# Flame Ionisation Detector (FID)
# for Agilent 1260 Infinity-SFC

## SFC-FID-System


SIM
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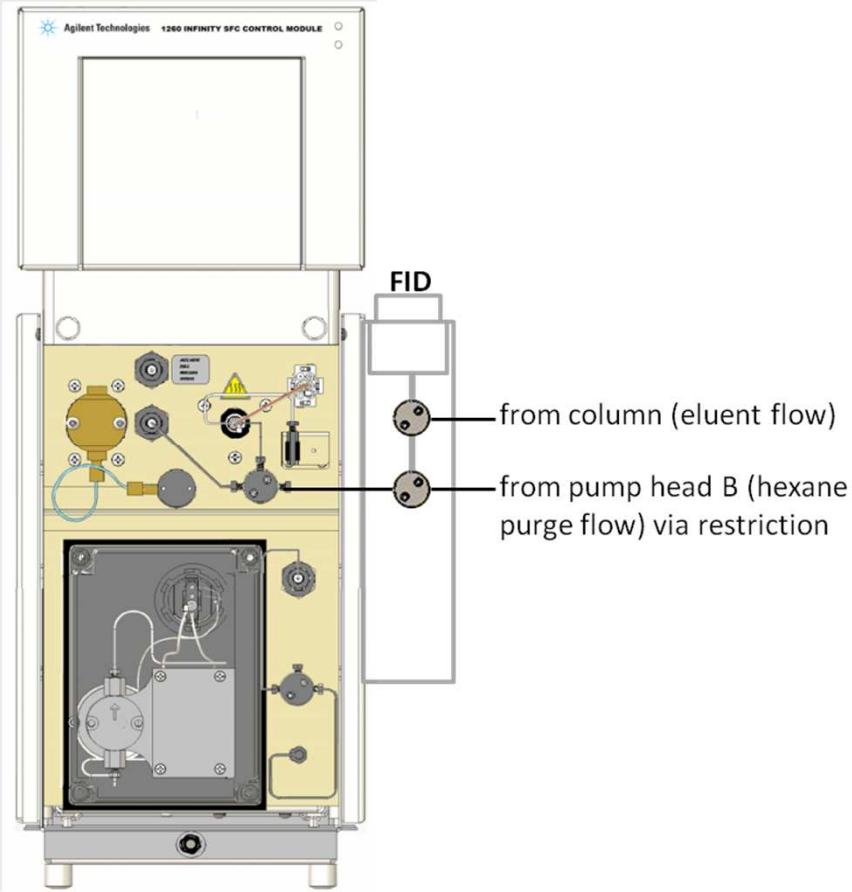
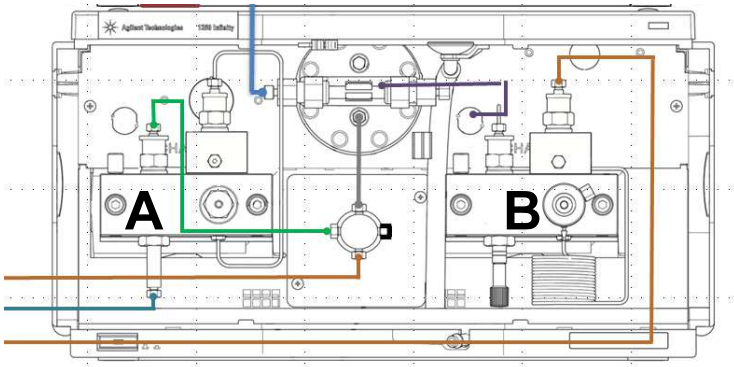
# Assembling of FID and Agilent SFC (with restrictor and hexane purging)

**SFC Module**

**FID**




**Binary Pump**




<u>binary pump</u>

channel A: $CO_2$

channel B: purging (optional, here: hexane)

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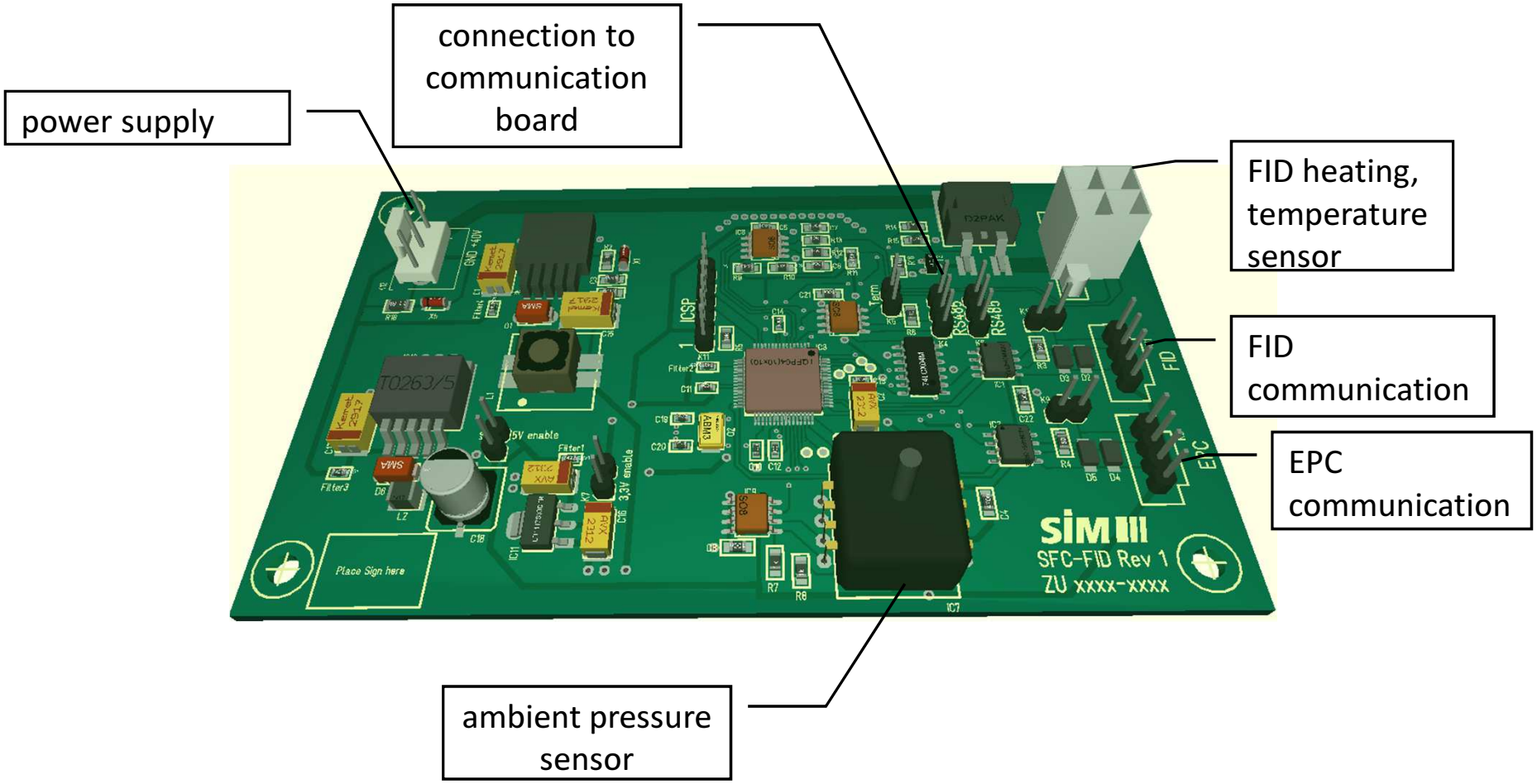
# Development of Electronic Components (I)

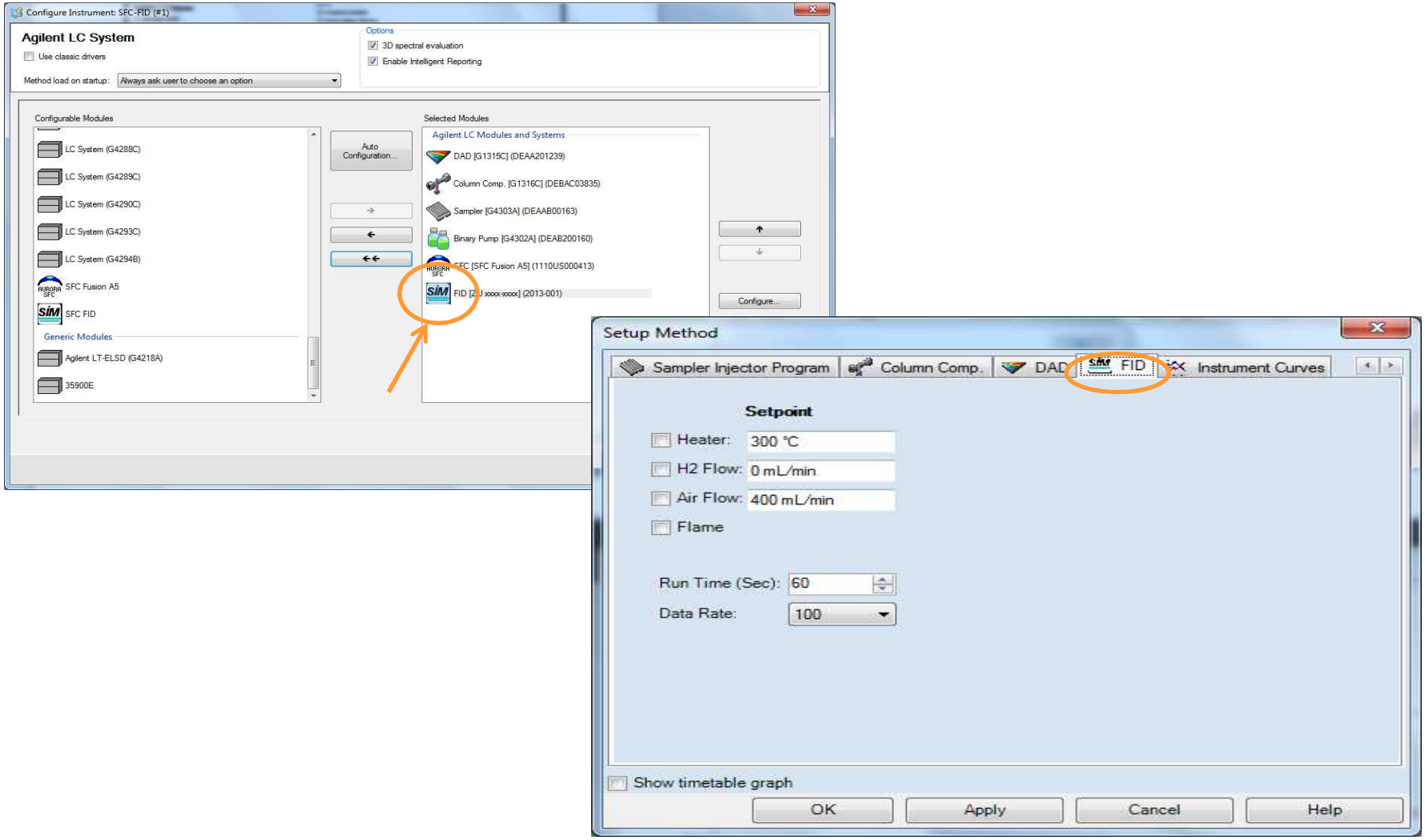
## EPC/FID-Board



# Development of Electronic Components (II)

## Communication-Board

# OpenLAB CDS ChemStation : FID Module Configuration / FID Parameters

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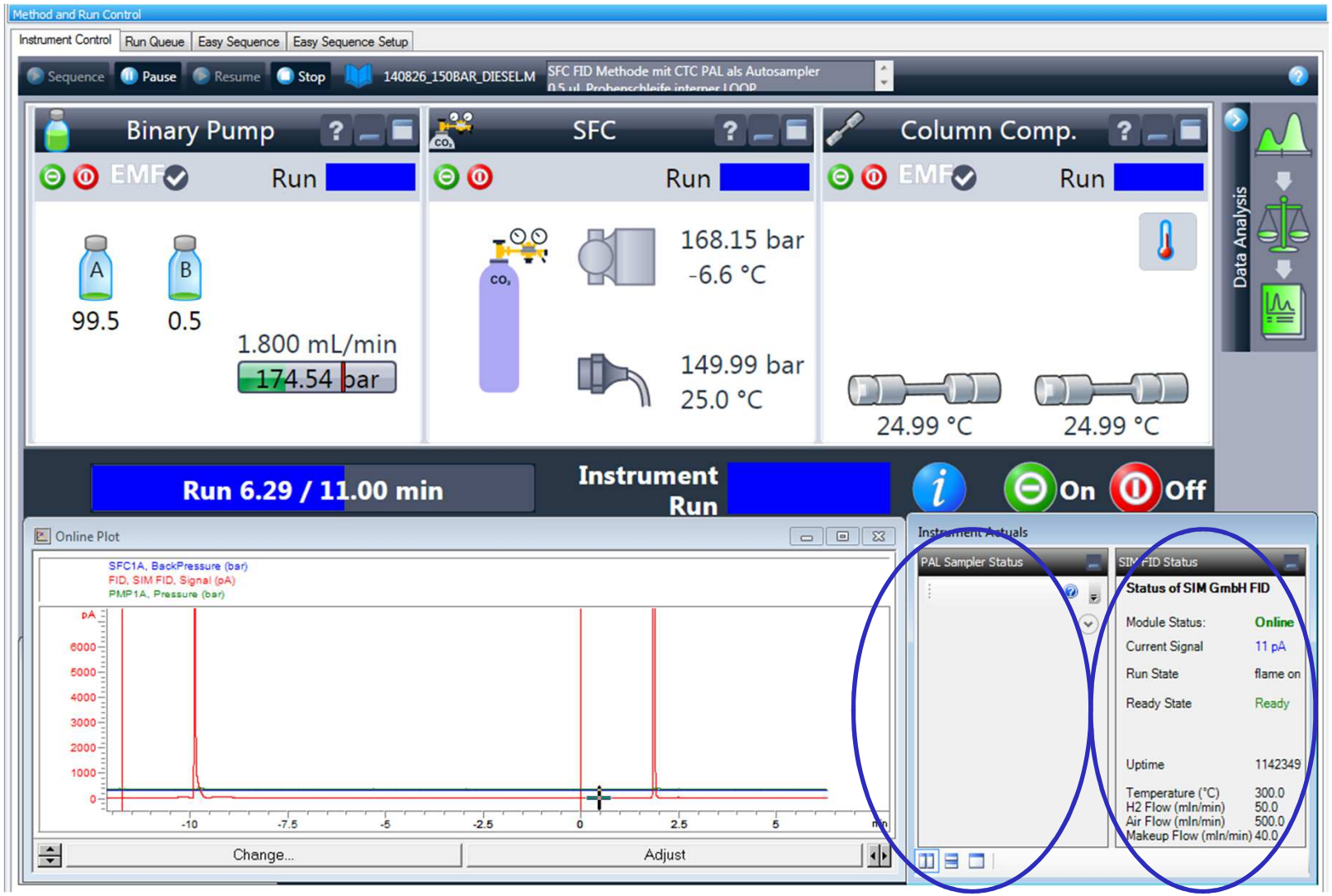
# Online Screen: Status of SFC-FID and PAL




PAL FID

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# ASTM D-5186: Aromatic content of diesel fuels and aviation turbine fuels by SFC-FID

Composition of Performance Mixture:

75 % n-hexadecane ($C_{16}$)

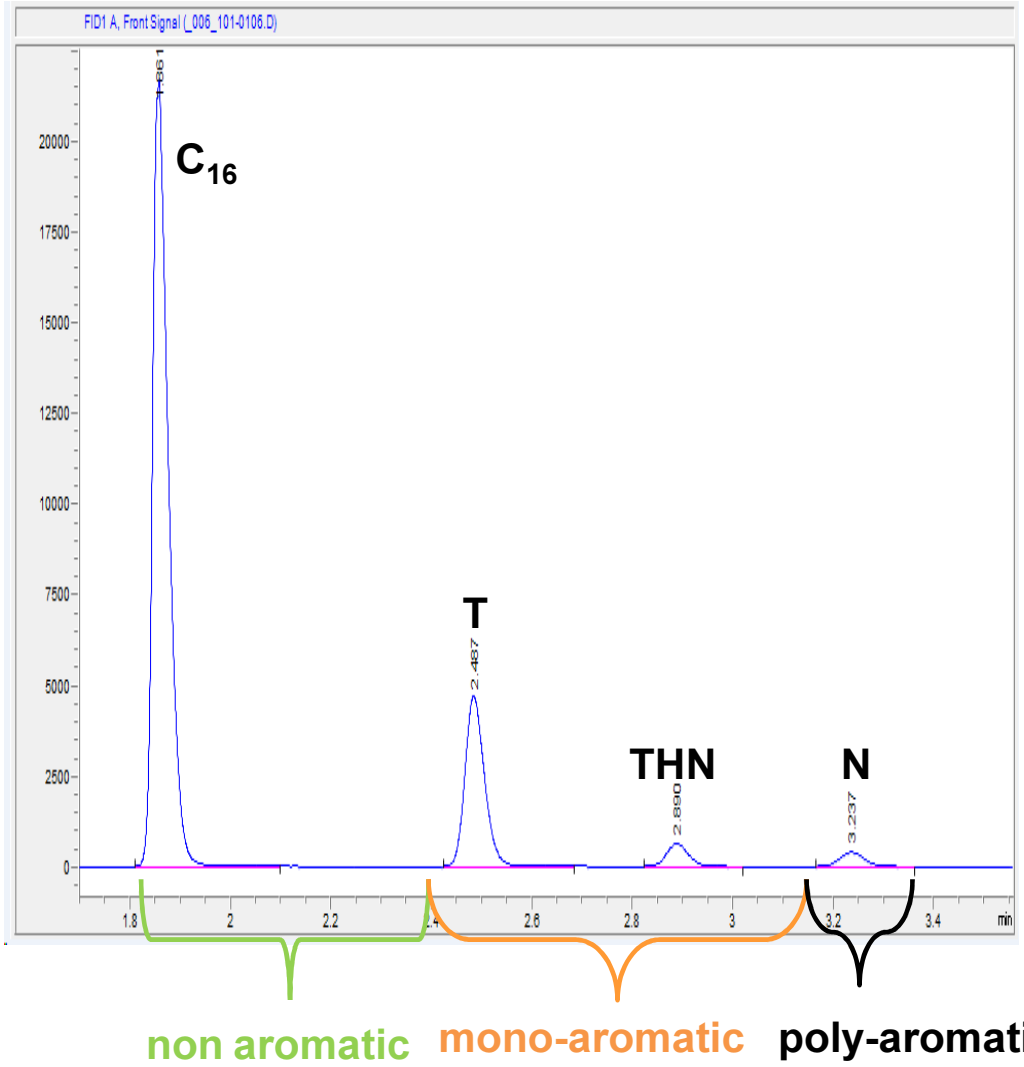
20% toluene (**T**)

3 % tetrahydronaphthaline (**THN**)

2 % naphthaline (**N**)

Analytical conditions:

| | |
|---|---|
| system: | 1260 LC, SFC-FID |
| column: | YMC-PACK-SIL_06, 250 × 4.5 mm,6 nm S-, 5 µm or Agilent ZORBAX RX-SIL, 4.6 × 250 mm, 5 µm |
| column temp.: | 25.0 °C |
| mob. phase: | 100 % $CO_2$ |
| flow : | 1.8 ml |
| SFC (BPR): | 25 °C (150 bar) |
| injection: | 0.5µl |
| FID : | 300°C<br>50 ml $H_2$, 500 ml air,<br>50 ml $N_2$ (make-up) |

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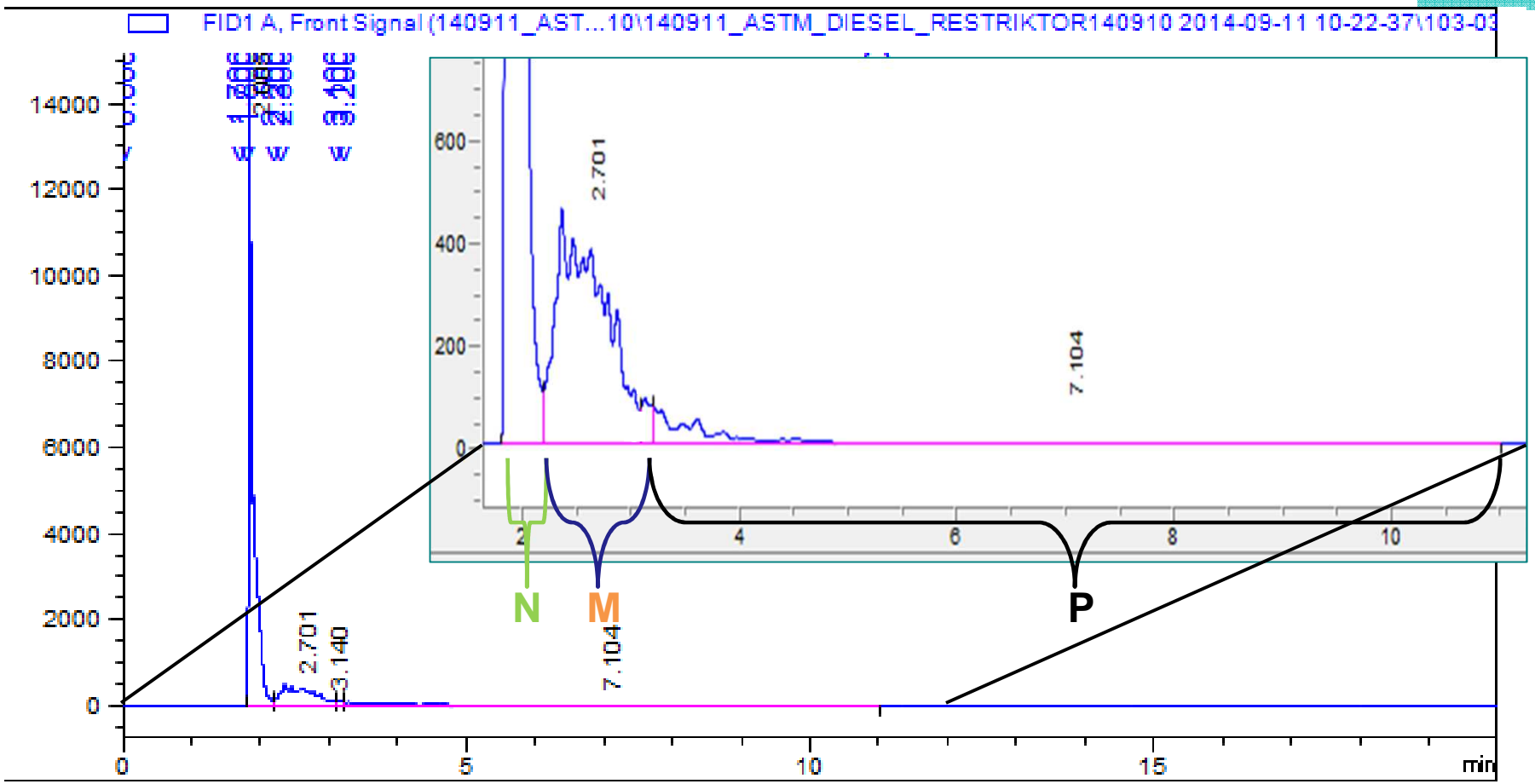
# Diesel-Sample – Integration and Calculation




**content of aromatic components (A): M (%) + P (%) = A (%)**

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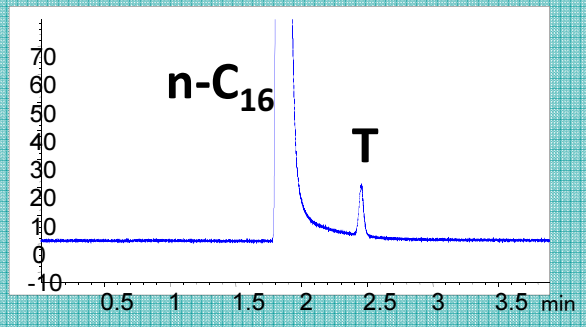
# ASTM D-5186: Analytical Requirements

| | Requirement | SIM SFC-FID |
|---|---|---|
| Resolution | $R_{NM}$(n-$C_{16}$ /T) ≥ **4**<br>$R_{MD}$(THN/N) ≥ **2** | $R_{NM}$(n-$C_{16}$ /T) = **10.1**<br>$R_{MD}$(THN/N) = **4.1** |
| Retention Time Reproducibility | RSD (n-$C_{16}$) **< 0.5**<br>RSD (T) **< 0.5** | RSD (n-$C_{16}$): **0.24 %**<br>RSD (T): **0.28 %** |
| Detector Sensitivity | detection of<br>0.1 mass% T in n-$C_{16}$ |  |



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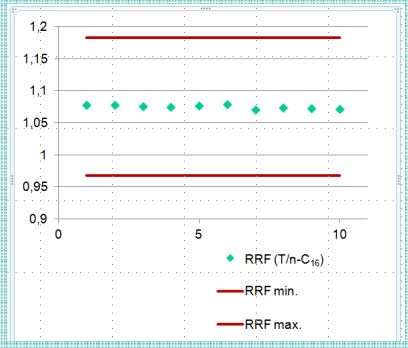
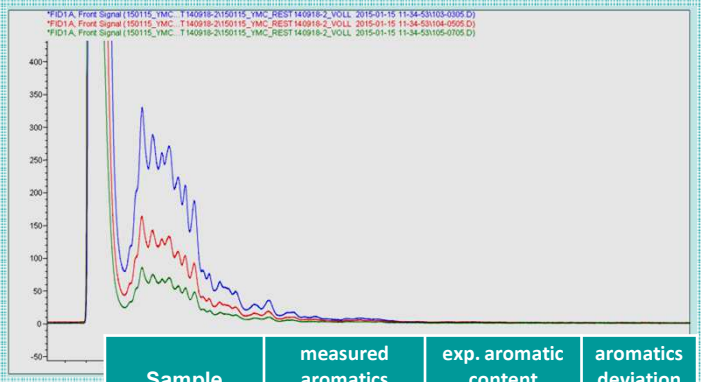
# ASTM D-5186: Analytical Requirements

| | Requirement | SIM SFC-FID |
|---|---|---|
| Detector Accuracy | relative response factors (RRF): RRF ± 10 % of theoretical value | example: toluene  |
| Detector Linearity Check | aromatic content of diesel fuel and two dilutions |  |



| Sample | measured aromatics (mass%) | exp. aromatic content (mass%) | aromatics deviation (mass%) |
|---|---|---|---|
| Fuel | 21.8 | | |
| F-Dilution* (1:1) | 11.1 | 10.8 | 0.3 |
| F-Dilution* (1:2) | 5.9 | 5.7 | 0.2 |

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# ASTM D-6550 : Olefin Content of Gasoline by SFC


- <u>two columns / two TCCs:</u>

  1. silica column (25 °C)
  2. silver loaded column (80 °C)

  → two thermost. column compartments necessary

- <u>valve-switching solution</u>
  forward-flow to trap the olefins on the $Ag^+$ column
  back-flush for quantification

- <u>detector:</u>

  SIM FID for SFC

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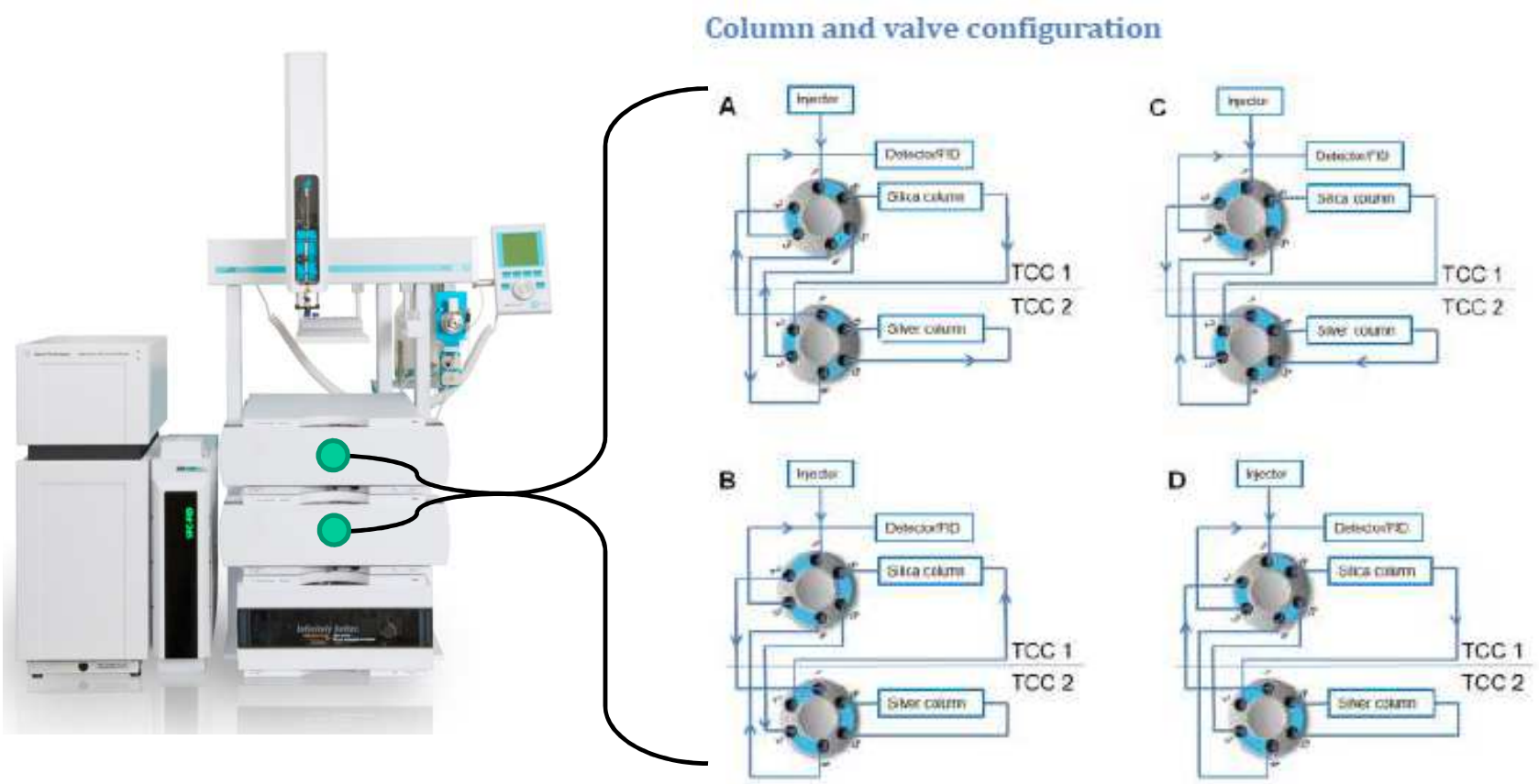
# ASTM D-6550 : Valve switching solution




- <u>two 6-port valves with 4 valve positions (A-D)</u>
  for forward-flush and backflush modes to separate and quantify the olefins
- <u>position D</u> allows Test Method **D5186** to be performed without changing the system

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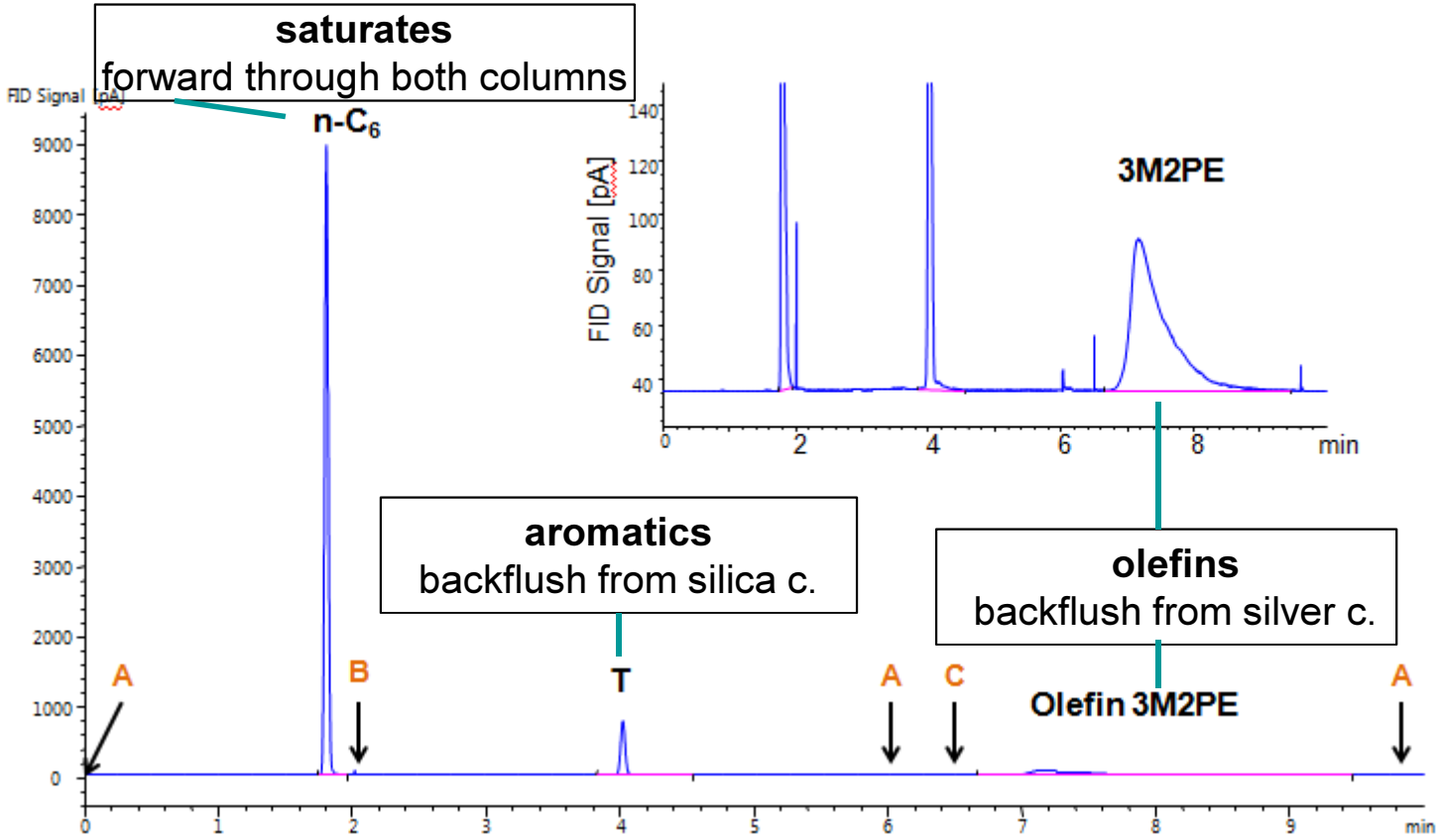
# ASTM D-6550 : Performance Test Mixture (PTM)




Performance Test Mixture (PTM):

80 % n-hexane (**n-$C_{16}$**) → **SATURATES**

10 % toluene (**T**) → **AROMATICS**

10 % 3-methyl-2-pentene, cis/trans (**3M2PE**) → **OLEFINS**

➔ final method after system optimization (switching times!)

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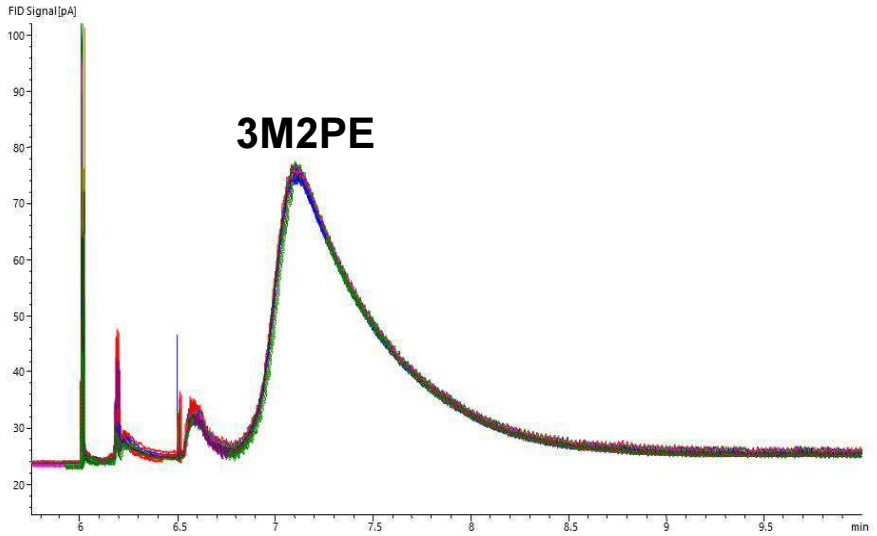
# ASTM D-6550 : Precision of Olefin Measurement

10 injections of PTM:




**requirement:**
RT-Repeatability < 0.5 %

**measured:**
0.04 – 0.13 % (RSD)

➔RT-repeatability is important for quantification:
column switching is performed on time basis

| | Hexane | | Toluene | | Olefin 3M2PE | |
|---|---|---|---|---|---|---|
| | RT | Area | RT | Area | RT | Area |
| Average | 1.8 | 16161 | 4.02 | 2198.6 | 7.16 | 2095.5 |
| SD | 0.002 | 130.6 | 0.002 | 17.7 | 0.003 | 26.0 |
| RSD [%] | **0.13** | **0.81** | **0.04** | **0.80** | **0.04** | **1.26** |

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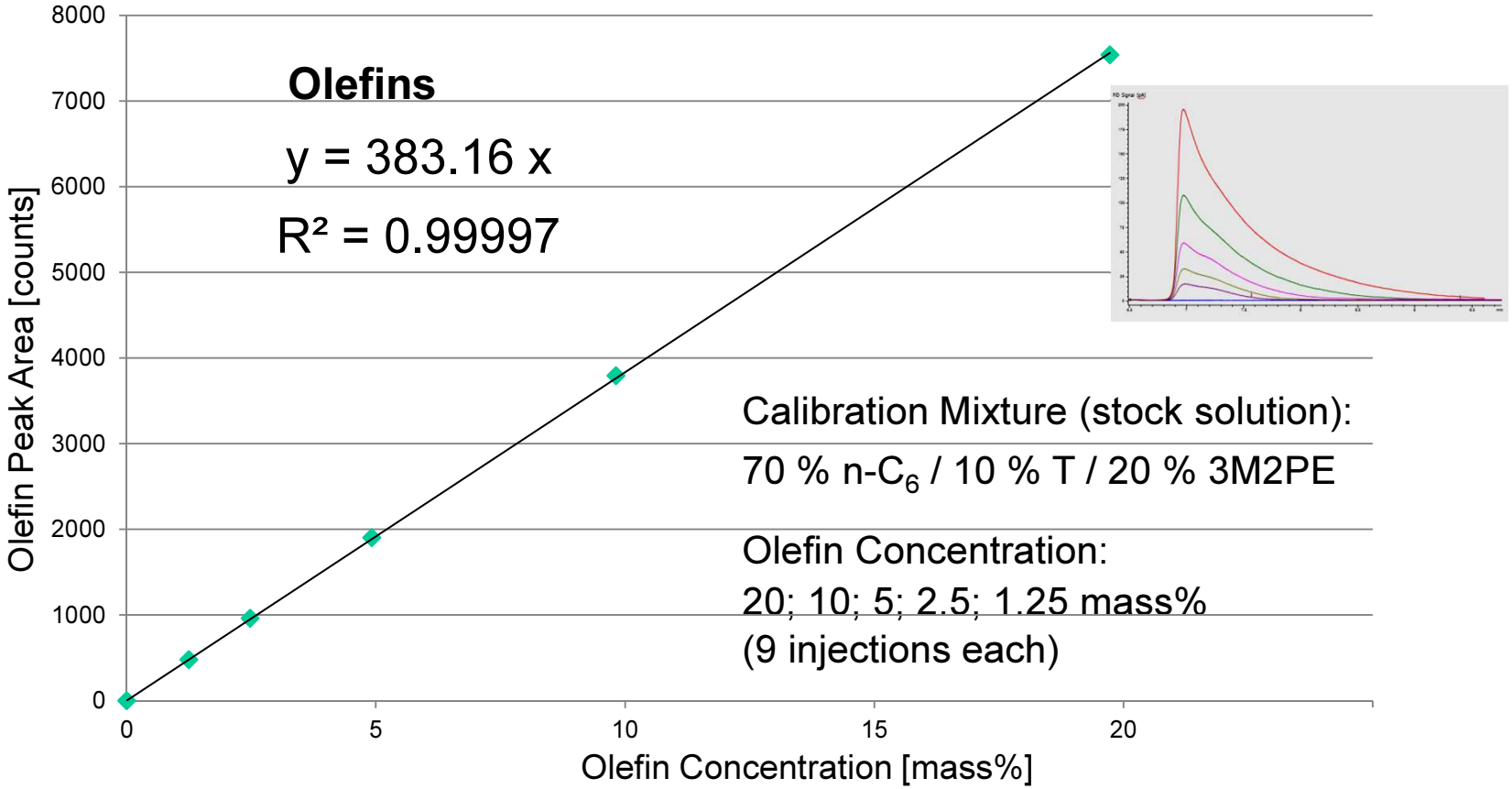
# ASTM D-6550 : Olefin Calibration




**requirement:** intercept statistically not different from zero ✓

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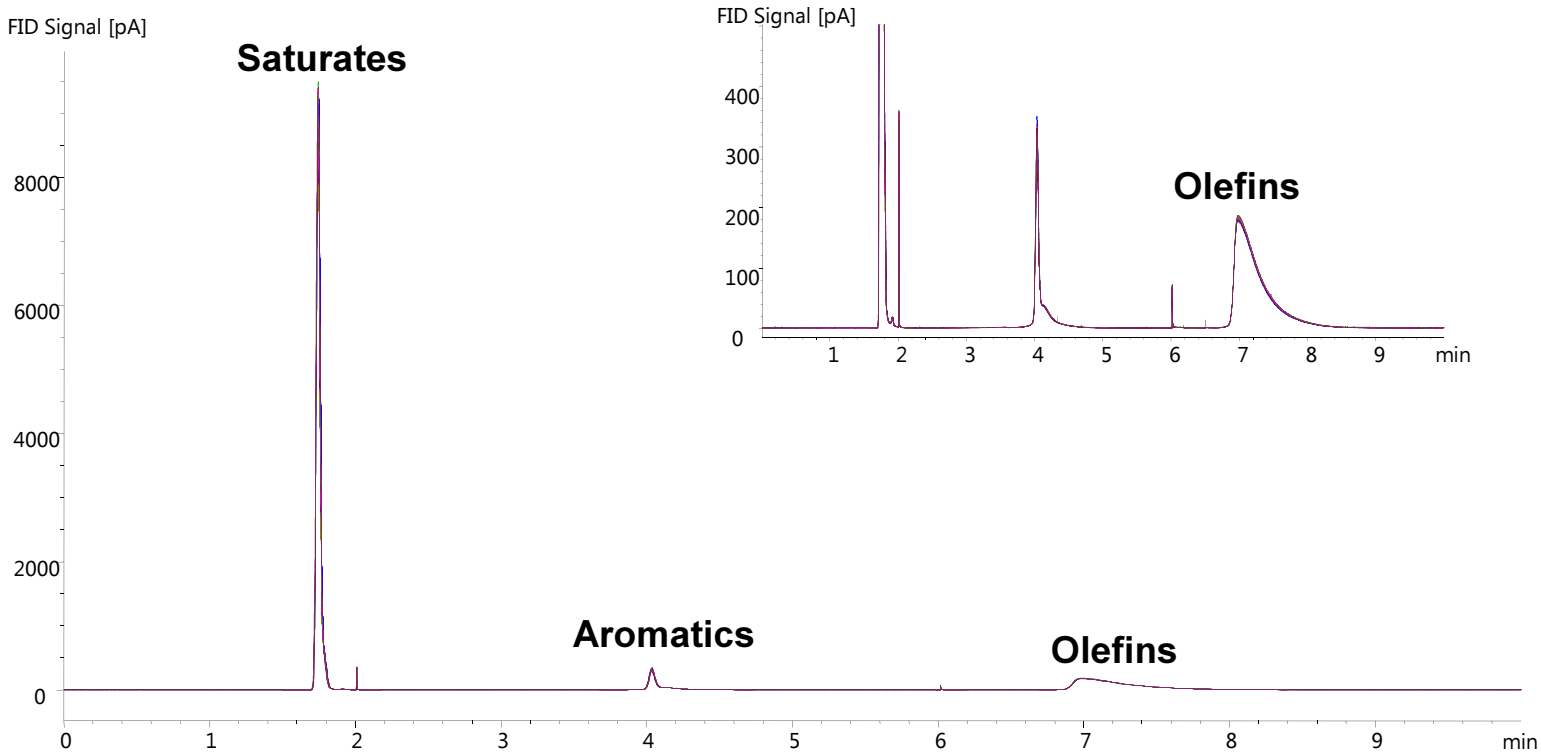
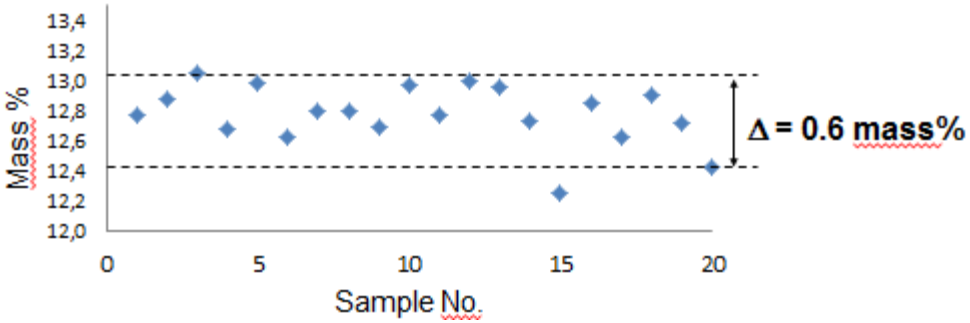
# ASTM D-6550 : Gasoline Sample










**repeatability limit:** 0.7 mass%
(shall not be exceeded in more than one case in twenty)

**gasoline sample:**

12.8 mass% olefins (mean value)

19 results, difference ≤ 0.6 mass% ✓

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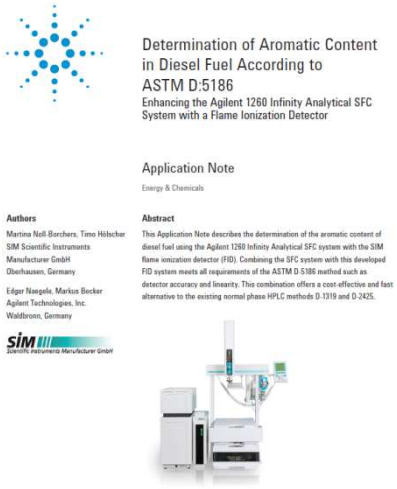
# SFC-FID Analyzers


Determination of Aromatic Content in Diesel Fuel According to ASTM D:5186

Enhancing the Agilent 1260 Infinity Analytical SFC System with a Flame Ionization Detector

Application Note

Energy & Chemicals

Authors

Martina Noll-Borchers, Timo Hölscher
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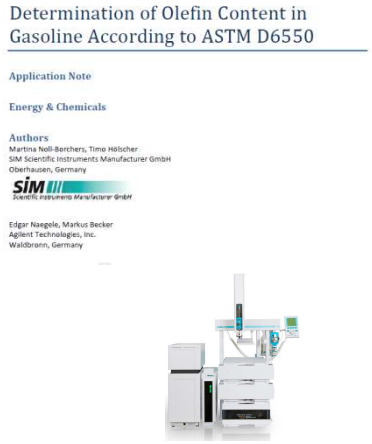
Waldbronn, Germany

Abstract

This Application Note describes the determination of the aromatic content of diesel fuel using the Agilent 1260 Infinity Analytical SFC system with the SIM flame ionization detector (FID). Combining the SFC system with this developed FID system meets all requirements of the ASTM D-5186 method such as detector accuracy and linearity. This combination offers a cost-effective and fast alternative to the existing normal phase HPLC methods D-1319 and D-2425.

**ASTM D5186: 5991-5682EN**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**ASTM D6550: Draft**

- **consist of** - 1260 SFC Control Module and Binary Pump
  - Thermostatted Column Compartment (1x or 2x together with column switching)
  - 1290 Infinity LC-Injector HTC (width 50 cm) incl. DLW Option
  - SIM FID for SFC with OpenLAB Driver (ChemStation)

➔ **two applications: ASTM D-5186 + ASTM D-6550** also with the same system

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**Thank you for your**

**attention!**

SIM
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