

Separation Systems, Inc.

100 Nightingale Lane
Gulf Breeze, FL 32561



Scope of the Method

This is the gas chromatographic method used to determine individual and total paraffins in the feed, raffinate and extract streams generated by the UOP Molex™ process ^(Note 1) by 'subtractive' chromatography. The lower limit of quantitation (LOQ) for an individual normal paraffin is 0.1 mass%.

The feed, raffinate or extract sample is analyzed synchronously using a single gas chromatograph (GC) with two separate channels. Each channel is identically configured with a split/splitless capillary inlet, capillary column and flame ionization detector. However, the inlet in one channel contains a 'standard' glass liner (no packing material) while the inlet on the other channel contains a glass liner packed with a special molecular material (UniSieve™) to remove any normal paraffins contained in the sample which prevents them from being introduced to/onto the chromatographic column. (Figure 3)

The total sample chromatogram produced by the channel without the specially packed inlet liner is superimposed (overlaid) on the (de-normalized) chromatogram produced by the channel containing the packed inlet liner. This is accomplished automatically through the use of Synchronizer™ software developed by Separation Systems, Inc. (Figures 4 and 5)

The 'Synchronizer' software automatically aligns the two chromatograms (individual peaks are temporally aligned) so the mass% of each normal paraffin can be quantified using an internal (normalization) calibration technique.

The method on which this system is based is an updated version of the original one (UOP-411-92) which called for the use of sequential thermal conductivity detectors (TCD) and a valve system to alternate between two different injection modes (pre and post-sieve). The sieve material described in the updated method is manufactured using a new process called UniSieve™.

Note 1 - UOP is a wholly-owned subsidiary of Honeywell Corporation.

Dual Channel GC Analyzer Configuration

The analytical system is based on the Agilent 7890B gas chromatograph (Figure 1).



Figure 1 – Fully Automated Dual Channel 7890B GC Configured for UOP 411-13

The 7890B is custom configured and tested for UOP 411-13 (Figure 2) by Separation Systems and includes the following:

- Programmable column oven with 20 individual ramps/21 plateaus; ambient +4 °C to 450 °C
- Full digital pneumatic control; 0.001 psi control from 0 to 150 psi
- (2) split/splitless capillary inlets with digital pneumatic control; 400 °C maximum; one equipped with an empty glass liner; the other with a glass liner packed with molecular sieve material.
- (2) flame ionization detectors (FID) with digital pneumatic control; 450 °C maximum
- (2) automatic injector ‘towers’; each with a 18 sample capacity
- Agilent OpenLAB GC Chemstation system control, data acquisition and reporting software
- Synchronizer™ software specially produced by Separation Systems (Part No. SS-185-01) per UOP 411-13^{Note2} to facilitate the temporal alignment of the chromatograms independently produced by the 2 GC channels and then identifies and quantifies the individual components
- (2) capillary columns; both identically coated with cross-linked stationary phase

Note 2 – UOP 411-13 is available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, USA. The UOP methods may be obtained through the ASTM website, www.astm.org or by contacting Customer Service at service@astm.org or 610-832-9585

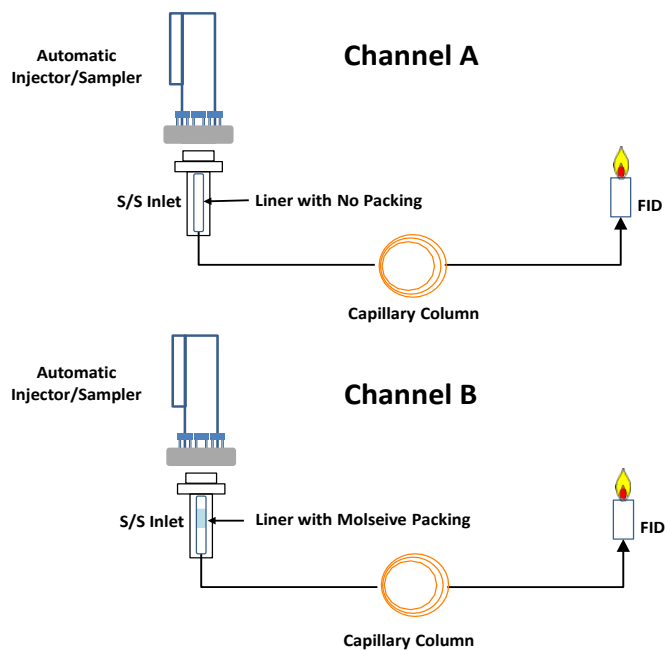


Figure 2 – Dual Channel Configuration of the GC for UOP 411-13



Figure 3 - Special Glass Inlet Liner with Molsieve Packing (Separation Systems, Inc. Part No. SS-053-500)

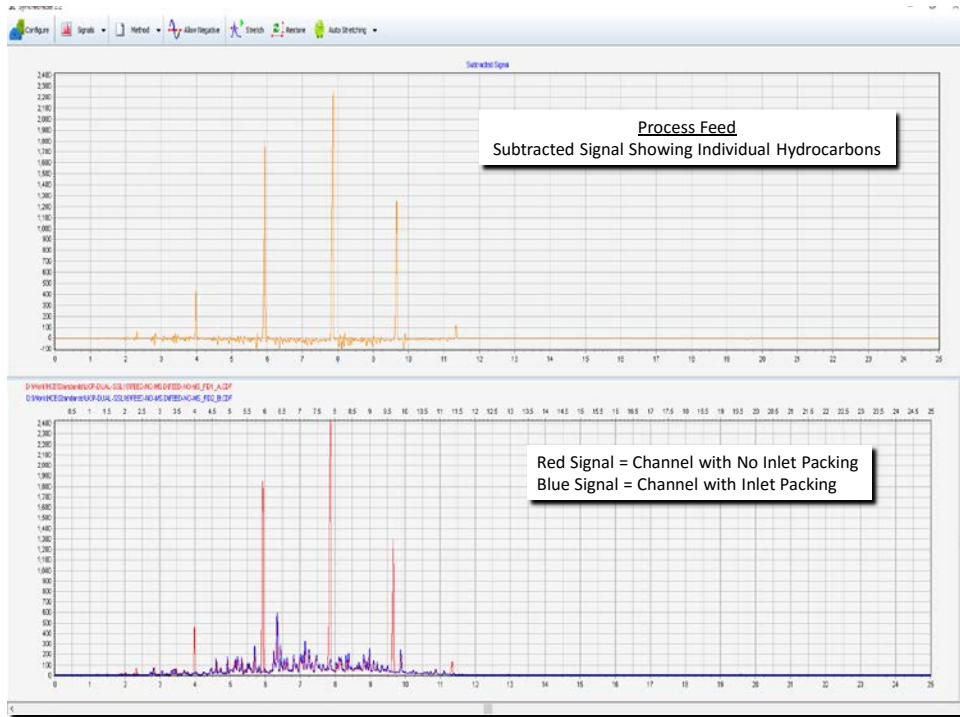


Figure 4 - Results from Process Feed Analysis

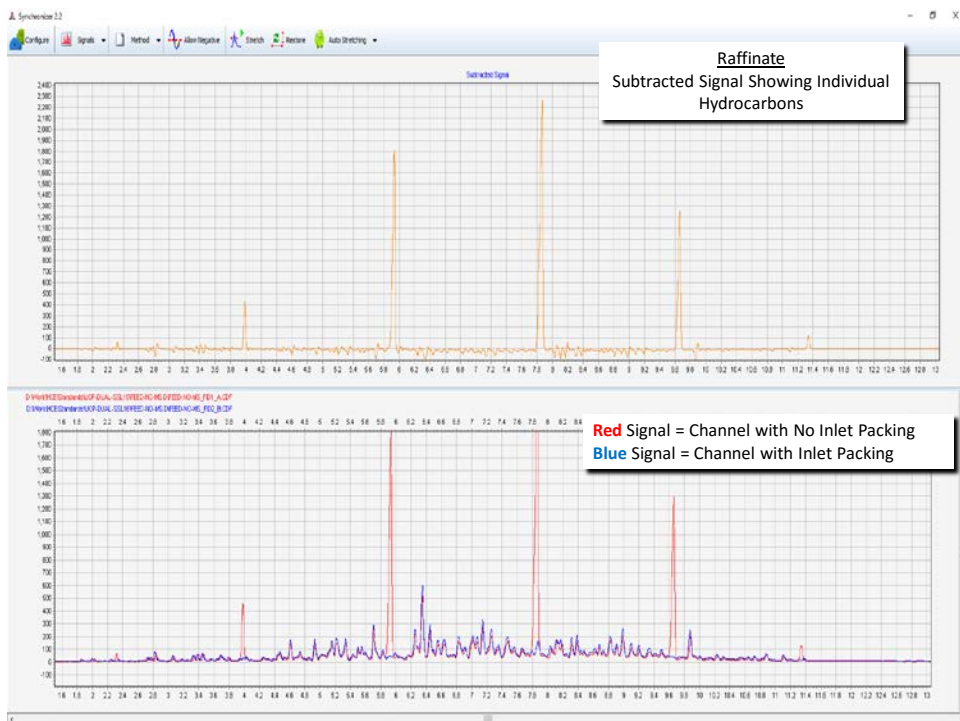


Figure 5 - Results from Raffinate Sample

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