost particles, or more elaborate glass wool plugs similar to those used to pack GC columns [3]. These trapping devices are typically connected to the PLOT column with “press-fit” or low-dead-volume metal connectors, both of which have a potential for leaks. Agilent was the first manufacturer to introduce integrated particle traps on both ends of the PLOT column. These columns are ideally suited to retain particles and, by eliminating leak sources, improve uptime dramatically. A series of Agilent J&W PLOT PT columns (PT means Particle Trap) is evaluated here, which show no observable spikes related to particle shedding. They are compared to standard PLOT columns with no trap, user-prepared traps, and press-tight connected traps.

Materials and Methods

An Agilent 7890A GC was fitted to an Agilent 7697A Headspace Unit and either an FID or TCD detector. Evaluation mixes were obtained from Scott Specialty Gases, Plumsteadville PA, and were 15 ppm nominal for hydrocarbons and 1% for fixed gases in 99.999% nitrogen balance.

Sample preparation

A blank headspace 20-mL vial was sealed, and a pair of 26-gauge needles were inserted with one used as an inlet and the other as a vent (Figure 1). Gas was discharged into the headspace vial allowing pressure to build and vent. This was repeated three times to ensure reasonable cycle purging prior to autosampler injection.

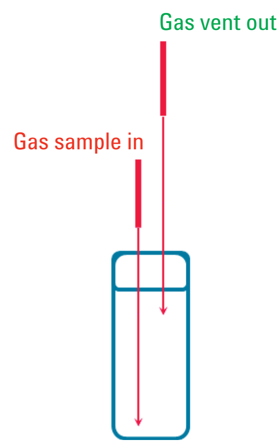

Figure 1. Gas introduction to headspace vial for sampling of fixed gases and C_1 to C_4 mixes.

Conditions

Columns:	See Table 1
Carrier:	Helium with varying flow depending on column phase and dimensions (Table 1) in constant flow mode
Headspace unit:	Oven 40 °C, valve 50 °C, transfer line 60 °C, flow rate 5 or 10 mL/min depending on column dimension, 10 mL/min used for megabore columns
Detector:	FID or TCD at 250 °C
Injection volume:	0.1 mL loop fitted to inlet valve of headspace unit
GC inlet:	Split mode at 5:1, typically at 70 °C or higher depending on column oven initial conditions

A rapid cool-down of the column (common with most modern GC ovens) was used to expose the column to high-velocity fan air, causing it to modestly shift/shake in the oven at the end of each run, thereby generating particles.

Table 1. Columns and conditions.

Agilent J&W GC column	Dimensions	Part number	Flow rate (mL/min)	Oven temperature program
HP-PLOT Al_2O_3 KCl	30 m × 0.53 mm, 15 µm film	19095P-K23	3	120 °C isothermal
CP-Al_2O_3/Na_2SO_4	50 m × 0.53 mm, 10 µm film	CP7568	4.7	130 °C isothermal
HP-PLOT Al_2O_3 S	30 m × 0.53 mm, 15 µm film	19095P-S23	3	120 °C isothermal
CP-Molsieve 5Å PT	25 m × 0.53 mm, 50 µm film	CP7538PT	3	80 °C isothermal
PoraPLOT Q PT	10 m × 0.32 mm, 10 µm film	CP7550PT	1	50 °C (5 minutes) then to 120 °C at 50 °C/min, hold 4.6 minutes
PoraPLOT U PT	25 m × 0.53 mm, 20 µm film	CP7584PT	2	85 °C isotherm
HP-PLOT Al_2O_3 KCl PT	50 m × 0.53 mm, 15 µm film	19095P-K25PT	3	100 °C (10 minutes) then to 120 °C at 30 °C/min, hold 3 minutes

Particle traps

- **User-prepared:** A small piece of clean glass wool (0.5 × 0.5 cm) was placed into the void space of an Agilent Ultra Inert CFT union. One end was fitted with a 25-cm piece of deactivated fused silica tubing (0.53-mm id), the other end was fitted to the column outlet.
- **Manufacturer-prepared:** A 2.5-m section of thick film phase, 100% methyl polysiloxane, was fitted to the column end with a press-tight connector.
- **Integrated pre-attached:** A 2.5-m section of thick film phase, 100% methyl polysiloxane, was incorporated during column manufacture from a single blank of fused silica tubing.

Supplies

Headspace vials:	20 mL (p/n 5182-0837), caps (p/n 5190-3987)
Electronic crimper:	20 mm (p/n 5190-3189), decapper (p/n 5040-4671)
TCD ferrules:	Front, 0.53 mm (p/n 5182-9676), 0.32 mm (p/n 5182-9677), back (p/n 5182-3477)
Hydrocarbon mix:	15 ppm each in nitrogen, 1 × 4 L, Scott Specialty Gas (p/n 22566) (Sigma-Aldrich Corp.)
Fixed gases mix:	1% each in nitrogen, 1 × 4 L, Scott Specialty Gas (p/n 501670) (Sigma-Aldrich Corp.)

Discharge/vent needles are supplied attached to the 4-L reference mixes.

Results and Discussion

As seen in Figure 2, PLOT stationary phase particle shedding occurs without a trap installed during operation. Figures 3 and 4 demonstrate that a user-made glass wool plug can still elute some particles during operation. Figure 5 shows an overlay of two fixed-gas separations, one with dual integrated particle traps and one with the traps removed.

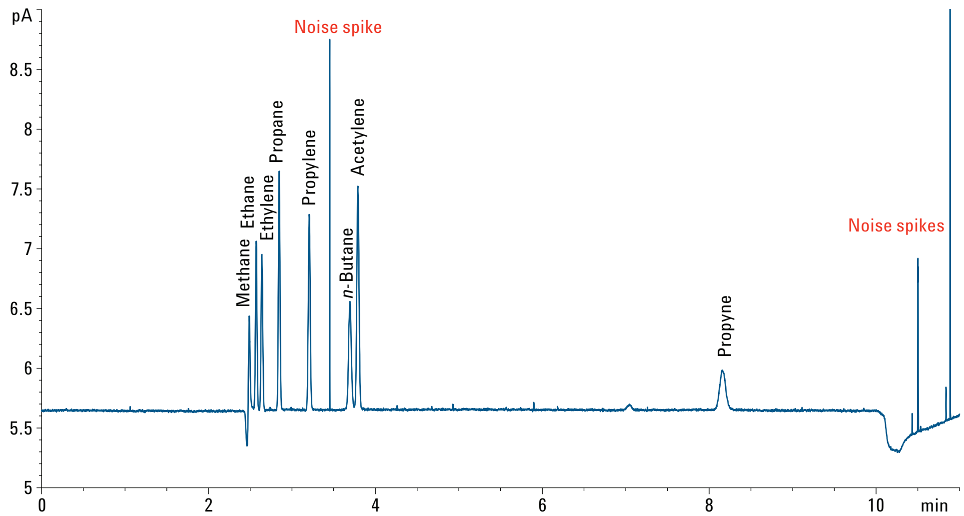

Figure 2. C_1 to C_4 hydrocarbon mix on Agilent J&W HP-PLOT Al_2O_3 KCl, 30 m × 0.53 mm, 15 µm film, without a trap, showing spikes during and at end of run on rapid cool-down on FID.

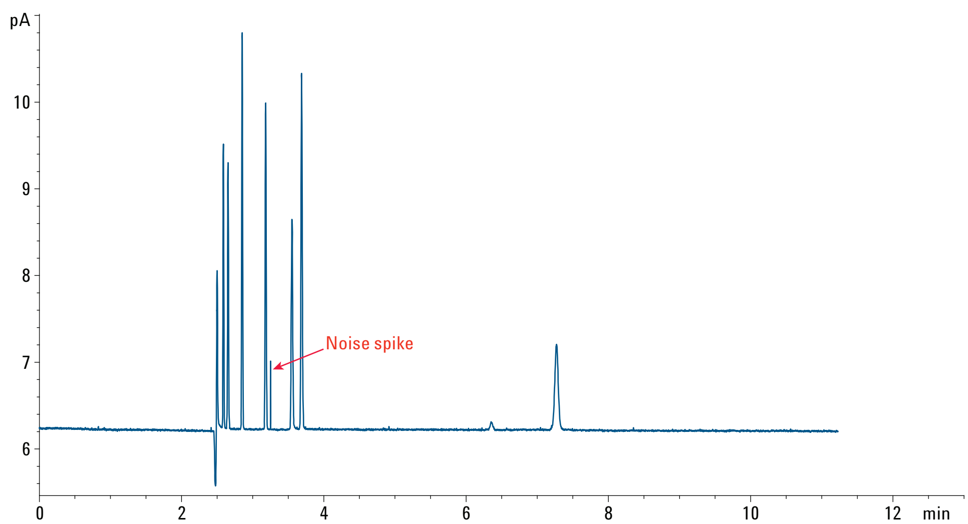

Figure 3. C_1 to C_4 hydrocarbon mix on Agilent J&W CP-PLOT Al_2O_3/Na_2SO_4, 50 m × 0.53 mm, 10 µm film, with a user-made glass-wool trap showing spikes during run still occurring on FID.

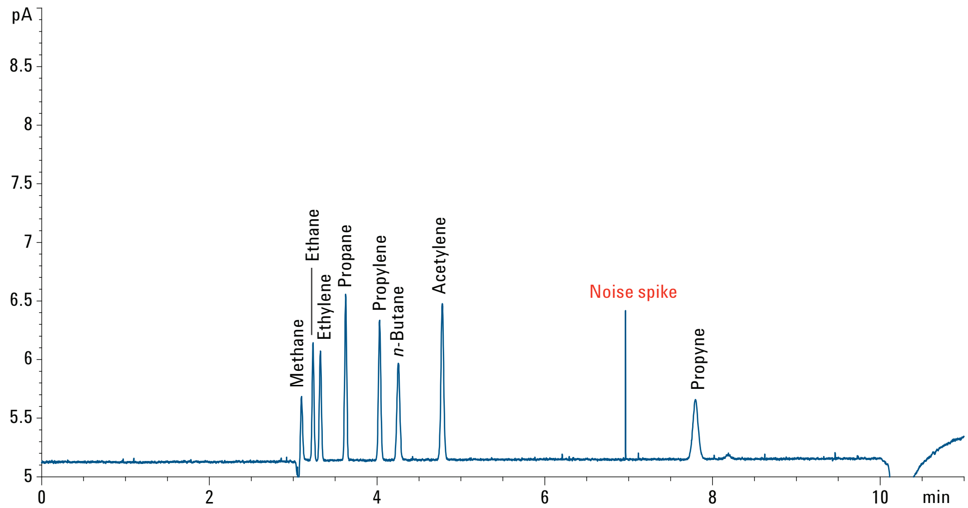

Figure 4. C_1 to C_4 hydrocarbon mix on Agilent J&W HP-PLOT Al_2O_3 S, 30 m × 0.53 mm, 15 µm film, showing occasional spikes with user-made glass-wool trap on FID.

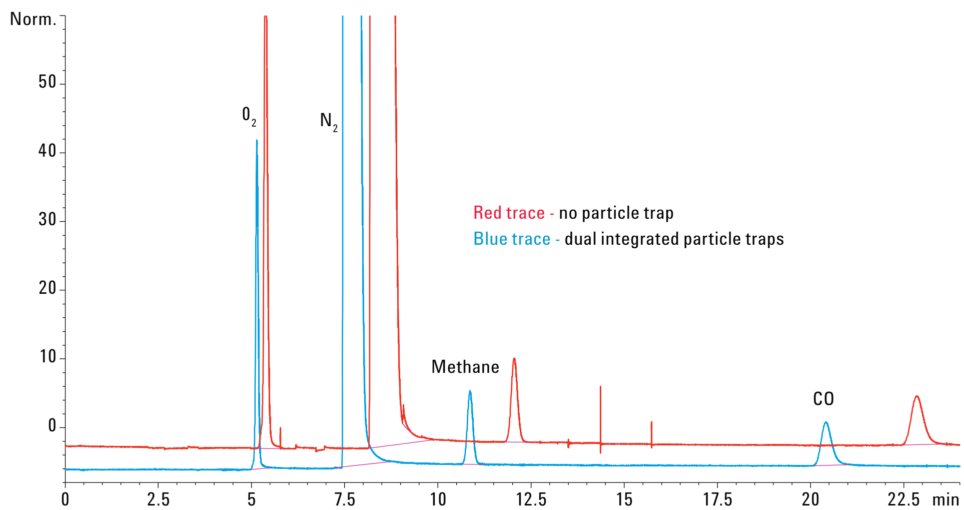

Figure 5. Fixed gases mix on Agilent J&W CP-Molsieve 5Å, 25 m × 0.53 mm, showing spikes when particle traps are removed (red trace) and no spikes with manufacturer dual-ended integrated traps (blue trace) on TCD.

Figures 6, 7, and 8 show that the integrated dual-ended particle trap technology completely removes particle shedding, affording greater reliability, especially for multicolumn installations that use valves for backflush or heart cutting techniques to optimize separations of complex mixtures. Integrated PT columns provide the ability to trim the ends as needed for long column life, and, overall, improve ease-of-use. While thick film particle traps can be installed with press tight connections they are prone to leaks requiring ongoing maintenance of the connectors. In addition, over time, stationary phase particles can accumulate in the union, which can cause changes in flow restriction.

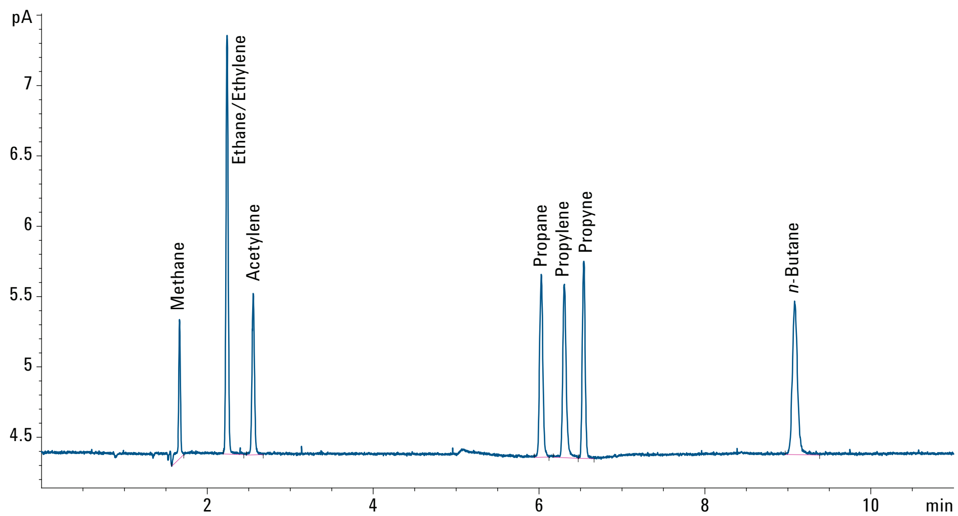

Figure 6. C_1 to C_4 hydrocarbon mix on Agilent J&W PoraPLOT Q PT, 10 m × 0.32 mm, with attached manufacturer-prepared integrated dual-ended particle trap, showing the absence of particles or spikes on FID.

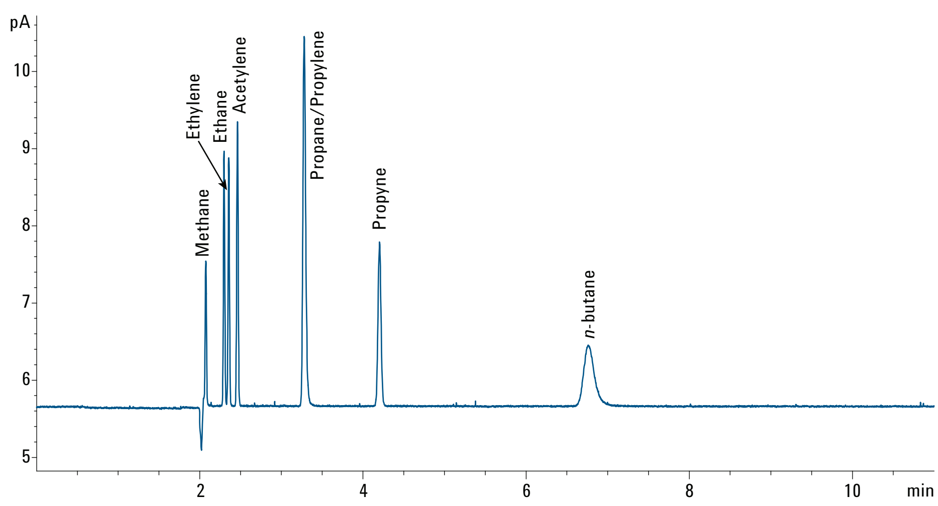

Figure 7. C_1 to C_4 hydrocarbon mix on Agilent J&W CP-PoraPLOT U PT, 25 m × 0.53 mm, 20 µm film, with attached manufacturer-prepared integrated dual-ended particle trap, showing the lack of particles or spikes on FID.

Conclusions

Agilent J&W PLOT PT GC columns with integrated thick-film particle traps provide a reliable leak-free means to reduce particle shedding during PLOT column operation, permitting more robust analysis of a broad range of gas samples. Whether the challenge involves recycled refrigerant recovery [4] or separating oxygen from argon [5] in a cryogenic process, Agilent J&W PLOT PT columns help to remove chromatographic spikes and eliminate the need to install particle traps through external connections. The Agilent J&W line of dual integrated particle-trap PLOT columns is an excellent choice for applications using valve switching and multiple detection strategies, or both.

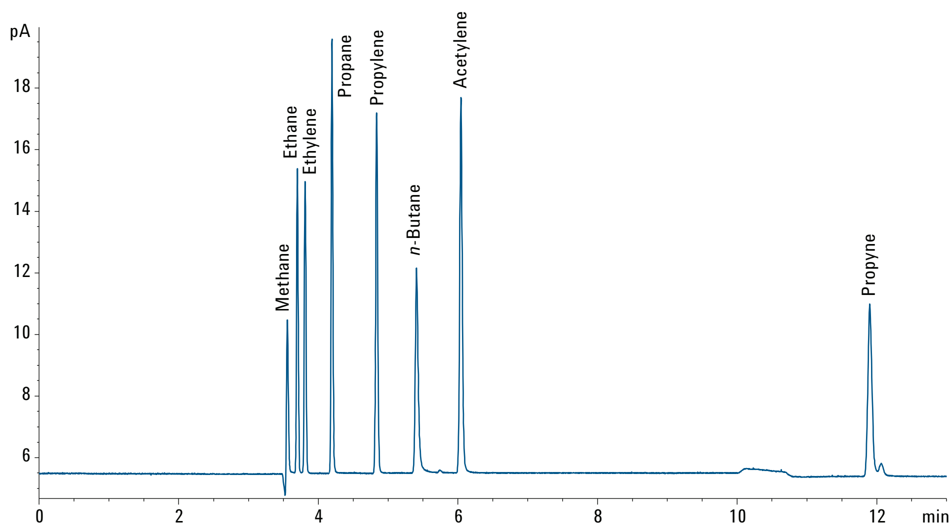

Figure 8. C_1 to C_4 hydrocarbon mix on Agilent J&W HP-PLOT Al_2O_3 KCl PT, 50 m × 0.53 mm, 15 µm film, with integrated dual-ended particle trap, showing lack of particles or spikes on FID.

References

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Printed in the USA
August 20, 2013
5991-2975EN

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