FAST TOTAL OLEFIN ACCORDING ASTM D6296 BY GAS CHROMATOGRAPHY

- **Full Determination of All Olefins within 20 Minutes**
- **Applicable for all Finished Motor Gasoline and Naphtha Streams**
- **Application Range: 0.2-35.0% Olefinic Streams**
- **Excellent Repeatability**

Keywords:
Gas Chromatography; Gasoline; Multidimensional Gas Chromatography; Olefins; Spark-ignition Engine Fuels

INTRODUCTION

This test method provides for the quantitative determination of total olefins in the C4 to C10 range in spark-ignition enginefuels or related hydrocarbon streams, such as naphthas and cracked naphthas.

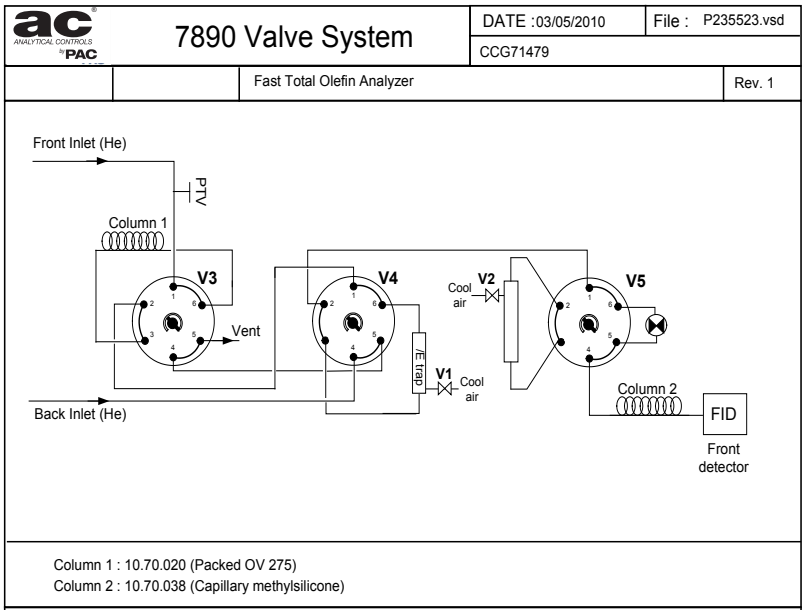
PRINCIPLE

A sample is introduced into the AC PTV inlet, excluding the use of split injections or carrier gas purging of the inlet septum as described in ASTM D6296. The valves are actuated at predetermined times to direct portions of the sample to appropriate columns and traps. The sample first passes through a polar column that retains C12+ hydrocarbons, all aromatics, C11+ olefins, and some alcohols, all of which are subsequently backflushed to vent. The fraction eluting from the polar column, which contains C11and lower boiling saturated hydrocarbons as well as decene and lower boiling olefins, enters an ether/alcohol trap where the ethers and alcohols are selectively retained and also subsequently backflushed.

The fraction eluting from the ether/alcohol trap, which consists of C11 and lower boiling saturated hydrocarbons and the olefins, enters an olefin trap. The olefins are selectively retained while the saturated hydrocarbons elute, pass through a nonpolar column, and are detected by a flame ionization detector (FID). When the saturated hydrocarbons have completely eluted to the FID, the nonpolar column oven is cooled and the olefins, which have been retained on the olefin trap, are desorbed by heating. The desorbed olefins enter and elute from the nonpolar column, which is temperature programmed to separate the olefins by boiling point, and are detected by the FID.

INSTRUMENT PARAMETERS

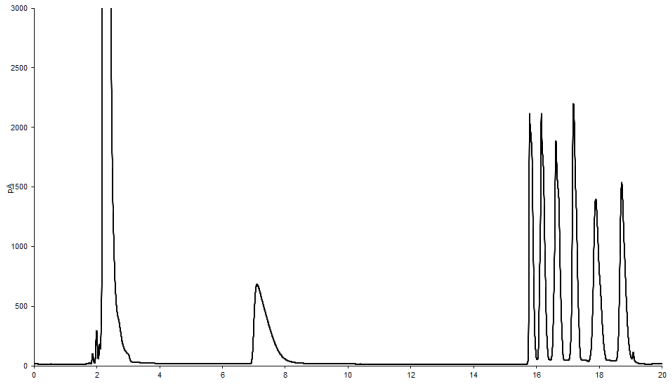
- Oven temperature
  - 160 °C for 10 min
  - then 50 °C/min to 50 °C for 1.3 min
  - then 20 °C/min to 160 °C for 1 min
- Run Time 20 min
- Olefin Trap
  - 140 °C for 13 min
  - then 80 °C/min to 280 °C for 3 min
  - then 100 °C/min to 140 °C for 0 min
- Ether/alcohol trap
  - 150 °C for 5.5 min
  - then 50 °C/min to 280 °C for 1 min
  - then 100 °C/min to 150 °C for 0 min
- AC PTV Inlet 200°C
  - Carrier Gas Helium
  - Carrier Gas 20 ml/min Constant Flow Mode
- Detector (FID) 190°C
- Injection Mode Automated Fast Injection
- Injection Volume 2.0 µl



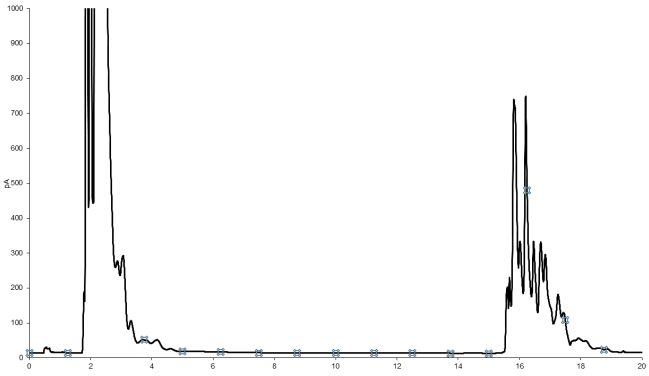
ANALYTICAL CONTROLS by PAC

INSTRUMENT CALIBRATION

The instrument calibration consists of two steps: performing a blank run to compensate the signal and a calibration run that determines the response factor of the system and the retention times of the olefins. External standardization is used as the calibration method. This has been configured in the analysis method. Sample 50.17.041 is used for calibration of the method. The calibration calculations calculate response factors for volume and weight percent calculations. The components n-hexene through n-nonene are used to calculate the response factor. The density for area slices at specific retention times (between carbon numbers) is calculated by using a linear interpolation of adjacent densities.



Figuur 2. FTO Calibration Standard



Figuur 3. FTO Analysis of Gasoline

REPORTING

A report template is included with the application and generates a report conforming to the standard test method in mass% and volume% total Olefin.
The density is used for the conversion from injection volume to mass. The calculation of total olefins is performed by the software when the calibration standard density, sample density, and (possible) dilution are given.
The system can be configured so it goes in standby-mode after a sequence of analyses, lowering gas consumption and keeping the system conditioned for the next runs.

PRECISION STATEMENT

The following precision data has been obtained during a Round Robin study.

Concentration Level (lv%)	Repeatability	Reproducibility
1	0.1	0.3
3	0.2	0.6
5	0.3	0.8
10	0.4	1.4
15	0.5	1.8
20	0.6	2.2
25	0.8	2.6
30	0.9	3.0
35	1.0	3.4
40	1.1	3.7
45	1.2	4.0
50	1.3	4.4

CONCLUSION

The combination of the configured GC system (see configuration above), selected capillary GC columns, proprietary injection port and liner, appropriate chemical standards as well as worldwide application installation and support by certified AC by PAC Service, provide a complete solution for the Fast measurement of Total Olefin concentration.

The FTO system analyzes streams with concentrations of 0.2%-35% olefins. This GC system determines C4 - C10 olefins in all finished motor gasolines, straight naphthas and FCC naphthas. It reports in weight % and liquid volume % and a full analysis takes just 20 minutes. The FTO analysis exceeds the repeatability capabilities of ASTM D 1319 (the Fluorescent Indicator Absorption-FIA-method) and incorporates ASTM D 6296 - Total Olefins in Spark-Ignition Engine Fuels by Multi-Dimensional GC.

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AC Analytical Controls® has been the recognized leader in chromatography analyzers for gas, naphtha and gasoline streams in crude oil refining since 1981. AC also provides technology for residuals analysis for the hydrocarbon processing industry. Applications cover the entire spectrum of petroleum, petrochemical and refinery, gas and natural gas analysis; ACs Turn-Key Application solutions include the AC Reformulyzer, SimDis, HiSpeed RGA and Customized instruments.