Lotus Consulting presents:

Hydrogen Fuel Analyzer

The Hydrogen Fuel Analyzer from Lotus Consulting provides impressive separations of trace impurities in hydrogen fuel samples. The system features two Scion 8500 Gas Chromatographs and one Scion 8700 Mass Spectrometer, configured in a primary/secondary setup where a sample is separately loaded into both instruments for a complete assessment of impurities in the hydrogen fuel gas by 10 separate detectors. The system meets the exacting requirements for most of the components in specifications listed in the California Energy Commission, Final Project Report, Measurement and Standards Requirements for Hydrogen and Biodiesel Used as a Transportation Fuel, CEC-600-2020-042, August 2020, www.energy.ca.gov/sites/default/files/2021-05/CEC-600-2020-42.pdf

The fully automated system is designed to be controlled from two Workstations where methods control both gas chromatographs and all six GC detectors. Samples are loaded through two 12-position automated samplers and passed on to separate valving schemes for each of the group measurements.

For Total Sulfur, the mandated level of 4 ppb V/V requires a large sample volume be cryo-focused and then directed to a columns for separation and then to a sensitive and selective pulsed flame photometric detector. The detected sulfurs are then mathematically summed to yield the total result.

H_2 Specification	Value
Hydrogen Fuel Index (minimum, %)	**99.99**
Total Trace Gases (maximum, ppm V/V)	**100**
Water (maximum, ppm V/V)	5
Total Hydrocarbons (maximum, ppm V/V)[i]	**2**
Methane (maximum ppm V/V)	**100**
Oxygen (maximum, ppm V/V)	**5**
Helium (maximum, ppm V/V)	**300**
Nitrogen and Argon (maximum, ppm V/V)	**300**
Carbon Dioxide (maximum, ppm V/V)	**2**
Carbon Monoxide (maximum, ppm V/V)	**0.2**
Total Sulfur (maximum, ppm V/V)	**0.004**
Formaldehyde (maximum, ppm V/V)	0.2
Formic Acid (maximum, ppm V/V)	0.2
Ammonia (maximum, ppm V/V)	0.1
Total Halogenated Compounds (maximum, ppm V/V)	**0.05**
Particulate Concentration (maximum, mg/kg @ NTP)	1

Total Hydrocarbons are readily measured by measuring an aliquot of the sample and directing it to a flame ionization detector without any separation. This detector is a near perfect carbon counter and results are reported as “ppm as Methane”. Methane is also measured by this detector.

Determinations of Oxygen and Argon are problematic in that they do not readily separate under most chromatographic conditions, making their individual measurement difficult, especially when one is dominant. This analyzer employs an Electron Capture Detector for low level of Oxygen, and a separate Pulsed Discharge Detector for Argon.

Nitrogen and Argon, at the levels required, is measurable with a pulsed discharge detector. A typical thermal conductivity detector can often measure down to this specification level; however, as these levels are action levels, the system must be capable of measuring much lower to ensure a proper recording of the actual result.

Helium can only be measured with a thermal conductivity detector with argon as the carrier gas. Care is required to ensure that helium elutes prior to hydrogen to avoid inaccuracies yielded when hydrogen elutes first. The mandated level is quite close to the limits for many detectors; only a top performing thermal conductivity detector can achieve accurate results at this level.

Carbon Monoxide and Carbon Dioxide are readily measured at the specification levels with a reduction catalyst after chromatography to convert them to Methane and detection by Flame Ionization Detector.

No detector is presently available to measure Total Halogenates without first separating and then quantitating each halogenate with a mass spectrometer and summing up the group to report as a total. Although this separation process is very tedious, it does provide an accurate assessment of halogenates present.

Water, Formaldehyde, Formic Acid, Ammonia, and Particulate Size are not measurable by gas chromatography at these levels and must be determined by other techniques.

Formic Acid cannot be measured by gas chromatographic techniques.

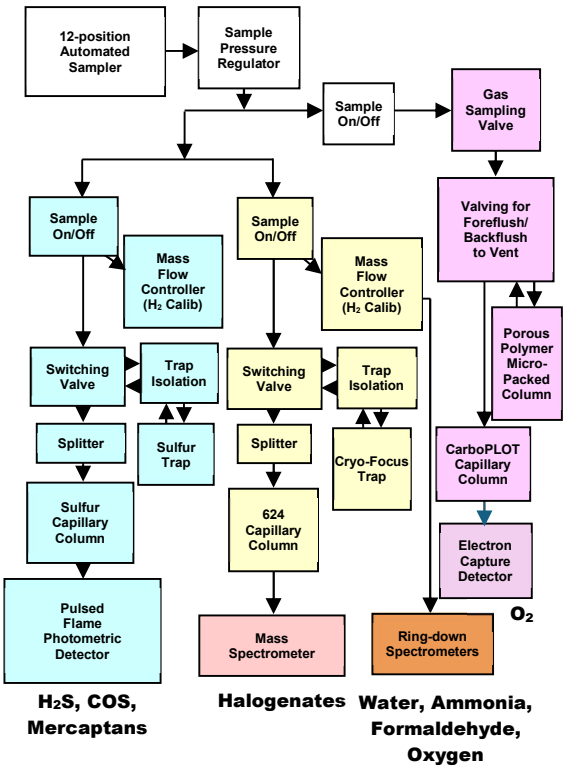
Measurement of these gases requires a very intricate gas chromatograph with multiple valves and columns, six detectors and multiple ovens for columns and valves. Two Scion 8500 Gas Chromatographs accommodate all the hardware needed to yield results from sample loadings. All valve actuations, temperature control for all valves, columns and detectors, data collection from all six detectors and report generation are set up through Workstations and appropriate methods.

System Operations

Samples and standards are attached to the 12-position automated sampler. One is used to load samples into two cryogenic traps and into a fixed volume sample loop. Flow through the sample loop is turned off just before injection to allow the loops to come to consistent pressure.

Two master methods control all operations of all valve actuations, column oven settings (including temperature programming of two independent column ovens), both cryogenic traps, and all parameters for the six required detectors and their auxiliary flows.

For sulfur, the process starts by allowing the sample to flow through to the mass flow controller prior to trapping to flush out the sample line. The volume loaded unto the trap is determined by the sample flow rate and the time interval for trapping. The trap is isolated during heat-up to ensure a sharp injection into the column.



For speciated halogenates, the process is similar to sulfur, except the column and detector are different.

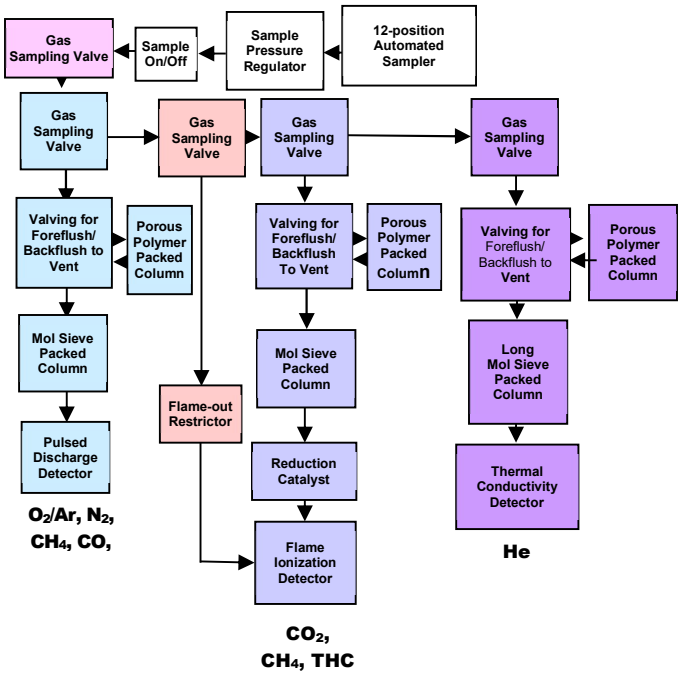
Carbon Dioxide and Methane are measured by injection of a fixed volume sample loop into a column combination selected to strip off heavy interferences and then speciate these analytes with monitoring by flame ionization detector and reduction catalyst in series.

Total Hydrocarbons are measured by simply directing a fixed volume aliquot directly to the flame ionization detector. As this is executed very rapidly, it can be performed in conjunction with the other measurement involving the flame ionization detector.

Helium can only be determined with a thermal conductivity detector with argon as its carrier and is separated from hydrogen by a long molecular sieve column. Potential interferences are stripped away.

Oxygen is measured with an electron capture detector (or Cavity Ring Down Spectrometer). Potential interferences are stripped away.

Nitrogen and Argon concentrations are determined with a pulsed discharge detector.



System Specifications

Concentrator Traps

- Temperature range: -186 °C to 400 °C
- 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 Cryogen usage: < 4 liters per sample
- Trap internal volume: ~100 microliters – cryofocus
- All temperatures at set and displayed to 0.1 °C
- All trap settings controlled/monitored through GC with platinum probe (RTD) and proportional controller (PID)
- Programmable in 25 temperature steps with holds

Automated Sampler

- Standard: two 12-position
- Micro-electric actuation, self-aligning
- Independently controlled valve oven
- Maximum temperature limit: 225 °C
- Sample position selected through workstation's sample lists
- Positions documented in final report and archived with data
- Sample lines heated through control of systems

Valving

- Fully automated under time-programmable control of GCs
- Total of 14 valves independently actuated
- Valves mounted in heated enclosures
- Micro-electric actuation, easy realignment
- Valco CWE Valves; maximum temperature: 225 °C; some with purged housing and special leak testing
- Valves can be turned on/off 31 separate event times within each method

Sampling

- Sample volumes for Halogenates and Sulfurs set with two independent mass flow controllers with volumes user-selectable through workstation from 5 ml to 1600 ml
- All other volumes are set with fixed volume sample loops
- Loaded sample volumes independent of canister pressure

Pneumatics

- Column flows for column systems employ true Electronic Flow Controller (EFC), not pressure control with computed flows
- Temperature-sensitive flow elements maintained at 45 °C
- Flows automatically adjusted for atmospheric pressure

General

- 10" High-resolution full-color touchscreen
- Ethernet communications between GC and Workstation
- Line voltage for GC: 120 V, 20 amperes; for MS: 120V, 15 amperes

Column Ovens

- Four independently controlled column ovens – two temperature programmable and two isothermal
- Programmable temperature range: -99 °C to 450 °C
- Temperature program rate: 0.1 °C/min to 150 °C/min
- Oven cool-down: 400 °C to 50 °C in 4.5 minutes without cryogen
- Programmable in 25 temperature steps with holds
- Coolant timeout to preserve cryogen when system idle
- Negative temperature programming to save coolant during sample loading

Flame Ionization Detector

- Configured for measurement of Total Hydrocarbons , Methane and CO_2 by post-column reduction to Methane
- Detection to < 0.05 ppm V/V Methane
- Automatic flame-out sensing and reignition
- Electronic flow controllers for supply gases

Reduction Catalyst

- Independently controlled oven to 450 °C
- Detection of CO_2 to < 0.05 ppm V/V
- Electronic flow controller for hydrogen flow

Pulsed Flame Photometric Detector

- Configured for sulfur mode
- Detection < 0.03 ppb V/V H_2S with cryotrapping
- Electronic flow controllers for supply gases
- Selectivity of Sulfur/Carbon > 10^5
- Linear range > 10^3

Thermal Conductivity Detector

- Optimized for detection of helium in hydrogen
- Detection < 10 ppm V/V Helium
- Constant mean temperature setting for filaments
- 20X signal amplification
- Four Tungsten-Rhenium filaments in a Wheatstone Bridge
- Electronic flow control of reference gas

Pulsed Discharge Detector

- Configured for measurement of Nitrogen, Argon and Neon and confirmation of Oxygen/Argon, CO_2 and CO
- Detection < 1 ppm V/V Nitrogen

Electron Capture Detector

- No license required (US)
- No wipe required (US)
- Makeup Gas Nitrogen
- Linear Range: > 10^3
- Detection Limit: < 0.5 ppmv Oxygen

Mass Spectrometer

- Optimized for detection of Halogenates
- Detection of Halogenates < 0.1 ppb V/V
- Quadruple Ion Trap Design
- Mass range: 10 to 1200 u, time programmable
- Scan rate: 20,000 u/second
- Resolution: better than unit mass (with 10% valley)
- Dual Stage Turbomolecular pumping rate: 310/400 L/sec
- RJ45 communication between MS and Workstation

Specifications subject to change without notice

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