

Capillary Flow Technology: Deans Switch

INCREASE THE RESOLVING POWER OF YOUR GC

The Measure of Confidence



Agilent Technologies

A practical solution for increasing selectivity in complex matrices



Complex sample matrices often have too many overlapping compounds to allow for proper resolution of the compound or compounds of interest. In these cases, additional selectivity is needed. Agilent offers a variety of solutions to the problem of interference from coeluting peaks in GC and to improve peak capacity, including Triple Quadrupole GC/MS, GC/Q-TOF, GCxGC, a family of selective detectors, and the Deans Switch. Choosing the proper tool is application-specific and based on the number and types of peaks to be analyzed.

The Deans Switch is an easy and affordable way to further resolve multiple components in complex matrices

The Deans Switch is used to perform **in-oven** heart-cutting or multidimensional GC (MDGC). In heart-cutting, the effluent from one column is diverted to a second column having a different stationary phase at specific times and for specific durations in the chromatographic run. The analyte of interest and any co-eluting impurities in the diverted peak are fully separated on the second column. Using this technique, analysts can determine trace components in a complex mixture or increase the resolving power of their GC by using two different columns in the same analytical run and on the same instrument. Additional selectivity can be achieved by using two different detectors.

Deans Switch technology has been in use since 1967. But the typical configurations that employ traditional rotary valves, stainless-steel tubing and fittings that have a high thermal mass and do not track oven temperature very well, are susceptible to leaks over time and can cause peak broadening.

Agilent has eliminated these chromatographic problems with its inert Capillary Flow Technology Deans Switch. Agilent's Deans Switch combines individual switch components into a single, smaller device that provides all the benefits of increased resolution, throughput, uptime, productivity, and accurate results of MDGC thanks to:

- Low thermal mass that allows the Deans Switch to closely follow the oven ramp
- Small, well-swept dead volumes that eliminate peak broadening
- Metal ferrules and fittings that eliminate leaks, even after many oven temperature cycles
- Inert surfaces throughout that prevent peak tailing or loss of analytes
- Backflush capabilities that can significantly reduce run times and prolong column life



How it works...

INCREASE SELECTIVITY SIMPLY AND EFFECTIVELY

The following illustrations (**figures 1, 2, and 3**) demonstrate how the Deans switch reliably directs effluent to the selected column to quickly and easily separate and quantify the compounds of interest.

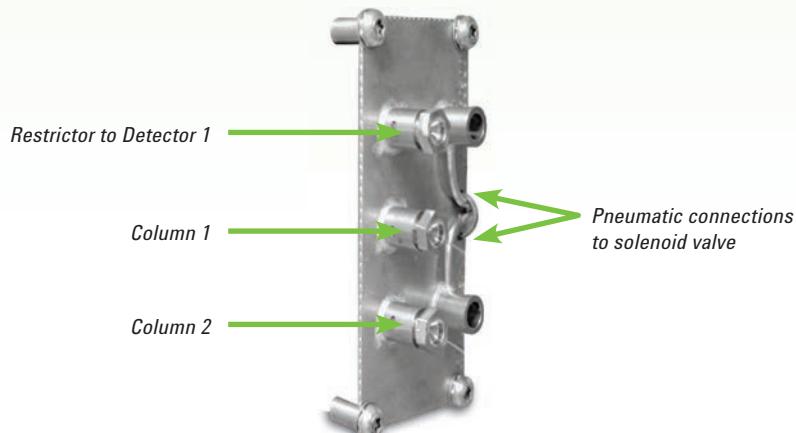


Figure 1. Photo of Capillary Flow Technology Deans Switch calling out its various connections.

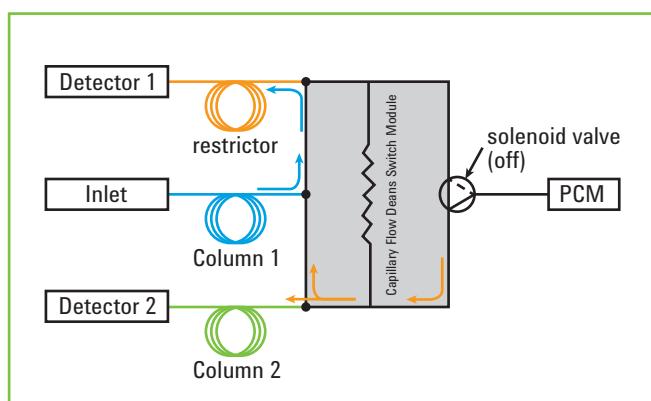


Figure 2. The effluent from column 1 goes directly to detector 1 through the short restrictor when the Deans Switch is in the "no cut" position.

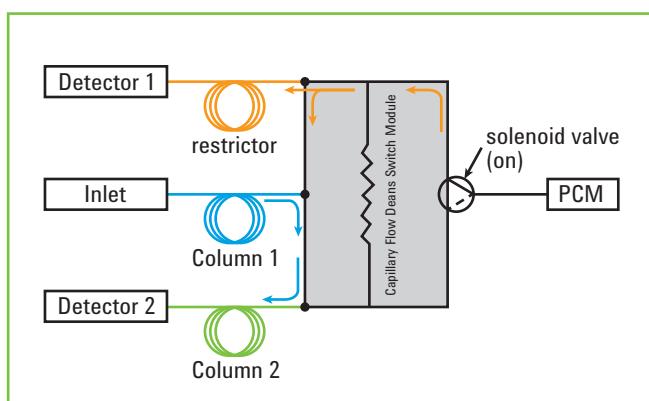


Figure 3. The effluent from column 1 is diverted to column 2 and detector 2 when the Deans Switch is in the "cut" position.

Note: A purged capillary flow device, such as the Deans Switch, introduces an additional flow in the sample stream. For detectors that operate at low flow rates, like the MSD and TCD, some decrease in sensitivity will occur.

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Increase throughput by cutting sample preparation time

Biodiesel is typically blended with petroleum diesel for commercial use to create a motor fuel with 1 to 20 volume percent total FAME content (designated as B1-B20). It is difficult to quantify the FAME content in these blends using conventional capillary GC due to the presence of the petroleum hydrocarbons. EN 14331 requires that the FAMEs and petroleum diesel be physically separated before analyzing the FAME fractions by GC. This is time-consuming and is only scoped for B5 or lower biodiesel blends.

Figures 4 and 5 show that using an Agilent 7890 GC system with a Capillary Flow Technology Deans Switch, FAME type and content can be determined in any diesel blend without the need for physical separation of the FAMEs prior to GC analysis. Because of the unsurpassed retention time stability of the 7890 GC with the Deans Switch, maximum productivity was realized by making multiple cuts during the same analytical run with no shift in retention times.

Analysis of Fatty Acid Methyl Ester (FAME) Content and Distribution in Biodiesel Blends Using Heart-Cutting 2D Gas Chromatography

Agilent Application Note 5989-8107EN

Industries:

Hydrocarbon Processing/Biofuels

Columns used:

Primary – Agilent HP-5ms,
15 m x 0.25 mm, 0.1 µm
(Agilent part number 19091S-331)

Secondary – Agilent HP-INNOWax,
30 m x 0.25 mm, 0.5 µm
(Agilent part number 19091N-233)

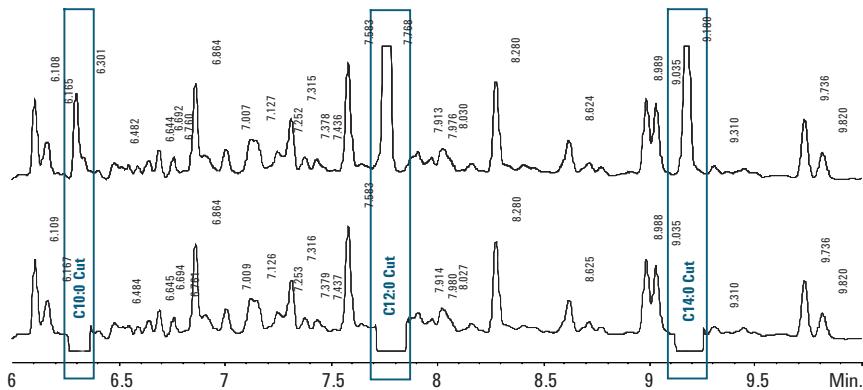


Figure 4. The top chromatogram shows the biodiesel separation on the primary column before the heart-cuts. The bottom shows separation after the heart-cuts. Note that after the first heart-cut of the C10:0 FAME, all subsequent eluting peaks have the same retention time on the primary column, allowing the precise heartcutting of the C12:0 and C14:0 FAMEs on the secondary column.

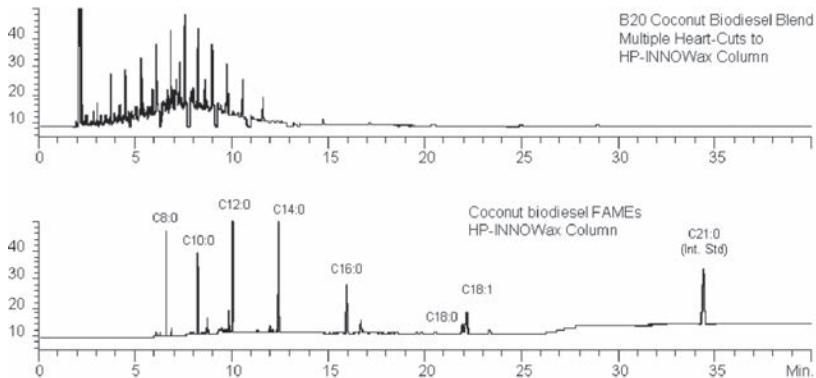


Figure 5. Complete separation of coconut FAMEs in B20 biodiesel blend using the Agilent Deans Switch. (The top chromatogram shows the cuts made on the primary HP-5ms column. The bottom chromatogram shows the separations achieved on the second HP-INNOWax column.)

Fish oil samples spiked with Aroclors or individual PCB congeners were diluted and injected directly into an Agilent 7890 GC with dual ECDs. No other sample preparation was performed. Seven cuts (**Figure 6**) were performed in the primary chromatogram to divert the target PCBs to a second column (**Figure 7**) where they were separated from other PCBs and matrix components that co-elute with them on the primary column. The Deans Switch can also be used to backflush later

eluting compounds (**Figure 8**). In this application, backflushing was employed to remove any remaining fish oil components that accumulate on the column and cause carryover and retention time shifts, even after a single run. Backflushing forces unwanted sample components backwards through the column and out through the inlet. This eliminates the need for lengthy bakeout routines, improves chromatographic performance, and significantly extends column life.

Direct Injection of Fish Oil for the GC/ECD Analysis of PCBs

Agilent Application Note 5989-6095EN

Industries:

Environmental, Foods,
Pharmaceuticals, Consumer Products

Columns used:

Primary – Agilent J&W DB-XLB,
30 m x 0.18 mm, 0.18 μ m
(Agilent part no 121-1232)

Secondary – Agilent J&W DB-200,
30 m x 0.25 mm, 0.50 μ m
(Agilent part no 121-2033)

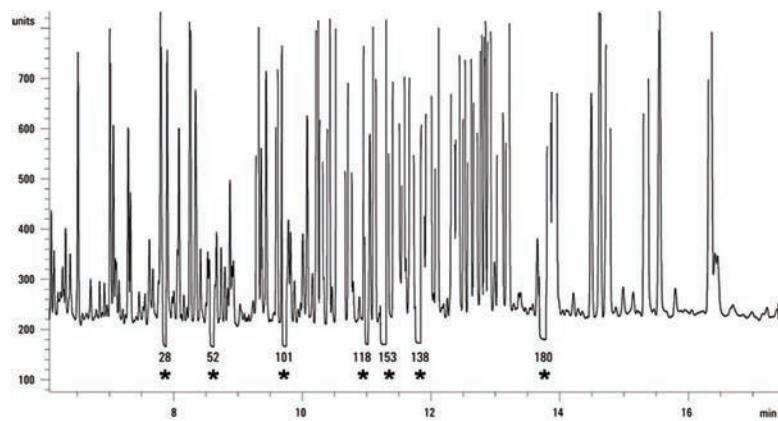


Figure 6. Asterisks indicate where heart-cuts were made for the seven target PCBs on the primary DB-XLB column. The unmatched retention time (RT) stability of Agilent GC systems with the Capillary Flow Technology Deans Switch means that the act of heart-cutting does not affect the RT of subsequent peaks in the primary column. This allows multiple exact heart-cuts to be made in a single run.

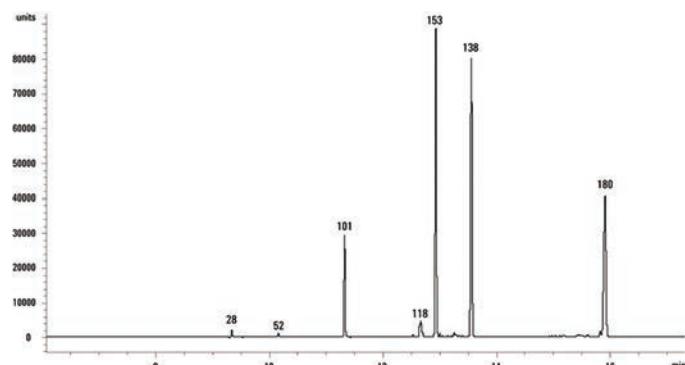


Figure 7. GC/ECD chromatogram from the second column (DB-200) that was heart-cut from the first column. Except for PCB congener 118, the target PCBs were resolved from co-eluting interferences.

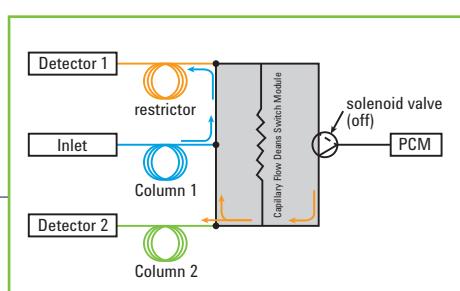


Figure 8. When the Deans Switch is in backflush mode, the inlet pressure is dropped to 0 or 1 psi, while the PCM pressure is raised to 80 psi.

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Save time and increase overall productivity

Using a Capillary Flow Deans Switch, both trace oxygenates and hydrocarbons are analyzed in a single run, thereby eliminating the need to conduct two separate analyses on different instruments. Light hydrocarbons are cut from the primary HP-INNOWax column to a secondary PLOT column. This ensures the necessary separation for each class of compound and prevents oxygenates from adsorbing on the PLOT column, protecting it from damage and increasing its useful lifetime.

In **Figure 9**, the top chromatogram shows the separation of the unresolved hydrocarbons from the oxygenates. The boxed area shows the cut time for the Deans Switch, where the hydrocarbons were diverted to the PLOT column. The bottom chromatogram shows the separation of the hydrocarbons that were cut from the INNOWax column.

Simultaneous Analysis of Trace Oxygenates and Hydrocarbons in Ethylene Feedstocks

Agilent Application Brief 5989-6082EN

Industries:

Hydrocarbon Processing,
Specialty Chemical

Columns used:

Primary – Agilent HP-INNOWax,
30 m x 0.32 mm, 0.50 µm
(Agilent part no 19091N-213)

Secondary – Agilent Alumina HP PLOT M,
30 m x 0.53 mm, 15 µm
(Agilent part no 19095P-M23)

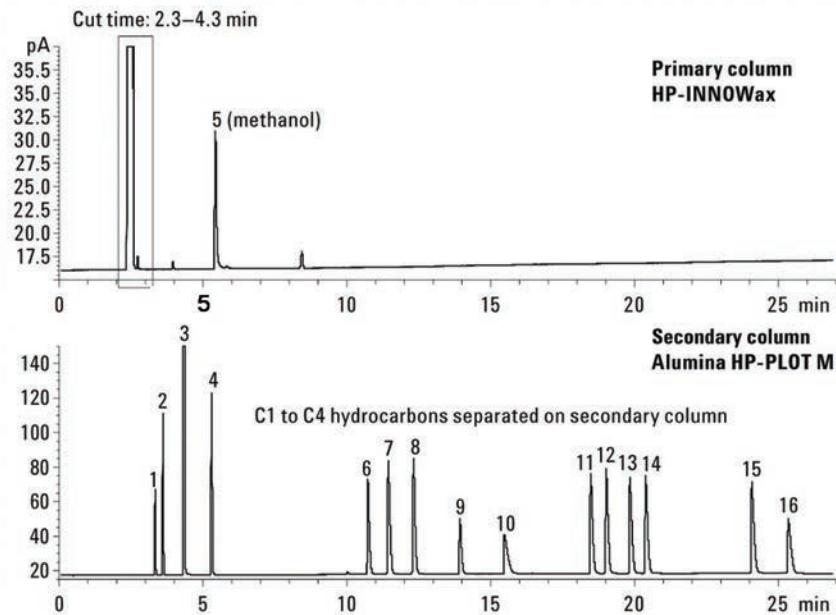


Figure 9. Separation of trace oxygenates and light hydrocarbons in ethylene.

When oral fluids are used for drug testing, mass spectrometry confirmation must detect drugs of abuse at concentrations lower than in urine testing. This is complicated by interferences in the complex sample matrix.

Rather than using more expensive techniques such as, GC/MS/MS or LC/MS/MS, GC/MS with a Deans Switch was used to provide the required selectivity and sensitivity for confirmation of THC in oral fluid.

Backflushing was used to remove any heavy materials at the head of the primary column. This offers significant benefits, including:

- Shorter analysis times
- Reduced need for source cleaning
- Increased column life
- Cleaner chromatographic baselines

As **Figures 10** and **11** demonstrate, MDGC with SIM mode detects THC in the oral fluid sample at the required sensitivity with no interference from other matrix components. Results showed very good agreement with those obtained using an ELISA immunoassay screening technique.

Confirmation of THC in Oral Fluids

Agilent Application Note 5989-5668EN

Industries:

Forensics, Drug Testing

Columns used:

Primary – Agilent J&W DB-1ms,
15 m x 0.25 mm, 0.25 µm
(Agilent part number 122-0112)

Secondary – Agilent J&W DB-17 ms,
15 m x 0.25 mm, 0.25 µm
(Agilent part number 122-4712)

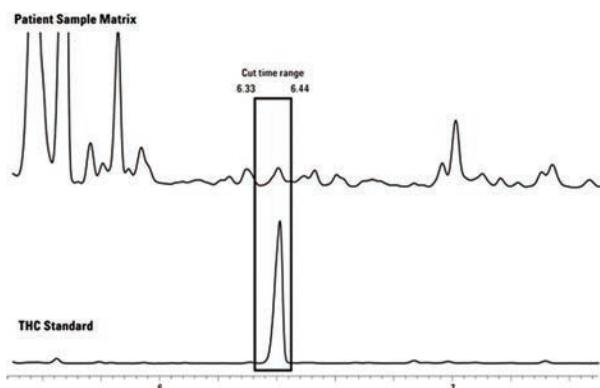


Figure 10. Expanded view of THC-TMS elution range with cut times.

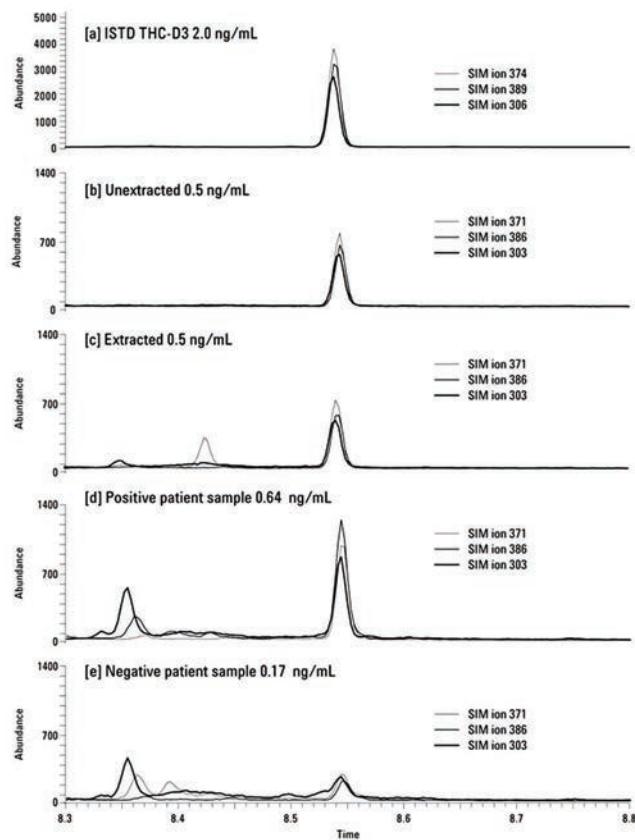
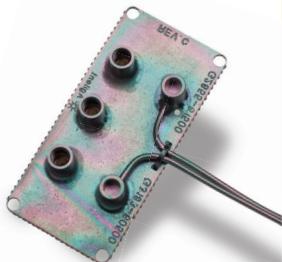


Figure 11. SIM ion chromatograms. (a) Internal standard THC-D3 at 2 ng/mL; (b) Unextracted THC cutoff at 0.5 ng/mL; (c) Extracted THC cutoff at 0.5 ng/mL; (d) Positive patient THC sample at 0.64 ng/mL; (e) Negative patient sample at 0.17 ng/mL.

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Here's everything you need to gain increased resolution



7890 GC or 6890N GC with two detectors and two columns

Deans Switch option or accessory

- On-site Installation and verification are strongly recommended.

Flow source – Pneumatics control module (PCM) (*recommended*)

- A PCM is recommended to monitor/control flow.
- An Aux electronic pneumatics control (EPC) may be used but only provides pressure control. If Aux EPC is used, order Aux EPC Purge Regulator Kit.

High-temperature SilTite ferrules and fittings*

Deactivated silica tubing for detector restrictor*

- Restrictor is needed to maintain equal pressure across the detector channels.
- Appropriate restrictor dimensions can be determined using the Deans Switch Calculator.

Dean Switch Calculator software*

- This tool allows the user to select or enter known factors (oven temperature, detector type, column dimensions, flow rates, etc.) and uses these to determine variables such as restrictor length and flow, pressure set points and so on.

* Included with the Deans Switch option and accessory.

For more information

Read: **Agilent G2855B Deans Switching Installation and Operation Manual, Publication Number G2855-90120**

Learn more about Agilent's Capillary Flow Technology at
agilent.com/chem/CapillaryFlowTechnology

Buy online or find an Agilent customer center in your country:
agilent.com/chem/contactus

U.S. and Canada: **1-800-227-9770** or agilent_inquiries@agilent.com

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Printed in the USA June 10, 2013

5989-9384EN



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