

Stand-Alone Concentrator (SAC)

The Stand-Alone Concentrator from Lotus Consulting is designed to work with most GCs or GC/mass spectrometers. It provides impressive focusing of trace hydrocarbons in ambient air samples into a very small volume to be then injected into an attached gas chromatograph or GC/MS. The system utilizes the Varian 3800 as a controller with built-in high performance sample concentrator and valving. The concentrator trap and some valving are to be located on the companion GC to minimize distance between trap and column. This system handles both pressurized canisters and Tedlar bags without hardware changes. And the system meets the exacting requirements for the US EPA PAMS implementation for speciation of ozone precursors and the California Air Resources Board SOP No. MLD032 for determination of non-methane hydrocarbon compounds in ambient air.

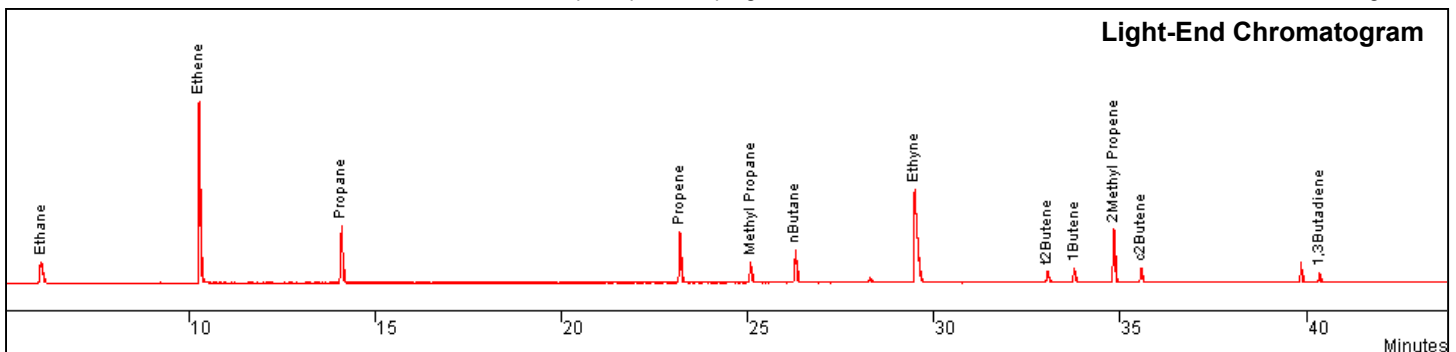
The fully automated system is designed to fully speciate nearly all hydrocarbons from Ethane to Tridecane to levels below 0.2 ppbC (300 ml injection). Samples are loaded through a 16-position (optional 31-position) automated sampler and trapped onto a low-volume cold trap with a mass flow controller (MFC) setting the sample size. The trap is isolated during heat-up and then opened to the column to provide very effective transfer to the analytical column.

Full speciation of hydrocarbons in ambient air is undoubtedly one of the most difficult analyses in gas chromatography. Samples must be concentrated into a small volume to enhance detection. A very large number of possible hydrocarbons (>300) must be “fully” resolved to avoid improperly assigning concentrations from overlapping peaks. And the full gamut of peaks must be identified and quantitated with limited standards (Propane and Benzene). The system involves a cold trap, at least 4 automated valves, 16- or 31-position automated sampler and one workstation. All of these operations utilize nearly all of the powerful and comprehensive capabilities of the Varian 3800 and Star Workstation. Such a complex analyzer 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 Stand-Alone Concentrator can be readily upgraded to a fully functional Trace Hydrocarbon System with the addition of detectors, flow pneumatics and columns.



Ambient air sample from Chongqing, China, October 22, 1999. 300 ml sample loading. Column temperature program: 0 °C, 9 min. hold, 3 °C/minute to 90 °C, 1 min hold, 5 °C/minute to 200 °C. Trap temperature program: -172°C, 9 min. hold, 300 °C/minute to 200 °C. Detector range - 12.



SOLUTIONS TO DIFFICULT ANALYTICAL PROBLEMS...

Clean System Blanks

- Empty tubing or glass beads for trap
 - No thermal breakdown products
 - Maximum temperature limit of 450 °C
- All valves are heated; limited to 350 °C max, except column switching valve (225 °C limit)
- Cryogenic cleansing of purge gas –
vented after each cycle

Efficient Recovery of Light-End Components

- Area reproducibility for Ethane - < 2 %
- Accurate control of trap temperature (< ±2 °C) over complete temperature range
 - Self-calibrating platinum probe (RTD)
- Stable control of trap temperature (< ±2 °C)
 - Proportional controller
 - Close contact between heater, cryogen and trap
 - silver-soldered connection
- Efficient trapping of Ethane on empty tubing or glass beads at -172°C

Full Recovery of “Heavy” Hydrocarbons

- All sample lines heated – no cold spots
- Smooth and inert sample lines – electroformed nickel
- Trap desorbing temperatures to 450 °C
- Effective release (>90 % of C₁₂; >80 % of C₁₃) from empty tubing or glass beads at 200 °C
- Maximum heating rate – 300 °C/min

Elimination of Interfering Artifacts

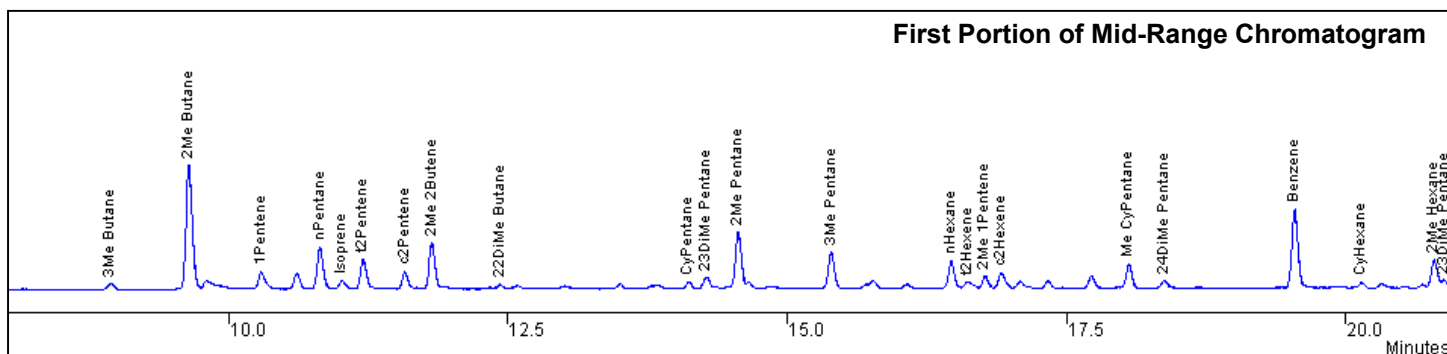
- Empty tubing or glass bead traps standard
- No thermal breakdown that would yield interfering hydrocarbons (i.e. Benzene with Tenax)
- No reaction with NO_x that would yield interfering hydrocarbons (Ethene)
- Trap temperature limit to 450 °C

Sharp Chromatographic Peaks

- Minimum distance from trap to column (~15 cm)
- Trap isolation during trap heating
- Columns attached directly to column switching valve in column oven
 - minimum effect of extra-column volumes at critical chromatographic point
- No refocusing required
- Trap volume:
 - Empty tubing - ~120 microliters
 - Glass bead trap – ~600 microliters
- Ethane peakwidth_{½ height}: < 4 sec

Accurate Measure of Sample Volume

- Sample flow to vent just before trapping
 - Stabilizes MFC
 - Sweeps sample lines with new sample
- Volume-measuring flow path swept with nitrogen prior to trap heating
- Accurate volumes from 25 ml to 2400 ml
- Sample pressure can be below atmospheric and still maintain proper loading
- Sample loading independent of canister pressure



...AND MORE SOLUTIONS

High Concentrations of CO₂

- Mass Flow Controller (MFC) not accurate with major portions of CO₂
 - MFC usually calibrated for air; cannot handle gas mixtures properly
 - 50% CO₂ yields **double** the sample volume!!
- Optional fixed volume sample loop to 100 ml
 - Multiple loadings to trap for larger volumes
 - Full recovery of all hydrocarbons
 - Accurate measure of sample volume - even mixtures

Water Treatment

- Nafion dryer effective for all hydrocarbons on PAMS list
 - Allows full recovery of both light-ends and heavies
- Bypass of dryer
 - Restricts large sample volumes (<100 ml)
 - Allows full recovery, including very polar compounds

Minimal Carry-over

- Nafion dryer, sample pressure regulator and trap continuously purged with nitrogen when sample not loading
- Sample lines swept to vent with new sample just before trapping
- Carry-over « 0.1 %

Quantitation Reproducibility

- Sample lines purged to vent prior to loading
- Measuring flow path swept with nitrogen prior to trap heating
- Typical area reproducibility - < 3 %

Proper Introduction of Surrogate/ Internal Standard

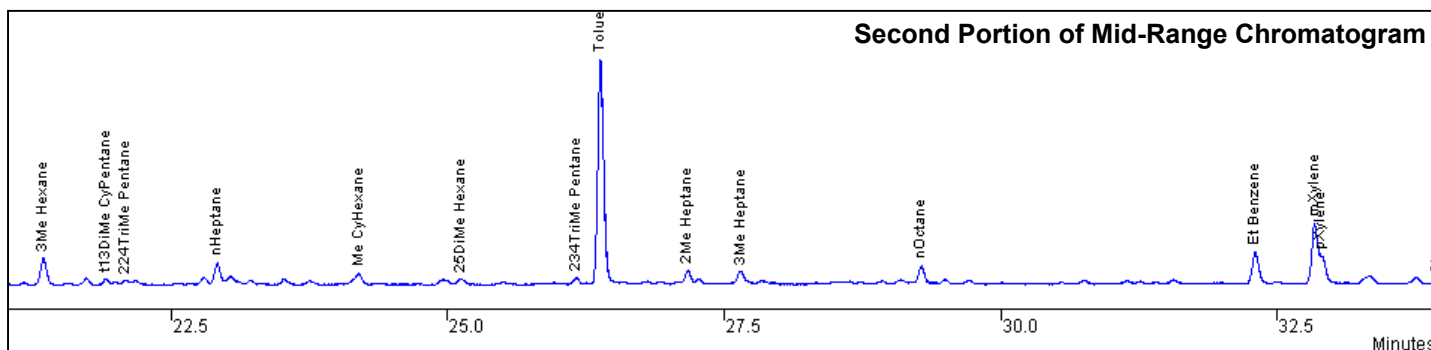
- Fixed-volume sample loop
- Loop comes to atmosphere before injection
- Loaded onto trap as trap is purged

Monitoring of Operations

- Optional recording of MFC flows and reporting of sample volume
- Sample line leak test documentation
- Visual indication of sample loading
- User-specified temperature limits for all thermal zones

Data Processing

- Single stored data file contains run method including trap parameters, stream position, run log and error messages
- Data collection, report generation, system control, custom report and StarFinder operate in Windows 95, 98, NT and 2000
- Multi-level security with passwords
- File names can be labeled with sample id, injection date/time and module source as variables



Specifications

Concentrator Trap

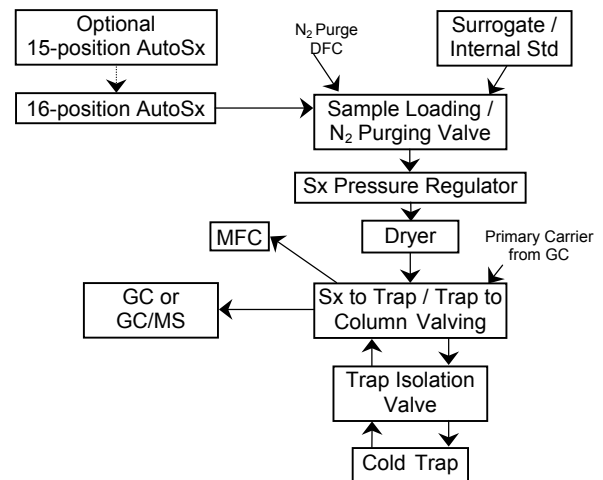
- Maximum heating rate: >300 °C/minute
- Maximum cooling rate: typically >400 °C/minute
- Temperature stability: <2 °C after 1 minute stabilization
- Temperature overshoot:: max. <10 °C, typically <5 °C
- Trap internal volume: ~120 microliters – empty tubing
~600 microliters – glass beads
- All trap settings controlled/monitored through GC with platinum probe (RTD) and proportional controller (PID)
- Programmable in 5 temperature steps with holds

Automated Sampler

- Standard: 16-position; Optional: 31-position
- Micro-electric actuation, self-aligning
- Independently controlled valve oven, mounted in canister tree (optional); heated transfer line to SAC
- Maximum temperature limit: 350 °C
- Position documented in final report and archived with data

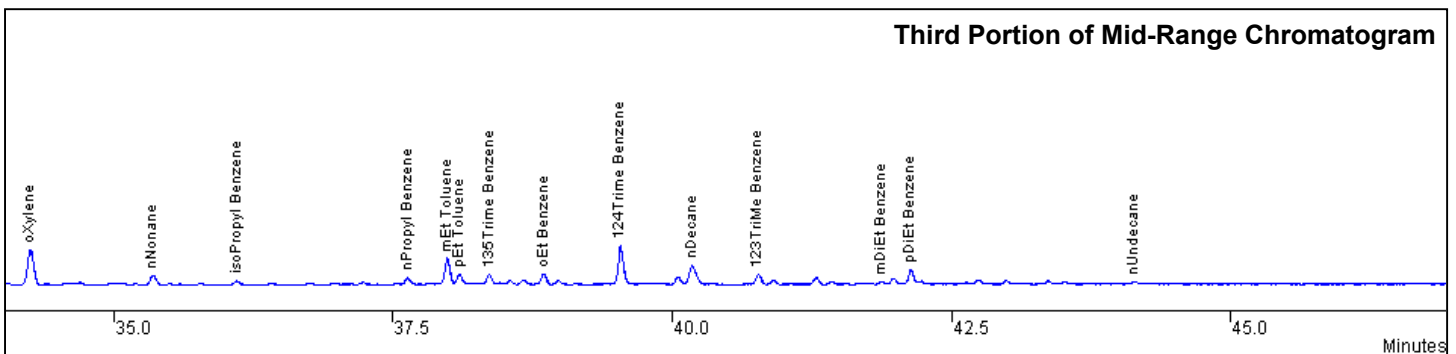
Valving

- Fully automated under time-programmable control of GC
- Valves mounted in heated enclosures
- Micro-electric actuation, easy realignment
- Valves - Valco Series CWT; maximum temperature: 350 °C
- Valves can be turned on/off 21 separate event times within single method
- Automatic addition of surrogate/internal standard



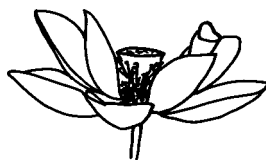
Typical System Performance

- Detection limit: < 0.2 ppbC for all hydrocarbons
- Typical linearity: $R^2 > 0.99995$ from 1.4 to 250 ppbC
- Concentration reproducibility: < 3% at 10X detection limit
- Sample carry-over: < 0.1%



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