

Lotus Consulting presents:

Hydrogen Fuel Analyzer

The Hydrogen Fuel Analyzer from Lotus Consulting provides impressive measurements of trace impurities in hydrogen fuel samples. The system features two Bruker 451/456 Gas Chromatographs, with Bruker SCION Mass Spectrometer, configured in a master/slave setup where a single sample is loaded simultaneously into both instruments for a complete assessment of impurities in the hydrogen fuel gas by 6 separate detectors. The system meets the exacting requirements for most of the components* in specifications listed in the California Code of Regulations, Title 4, Division 9, Chapter 6 (www.cdfa.ca.gov/measurement/pdfs/HydrogenPropText.pdf), which now are tied to Society of Automotive Engineers SAE J2719-2011.

The fully automated system is designed to be controlled from a single Workstation where a single method controls both gas chromatographs and all six detectors. Samples are loaded through a 16-position automated sampler 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 column 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.



*The two excluded analytes are formic acid and particulates..

H₂ Specification	Value
Hydrogen Fuel Index (minimum, %)	99.99
Total Trace Gases (maximum, ppm V/V)	300
Water (maximum, ppm V/V)	5
Total Hydrocarbons (maximum, ppm V/V) ⁱ	2
Oxygen (maximum, ppm V/V)	5
Helium (maximum, ppm V/V)	300
Nitrogen and Argon (maximum, ppm V/V)	100
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)	0.01
Formic Acid (maximum, ppm V/V)	0.2
Ammonia (maximum, ppm V/V)	0.1
Total Halogenated Compounds (maximum, ppm V/V)	0.05
Particulates Size (maximum, μm)	10
Particulate Concentration (maximum, μg/L @ 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”.

Determinations of Oxygen and Argon are problematic in that they do not separate under most chromatographic conditions, making their individual measurement difficult. This analyzer employs a high performance electron capture detector, which is very sensitive and selective to Oxygen.

Nitrogen, at the levels required, is measurable with a pulsed discharge detector. A typical thermal conductivity detector can often measure down close to this specification level; however, as these levels are action mandates, 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 nitrogen 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, and Ammonia are not measurable by gas chromatography at these levels and their concentrations are determined by Tiger Optics HALO 3 and Laser Trace 3X Ring-down Spectrometers (www.tigeroptics.com/app/tigeroptics).

Measurement of these gases requires a very intricate gas chromatograph with multiple valves and columns, six GC detectors and multiple ovens for columns and valves. Two Bruker 451/456 Gas Chromatographs, paired as a master/slave, can accommodate all the hardware needed to yield results from a single sample loading. All valve actuations, temperature control for all valves, columns and detectors, data collection from all six detectors and report generation are set up through a single Workstation and method.

System Operations

Samples and standards are attached to the 16-position automated sampler. Each one is simultaneously loaded into two cryogenic traps and into five fixed volume sample loops. Flow through the sample loops is turned off just before injection to allow the loops to come to consistent pressure. All analyses can be concurrent.

A single master method controls 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 GC 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 onto 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.

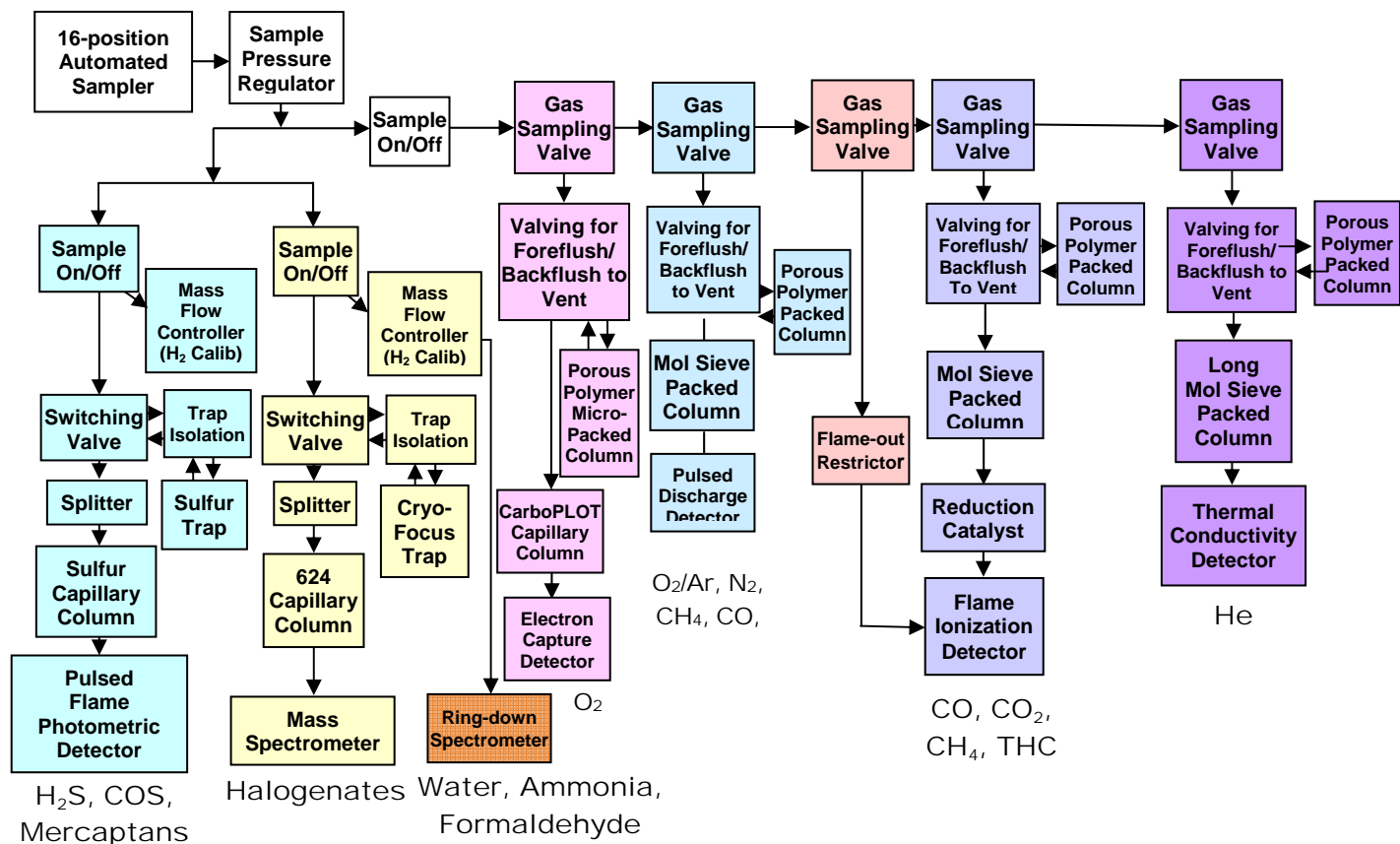
For speciated Halogenates, the process is similar to sulfur, except the column and detector are different.

Measurement of Oxygen by electron capture detector is readily performed at the required level. The columns used here are housed in a separate oven for independent temperature setting.

Other fixed gases 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 two detectors and reduction catalyst all 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 nitrogen as its carrier and is separated from hydrogen by a long molecular sieve column. Again, potential interferences are stripped away.



System Specifications

Concentrator Traps

- Temperature range: -196 °C to 400 °C with LN₂ coolant
- 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
~600 microliters - adsorbent trap
- 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 positions
- Micro-electric actuation, self-aligning
- Independently controlled valve oven
- Maximum temperature limit: 225 °C
- Sample position selected through workstation's sample list
- Position documented in final report and archived with data
- Sample lines heated through control of system

Valving

- Fully automated under time-programmable control of GC
- 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 21 separate event times
within single 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 most 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

- GC touch screen
- 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 100 °C/min
- Oven cool-down: 400 °C to 50 °C in 4.5 minutes without cryogen
- Programmable in 7 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 and Methane, and CO and CO₂ 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 CO₂ and CO L < 0.05 ppm V/V
- Electronic flow controller for catalyst hydrogen flow

Electron Capture Detector

- Set up for measurement of Oxygen in hydrogen
- Pulsed ⁶³Ni configuration
- Possible interferences stripped off to vent
- Detection: < 0.1 ppm V/V Oxygen
- Active cell volume: < 300 µl
- Make-up gas: nitrogen
- General license available in many jurisdictions (avoids radioactive site-licensing)

Pulsed Flame Photometric Detector

- Configured for sulfur mode
- Detection: < 0.03 ppb V/V H₂S with cryotrapping
- Electronic flow controllers for supply gases
- Selectivity of Sulfur/Carbon > 10⁵
- Linear range > 10²

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 and confirmation of Oxygen/Argon, CO₂ and CO
- Detection: < 1 ppm V/V Nitrogen and Argon
- Detector bypass during elution of Hydrogen

Ring Down Spectrometers

- Ammonia
 - Range 0-5 ppmV; LDL <0.35 ppbV
- Formaldehyde
 - Range 0-100 ppmV; LDL <8 ppbV
- Water
 - Range 0-20 ppmV; LDL <1.5 ppbV

Mass Spectrometer

- Optimized for detection of Halogenates
- Detection of Halogenates < 0.1 ppb V/V
- Single Quadrupole Design
- Mass range: 10 to 1,200 Da
- Resolution: better than unit mass (with 10% valley)
- Turbomolecular pumping rate: 350 L/sec
- USB communication between MS and Workstation

Specifications subject to change without notice

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