

Analysis of C1 to C10 Hydrocarbons Including C1 to C6 Paraffin/Olefin Isomer Separation at Ambient Temperature Using Dual FID and a Capillary Flow Technology Deans Switch

Application Note

Extended Hydrocarbon Analyzer

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Abstract

A gas chromatography method has been developed for the analysis of C1–C10 Hydrocarbons on a single injection, without cryogenic cooling, using a Capillary Flow Technology (CFT) Deans Switch. This application note describes a method for the high resolution analysis of C1–C6 paraffin and olefins, as well as an extended hydrocarbon analysis in both gas phase and liquid phase samples.



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Introduction

For the analysis of samples with a large boiling point range, nonpolar capillary columns that separate hydrocarbons by boiling point characteristics are typically used. Boiling point columns provide good resolution for the heavier hydrocarbon fraction. However, the resolution of the light hydrocarbon fraction suffers when C1 to C4 hydrocarbons are present in high concentrations. Boiling point columns are also not the columns of choice for the separation of C4/C5 olefins. PLOT columns provide enhanced separation for the light hydrocarbon fraction, but they exhibit recovery issues for the heavier fractions, making the analysis of the heavies difficult. A solution to this problem is to configure a CFT Deans Switch with a boiling point and a PLOT column. The boiling point column provides resolution for the heavy hydrocarbon fraction and the PLOT column provides resolution for the light hydrocarbon fraction.

For this analysis, an Agilent 7890A Gas Chromatograph was fitted with a 4-port liquid sample valve, in series with a 6-port gas sample valve, a split/splitless inlet, and a CFT Deans Switch, configured with a DB-1 (123-106B) and HP-PLOT Q (19091P-QQ4PT) column. The DB-1 was configured as the primary column and the HP-PLOT Q was configured as the secondary column with a short restrictor column opposite the secondary column, to balance the Deans Switch. In the Off position, the effluence of the DB-1 column was directed to the short fixed restrictor directly to the Back FID, while the HP-PLOT Q column carrier gas flow was delivered by the Deans Switch. When the Deans Switch was switched to ON, the effluence of the DB-1 column was directed through the HP-PLOT Q column to the front FID, while the fixed restrictor

carrier gas flow was delivered by the Deans Switch (Figure 1). This configuration allowed for a Heart Cut of the light fraction onto the HP-PLOT Q column for additional separation.

There are three ways to introduce samples with this configuration:

- Directly from a heated gas sample cylinder or directly from process through a gas sample valve
- Directly from a piston style pressurized liquid sample cylinder through a liquid sample valve
- Syringe injection directly into the GC inlet

This configuration allows for simultaneous separation of all components, while eliminating the possibility of contamination and adsorption of heavies, on the HP-PLOT Q column.

Application

This specific application required the measurement of C1 through C10 in ethylene and propylene to 1 ppm. Resolution of low-level ethane and propane as well as heavy hydrocarbons is required in less than 20 minutes. If water is not present and high resolution is required for C4 and C5 olefins, the HP-PLOT Q column could be substituted with an Alumina PLOT column. For this application, C1–C5 were selected to be analyzed on the HP-PLOT Q column and C6 and above was selected to be analyzed on the DB-1 column. The Deans Switch heart cut time was set by injection of a C1 to C6 straight chain gas phase paraffin standard with the Deans Switch off, to establish retention time on the DB-1 column.

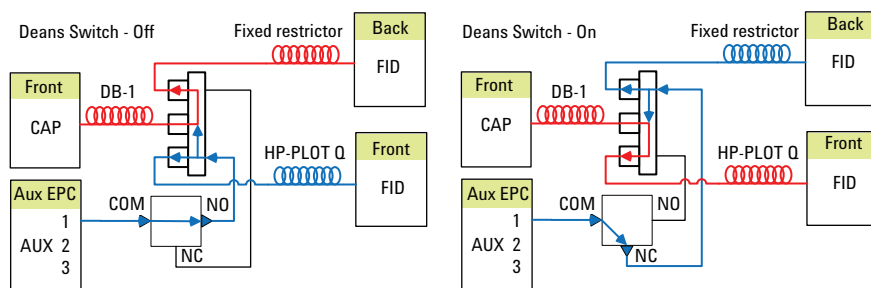


Figure 1. Deans Switch.

Results and Discussion

Figure 1 shows the results from the analysis of the paraffin standard with the Deans Switch in the Off position, bypassing the HP-PLOT Q column. This is necessary to set the cut times to the PLOT column. In this case, the cut times were set to 3 minutes and 6.5 minutes, as depicted in Figure 2.

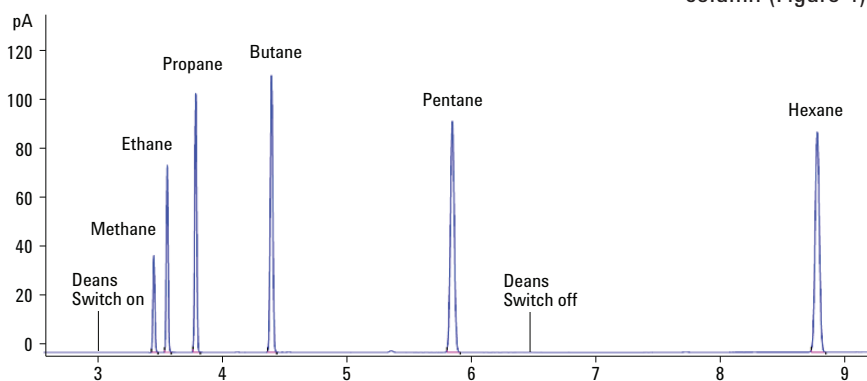


Figure 2. DB-1 only.

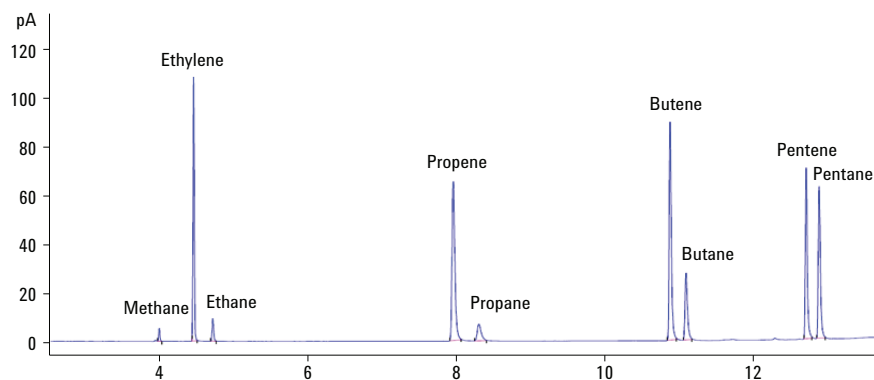


Figure 3. HP-PLOT Q column C1 through C5 analysis.

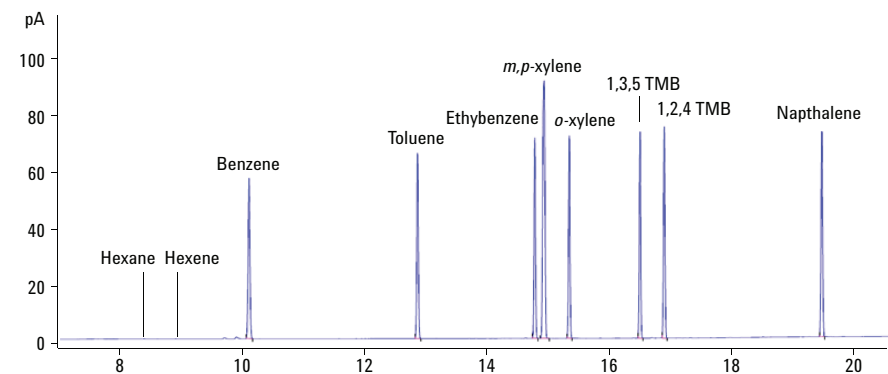


Figure 4. DB-1 column extended analysis.

Figure 3 and Figure 4 show the results from the analysis when the heart cut times are incorporated into the method. As the light fraction C1–C5 components elute from the DB-1 column, they are heart cut to the HP-PLOT Q column. These compounds are then further separated on the HP-PLOT Q, and detected by an FID as shown in Figure 3. After the heart cut is made, the remaining components, C6 and up, elute through the restrictor to the second FID, bypassing the HP-PLOT Q column (Figure 4).

Conclusion

Using a Capillary Flow Technology Deans Switch configured with a Dual FID provides an effective way to analyze a very broad spectrum of compounds with different retention characteristics. Both polar and nonpolar analysis can benefit using this technique. This approach provides a fast, flexible analysis with easy setup, without the need for cryogenic cooling for both gas and liquid phase samples.

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