

Multi-detector Trace Toxics System (MTTS) by Lotus Consulting

Measurement of toxic chemicals in ambient air involves identifying and quantitating many compounds, including Freons and other halogenates (such as chloromethane, bromoform and hexachlorobutadiene), aromatics (benzene, toluene, xylenes and naphthalene), hydrocarbons (from ethane to tridecane), oxygenates (including methyl tert-butyl ether – MTBE) and reduced sulfurs (such as hydrogen sulfide). The resulting compound list can have well over 300 species. Gas chromatography with capillary columns is the prime analytical technique for most of these, but its usual assortment of detectors is not sensitive enough for direct injection; samples need to be concentrated to bring their ambient levels within range of these detectors.



Multi-detector Trace Toxics System by Lotus Consulting is shown mounted inside a mobile van, with all four detectors fully functional to measure a wide array of toxic compounds in a single sample.

Also, one detector is not suitable to properly detect the whole range of analytes. Mass Spectrometry is the best technique for monitoring many of these species, as it is able to both positively identify the compound and also relate it to a known standard to report the amount present. However, it does not perform very well for light gases, such as hydrogen sulfide (molecular weight too low – MW 32), nor for the light hydrocarbons (including ethane, ethane, propane and the butanes/butanes, again due to their low molecular weights and to the extreme spectral similarities among most hydrocarbons). And since its response to hydrocarbons varies for each hydrocarbon species, a standard with all possible hydrocarbon analytes is mandated, but with well over 200 possible hydrocarbons found in ambient air, this standard is not available.

The Pulsed Flame Photometric Detector (PFPD) is both very sensitive to sulfur compounds and it is quite selective to sulfur-containing species, as it looks for the delayed sulfur emission photometrically to separate the sulfur response from possible hydrocarbon emission interference. This detector works very well for picking out hydrogen sulfide and other sulfides in a complex matrix as ambient air.

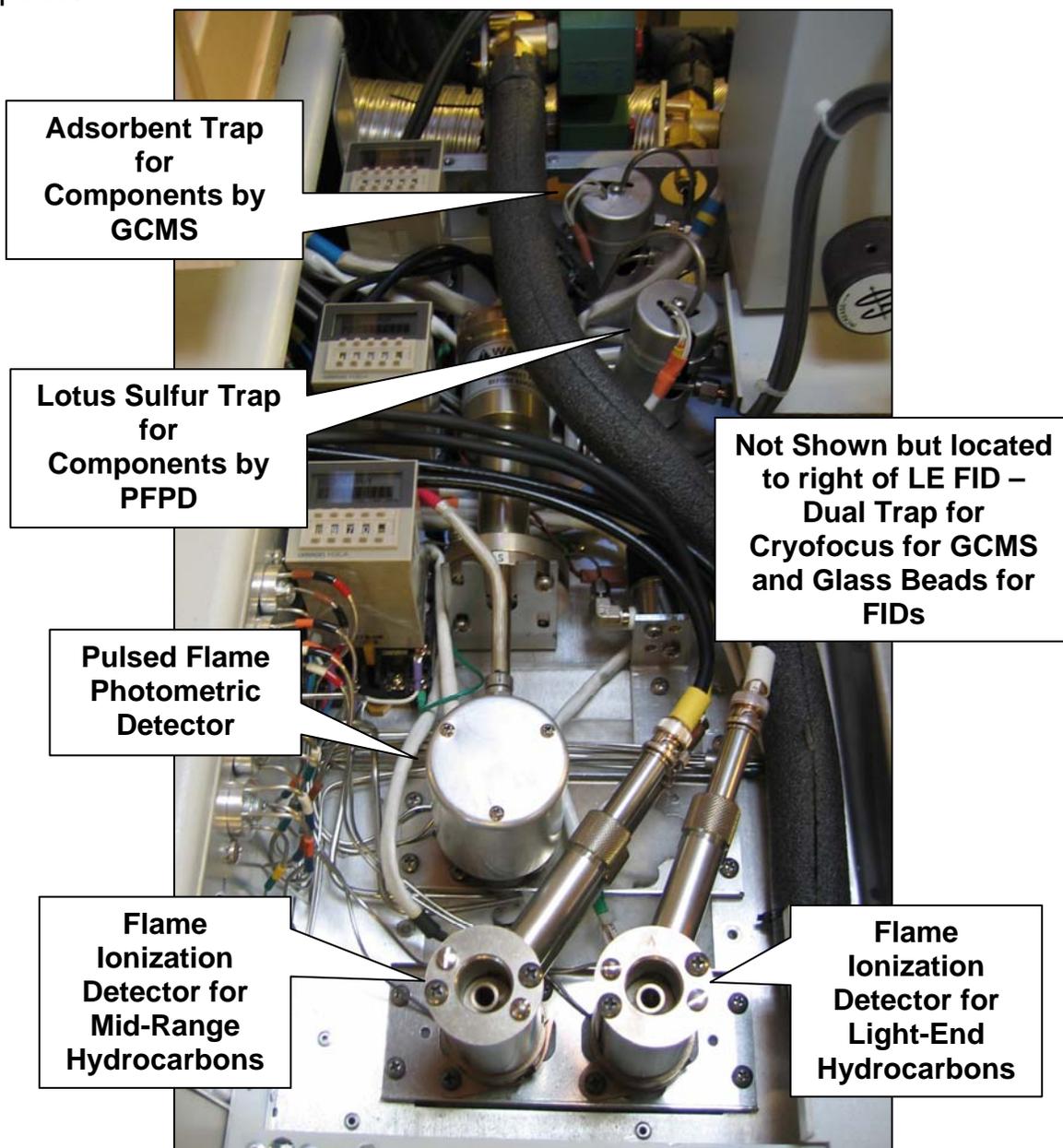
Flame Ionization Detector (FID) is the detector of choice for hydrocarbons, such as alkanes, alkenes, alkynes and aromatics, as it is a very good carbon counter. To quantitate with the FID, we need only to calibrate with propane and benzene and then apply these response factors to all other hydrocarbons. This process fails for oxygenates and halogenates, as these do not burn well in the FID, and we must detect them by Mass Spectrometry.

Obviously, due to the complex nature of the possible species contained in ambient air, a single column cannot fully resolve all analytes well, especially when we need to use multiple detectors. Splitting the sample stream after the concentration steps only deteriorates the sensitivity of the measurement. By employing a separate trapping process for each column/detector system, we can employ the optimum column for each procedure.

Even then, hydrocarbons are not fully separated with a single column, either the light-ends (especially ethane/ethene/ethyne and the butenes) are not adequately resolved, or the analysis time is way too long, or the column is too sensitive to degradation with residual water from the sample. To fully speciate all hydrocarbons, a special column configuration allows the light-end hydrocarbons (ethane through the butanes) to be fully separated on an Alumina PLOT column and the mid-range hydrocarbons (isopentane through tridecane) to be resolved, roughly by boiling points, quite nicely on a second column.

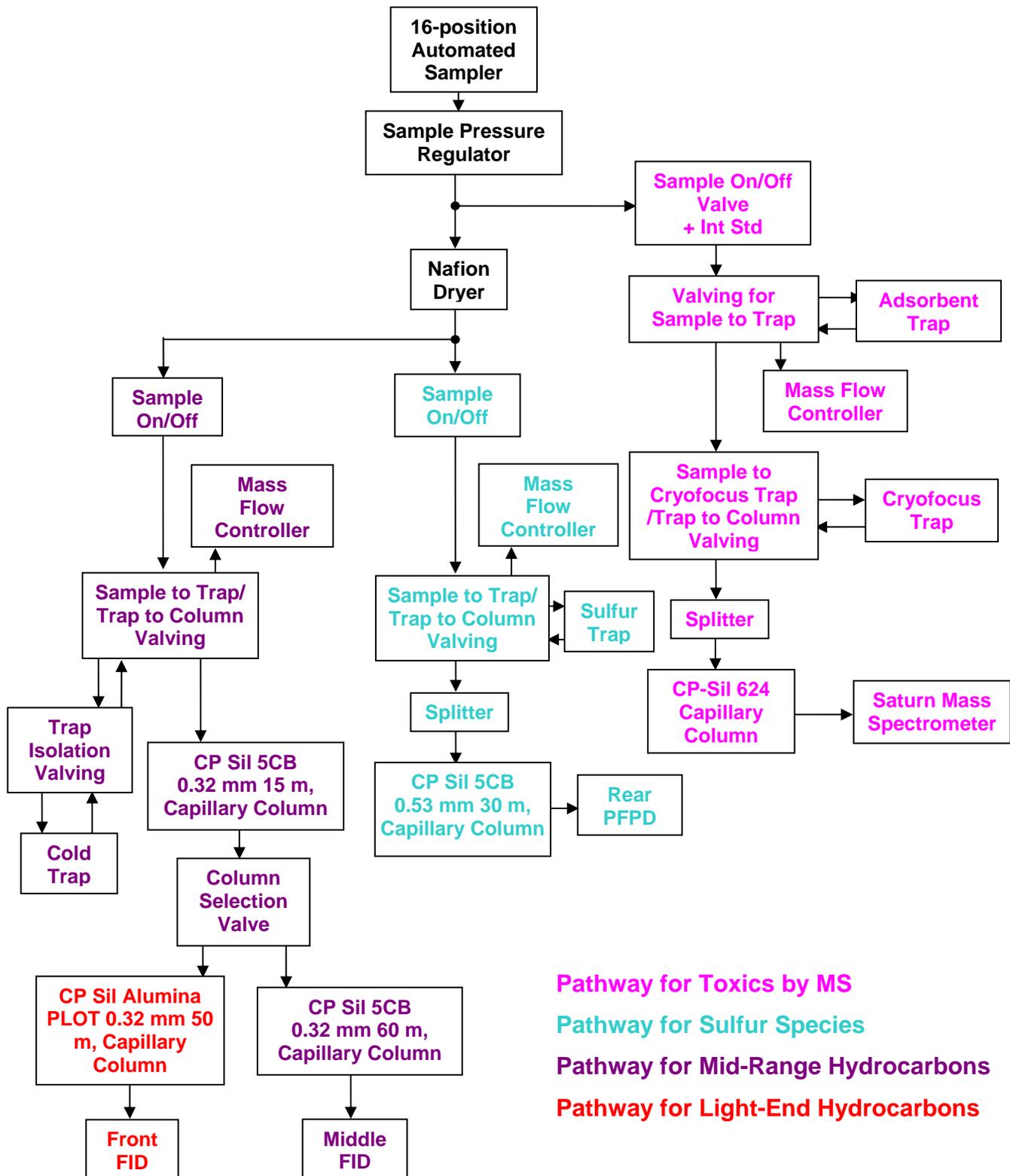
One approach to handling all of these traps, columns and detectors is to break up each one into a separate chromatographic system, with added costs to maintain three or four chromatographs, and longer analysis times as the sample is manually transferred among the systems.

The powerful capabilities of the Varian 3800 Gas Chromatograph, combined with the high performances of the Varian 3800 detectors and the Varian 2200 or 4000 Ion Trap Mass Spectrometer, permit all three techniques to be combined into a single system. Now three concentrator traps, the needed automated valving including multiple-position automated sampler, five required capillary columns, and four detectors can be installed on one machine. The Varian 3800 has the unique capability to control three separate sub-ambient thermal zones for the traps, and seven time-programmable external events for automated valve operations, and to simultaneously and independently operate three GC detectors, along with a Saturn Mass Spectrometer. It has a large enough column oven to accommodate the five needed capillary columns. And it all fits into a nice compact package to minimize required bench space.

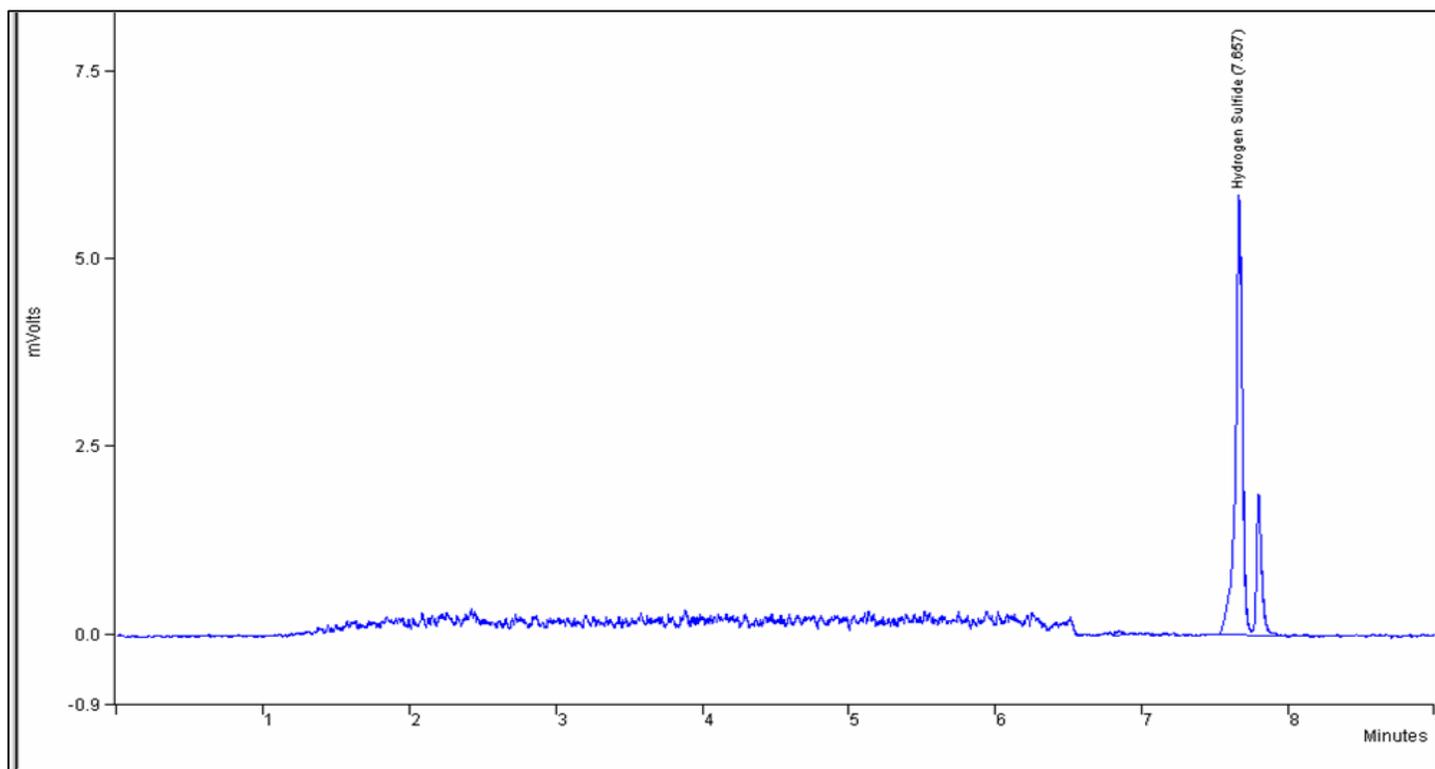


Concentrator traps and detectors are located inside the top covers of the system.

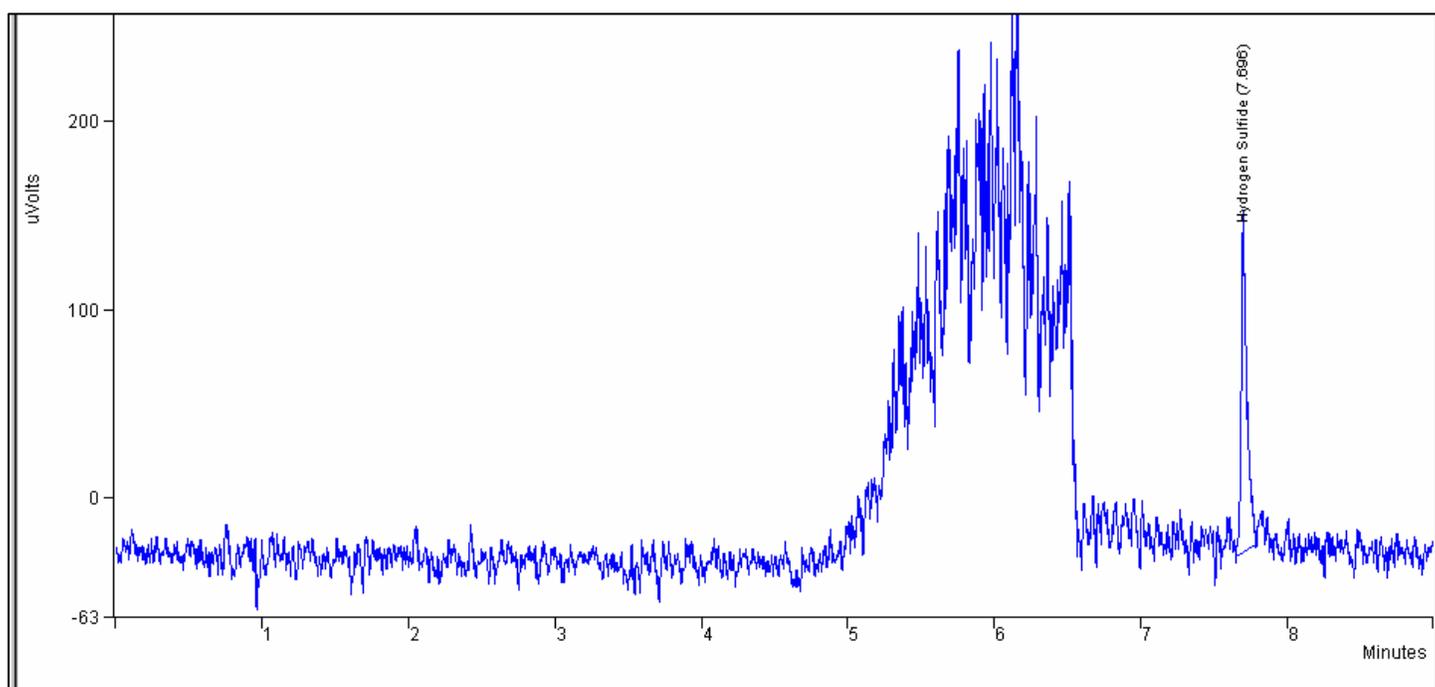
Schematic Diagram for Multi-detector Trace Toxics System



PFPD Chromatogram of Room Air with 200 ml volume injected

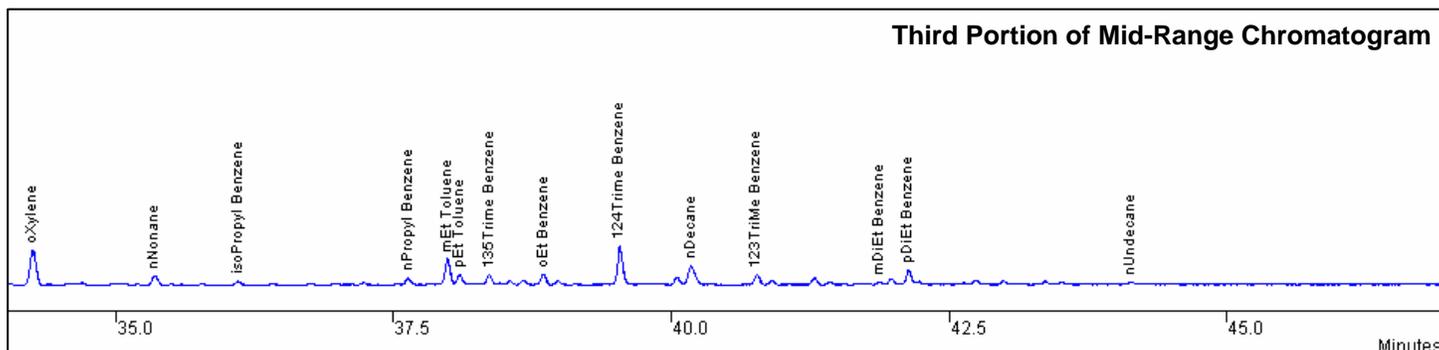
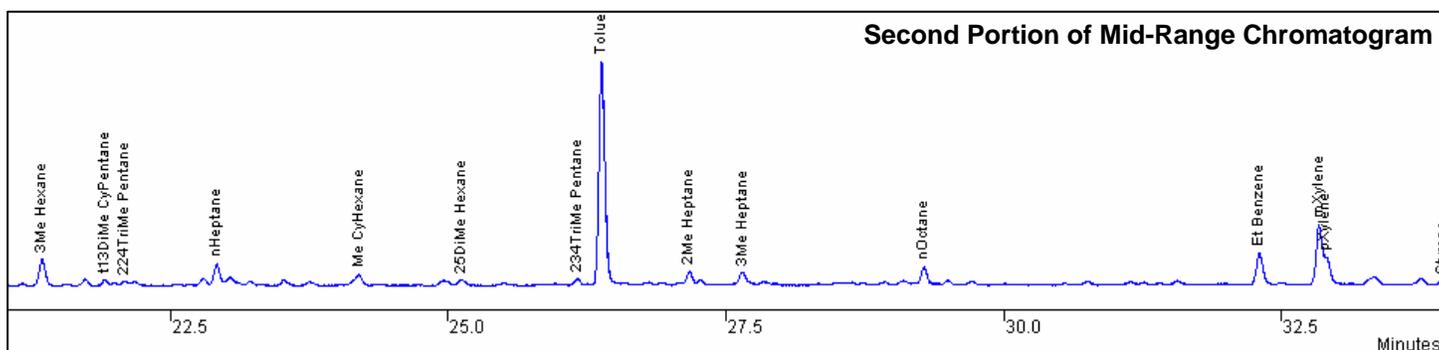
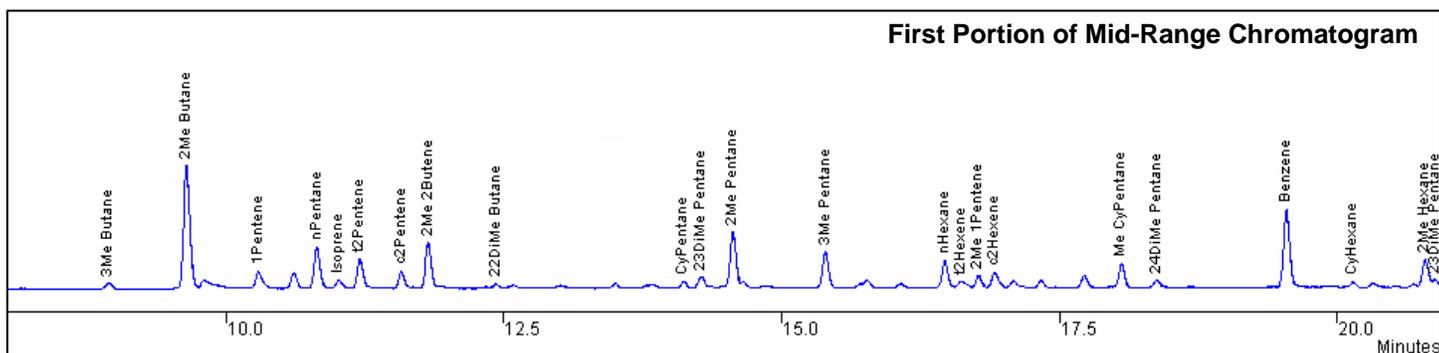
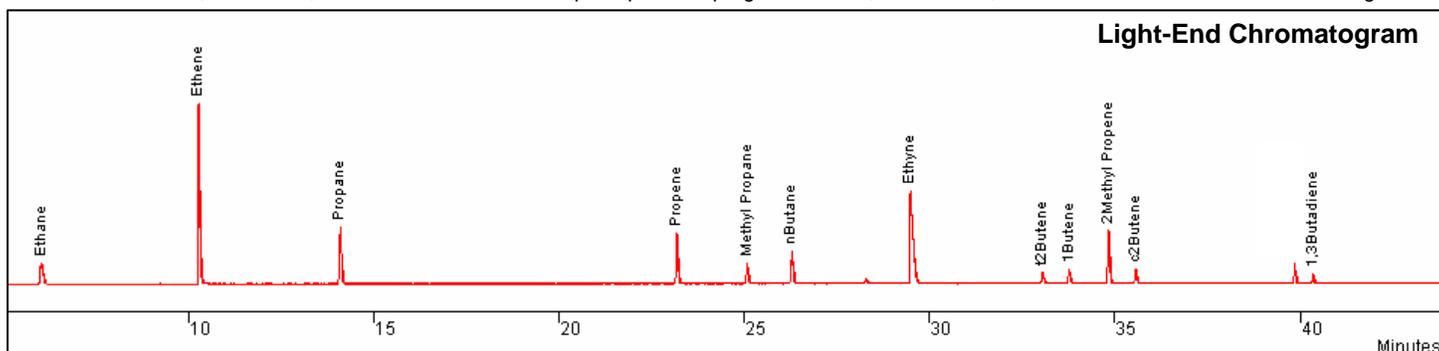


PFPD Chromatogram of 0.3 ppb Hydrogen Sulfide Standard in Nitrogen



Dual FID Chromatograms of Ambient Air with 300 ml volume injected

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.



Features for Multi-detector Trace Toxics System

- Simultaneous and independent measurement of Toxic Compounds by **GCMS**, Sulfur Components by **Pulsed Flame Photometric Detector**, and C₂-C₁₃ Hydrocarbons by **Dual Flame Ionization Detectors** on a single chromatographic system.
- All the features and performances of Ultra Trace Toxics System, Trace Sulfur System, and Ultra Trace Hydrocarbon System by Lotus Consulting, combined into a single system with a compact footprint.
- System software selection for simultaneous display and reporting for all four chromatograms or any combination – no hardware changes required to switch operations.
- Fully automated valving with micro-electric actuators.
- All valves and sample lines heated to above ambient temperatures to prevent condensation of sample components.
- 4, 8, 10, 12 or 16-position automated sampler, with position selected through system control SampleList and position documented in final report.
- Sample volumes loaded with three separate mass flow controllers to allow independent volumes for each detector set.
- Major column flows set with electronic flow controllers with flows documented in final report.
- All GC detector flows controlled with detector electronic flow controllers and documented in final report.
- Single method control of all four detectors and their related flows, temperatures for all three concentrator traps, column temperatures, most carrier flows, automated valving actuations, chromatographic computations and report generation.
- Achievable detection limits –
 - GCMS: << 0.1 ppb V/V for most TO-15 compounds
Note – these limits are achieved with full scan MS spectra
 - PFPD: < 0.03 ppb V/V for reduced sulfurs
 - FID: 0.2 ppb V/V for most hydrocarbons
- Traps materials –
 - ◆ GCMS – primary trap: combo glass beads / carbon adsorbents
– cryofocus trap: 100 µl empty tubing
 - ◆ PFPD – Lotus sulfur trap
 - ◆ FID – glass beads

Note – Tenax is specifically excluded from all traps to eliminate possible contamination due to thermal degradation products from the trap.

Lotus Consulting

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