SYSTEM DESIGN OF THE ULTRA HYDROCARBON SYSTEM *by Lotus Consulting*

Full speciation of hydrocarbons in exhaust and ambient air samples requires, arguably, the most complex gas chromatographic system ever implemented. Samples must be concentrated into a small volume to enhance detectivity, a very large number of components must be "fully" separated, and individual peaks must be identified and quantified. The system involves either one dual or two separate cold traps, six or seven automated valves, 16-position automated sampler, three capillary columns, two or four detectors, and one workstation.

To provide concentration levels within the capabilities of mandated detectors and still maintain sharp chromatographic peaks, hydrocarbon components must be concentrated from a typical volume of 30 ml down to 100 μl prior to injection onto the column. Detection limits with a flame ionization detector become less than 10 ppbC for a 30 ml sample.

For full unattended operations, automated valving is employed to quantitatively transfer a sample aliquot from one of 16 connected sample containers to the concentrator trap and, after trapping, transfer the sample to the column.

The ultimate separation of hydrocarbons is achieved chromatographically with two columns plumbed in parallel operations – one for light-end components from ethene to hexane and the other for components to tridecane. Water and heavy hydrocarbons are kept from the light-end column with a stripper column prior to trapping.

All of these operations utilize nearly all of the powerful and comprehensive capabilities of the Scion 456 Gas Chromatograph and the Scion MSWS Workstation employed in the Ultra Hydrocarbon System by Lotus Consulting. Such a complex system requires assurances that the data is valid, that the system is fully functional and easy to use, and that the ultimate performance is achievable. The following are major features of the Lotus Consulting Ultra Hydrocarbon System and their useful benefits.

FEATURES	BENEFITS

QUALITY ASSURANCES

• Automated sampler position tied to master sample list	+ Avoids mislabeling of samples
• Automated sampler position documented in final report	+ Confirms proper sample labeling
• Visual indication of sample loading	+ Assists diagnosis if no peaks detected
• Sample carryover <0.1%	+ Impedes high concentration samples from inducing peaks in next sample
• Automatic internal standard/surrogate addition	+ Verifies proper trapping and chromatography; allows correction for many sampling errors
• Indicator for cold trap plugging by monitoring purge gas pressure	+ Assures proper sample loading
• Platinum temperature probes in all zones, including cold trap	+ Self-calibrating and very accurate; maintains inter-instrument consistency
• Column pressure/flow/velocity documentation in final report with Electronic Flow Controllers (EFC)	+ Assists diagnosis of leaks, depleted carrier gas supply, and improper carrier gas flows
• Detector gas flows documented in final report with Detector Electronic Flow Controllers (DEFC)	+ Verification of proper flame conditons
• User-settable stabilization time prior to start of each run	+ Ensures consistent instrument starting conditions; achieves better retention time reproducibilities
• “Ready/Not Ready” status interlocks for temperature zones, column flow, velocity, pressures	+ Prohibits injections into a faulty instrument
• Optional PID in series with FID	+ Verifies identification of peaks, especially olefins and aromatics
• FID “flame-out” and reignition	+ Gives diagnosis of a faulty instrument condition

•	FID flame background displayed	+ Permits diagnosis of contaminated system, including column, supply gases, and sample carryover; verifies flame ignition
•	Detector noise monitored at start of each run and documented in final report	+ Maintains proper peak detection automatically; diagnoses of faulty detector
•	Full diagnostic self-checking of chromatograph at power-up	+ Ensures fully functional chromatograph
•	410 different error messages on GC (*i.e.,* Fault 110 – Temp Sensor Probe Fault; Fault 37 – Power Line Ground Fault; Fault 257 – Detector Output Signal Inaccurate)	+ Detects errors and offers diagnoses many facets of chromatograph; serious errors will halt operations
•	121 different error messages on Workstation (*i.e.,* "Suspended sequence, disk is full"; "Module connection has been broken")	+ Detects errors and offers diagnoses for many facets of Workstations; serious errors will halt operations
•	Complete error documentation in final report	+ Assists diagnosis of difficulties
•	Run Log documentation in final report, including run settings for all chromatographic, trap, and detector parameters	+ Ensures proper run conditions were used; facilitates method development with documented run parameters
•	Time/date stamped in final report for injection, recalculation, and printing	+ Provides full documentation of report timings
•	"Verification" sample type	+ Checks proper calibration and halts sequence if out of tolerance
•	Single stored data file contains raw data for two channels, final reports, run method, run log, and error messages	+ Enables easy reconstruction and redocumentation of final report; no confusion over which method and run conditions were used

FEATURES	BENEFITS

OPERATOR CONVENIENCES

• All valving fully automated	+ Allows full unattended operations
• Random access to any automated sampler position in sample list	+ Permits recalibration and rerun of control sample(s)/blank without using extra inlet ports
• Manual sample position advancement on instrument and on Workstation	+ Facilitates repositioning of automated sampler
• Colorful visual display of automated sampler positions: RED – samples to be analyzed; GREEN – current sample; BLUE – sample completed	+ Gives easy overview of sampler operations
• Entries to sample list while system is running	+ Enables changes to sample order without stopping operations
• Single keyboard for total system control	+ Reduces operator confusion; integrates system operation
• Complete system status on one screen	+ Easy overview of complete system operations
• Single method for control of chromatograph, cold trap, automated sampler, data collection, and report generation	+ Avoids confusion of tying separate operations together
• Manual override of chromatograph settings while running	+ Maximizes flexibility during method development
• Visual display of column temperature ramp during method building	+ Illustrates programming profile for easy verfication
• User-specified temperature limits for columns, cold trap, valving, and detectors	+ Protects hardware from operator miscues

•	User-settable coolant timeout	+ Conserves coolant when system is idle; requires no operator intervention for turning off coolant at end of sequence
•	Electronic flow controllers on column carrier and purge gases	+ Allows easy reset of flow setting; results in more consistent retention times
•	Both electronic pressure readout (EFC) and pressure gauges on purge and column gas flows	+ Enables easy setting and verification of column flows/settings
•	Electronic flow controls on instrument for flame gases/make-up gas	+ Enables easy setting and verification of all detector flows/settings
•	Very obvious green/red front panel lights for “ready/not ready” status	+ Gives simple overview of system status; alerts operator to potential problems
•	Analyses not allowed to proceed unless all status indicators are positive	+ Ensures samples are run only when system is ready; avoids sample reruns due to faulty run conditions
•	Automatic switching (optional) to standby conditions at end of sequence	+ Eliminates manual intervention at end of sequence
•	Data collection, report generation, and system control software is a Windows-based software package	+ Yields full flexibility in interaction; allows multi-tasking with other programs

FEATURES	BENEFITS

PERFORMANCE

• Low-volume cold trap	+ Minimizes injection volume from trap to column to keep peaks sharp for better detection and separation
• Cold trap temperature range from -196 °C to 450 °C	+ Allows cryogenic trapping without packings for minimum carryover and clean backgrounds
• Cold trap program rates from 0.1 to 250 °C/min	+ Gives complete flexibility in optimizing cold trap program
• Cold trap isolation during heat-up	+ Maintains sharp transfer to column without need to refocus
• Typically 2 liters of cryogen to cool trap, stabilize for 2 minutes, and sample for 10 minutes	+ Minimizes cost-per-analysis for cryogen
• ±2 °C stability of cold trap temperature	+ Permits complete trapping of C_2+ but exclusion of methane, oxygen, and carbon monoxide
• Cold trap-to-column distance minimized	+ Maintains minimum peak broadening
• Fixed loop sampling	+ Yields ultimate reproducibility for final concentrations
• Sample loop always at atmospheric pressure just prior to injection	+ Achieves proper results for both canisters and Tedlar bags without hardware changes
• Specially treated interconnecting tubing and sample loops	+ Gives proper response factors for all hydrocarbons, including aromatics
• Heated electronic flow controllers	+ Maintains constant flow (hence repeatable retention times) independent of changes in room temperature
• Electronic flow controlled carrier gas	+ Ensures better reproducibility of retention times and peak identities

•	Purge gas cryogenically cleansed during sample transfer from loop to trap	+ Eliminates any false peaks from purge gas
•	Dual column separations of C_2 to C_{13} hydrocarbons with single sample loading	+ Gives full separations of C_2s, C_3s, and C_4s and best separations of heavier hydrocarbons
•	Water and heavy hydrocarbons stripped off prior to trapping for light-end analyses	+ Maintains activity of Alumina column for light-end hydrocarbons, especially separation of ethyne from n-butane/isobutane; stripper column not involved in final chromatography
•	Column temperature range from -99 °C to 450 °C	+ Generates flexibility in optimizing chromatography with full temperature range
•	Column temperature program rates from 0.1 to 100 °C/min	+ Creates flexibility in optimizing chromatographic conditions
•	Four-step temperature program ramps with five hold for column oven	+ Allows optimization of chromatogram with slow ramp in beginning and faster ramp toward end with intermdiate holds
•	High performance FID - 50 msec time constant - noise <4 X 10^{-14} amps @ 50 msec - >2 pgC/sec detectivity - 15 mCoul/gC sensitivity - 10^7 linear range	+ Gives lower detection limits and great linear range; limitations become chromatographic
•	High performance PID optional for olefins and aromatics - 50 msec time constant - <10 pg benzene detectivity - >10^4 linear range - can operate in series with FID	+ Gives lower detection limits and great linear range; limitations become chromatographic
•	40 Hz data collection	+ Yields proper area allocation for sharp capillary peaks

LOTUS CONSULTING

5781 Campo Walk
Long Beach, California 90803
310/569-0128 FAX – 714/898-7461