GC Volatiles Systems



Principal Applications

- USEPA Methods 502.2, 503.1, 601, 602, 8010, and 8021
- ISO 15009 and 15680
- Standard Methods 6200C
- Halocarbons
- Aromatics, olefins
- Solvents
- Hazardous wastes

Principal Applications

- Massachusetts Volatile Petroleum Hydrocarbon (VPH) method
- Benzene, toluene, ethylbenzene, xylenes, and other aromatics (BTEX)
- USEPA Methods 602, 8015, and 8020
- GRO and DRO
- Leaking underground storage tank
 monitoring
- Fuel spills in soils
- Combined total leachate program
- Fuels in water (wastewater)
- ISO 15009 and 15680
- Standard Methods 6200C

System VPH and System BTEX





High-Performance Systems for Detecting Volatile Organic Compounds by Purge and Trap/GC

The System VOC is a fully integrated, high-performance GC system available from OI Analytical for analyzing volatile organic compounds (VOCs) in water and soil. This complete state-of-the-art system extracts VOCs from a representative sample, chromatographically separates, and selectively detects target compounds following specific regulatory methods. Available as a turn-key system from a single vendor, the System VOC includes the new OI Analytical Model 4660 Eclipse Purge-and-Trap Sample Concentrator, Low-Dead-Volume Injector, Agilent® Technologies 6890 GC, column, and choice of optional autosamplers. The System VOC is configured with a tandem PID/ELCD for analyzing halogenated and aromatic species found in USEPA Methods 601/602 and 502.2. Many regulatory methods specify using PID/ELCD tandem detectors for VOC analysis. Systems delivered in the U.S. also include installation and setup.

Operating Principles

The Eclipse Sample Concentrator exhaustively extracts VOCs from the sample, concentrates them on a solid adsorbent bed, and thermally desorbs them onto the GC column. Chromatographic separations are performed on a capillary column for complete resolution of all compounds, including the gases, without subambient temperature programming or cryo-focusing. A tandem PID/ELCD selectively detects, identifies, and quantifies compounds specified in USEPA methods. Systems incorporating mass spectrometers are also available for analysis using USEPA Methods 524.2, 624, and 8260.

- Preconfigured packages include all necessary hardware for analysis: OI Analytical Eclipse Purge-and-Trap Sample Concentrator, Model 5350 Tandem PID/ELCD, Agilent GC, columns, and standards
- Integrated systems emphasize ease of use and accuracy of results
- All systems include an appropriate GC capillary column
- Eclipse Sample Concentrator provides built-in patented Cyclone Water Management[™], rapid trap heating and cooling, and unique protective sparge filter design. Many productivity enhancement options are available
- Eclipse Sample Concentrator and autosamplers provide the fastest overall cycle time in the industry, maximizing your laboratory's productivity
- Tandem PID/ELCD requires only
 one detector port
- Third-generation ELCD features rapid-release reactor design and quick-change, nickel-tube disposable resin cartridges



GC Systems for Volatile Petroleum Hydrocarbons (VPH) and Benzene, Toluene, Ethylbenzene, Xylenes (BTEX) Analysis

The System VPH is a complete package tailored to measure aromatic and aliphatic hydrocarbons in environmental samples using the Massachusetts VPH and similar methods. This affordable system can also be customized for analyzing BTEX and other aromatic compounds, volatile gasoline range organics (GRO), and volatile diesel range organics (DRO).

The System VPH and System BTEX are identical to the System VOC but is customized for analyzing aromatics and aliphatics by using the reasonably-priced tandem PID/FID detector combination. The PID and FID detectors installed in series using OI Analytical's patented tandem configuration generate simultaneous aliphatic and aromatic chromatograms in a single analysis. The PID/FID is the required detector for many regulatory methods such as USEPA Method 8015, Massachusetts VPH, and many GRO methods. Like the System VOC, many methods in use in the world specify PID/FID tandem detectors for VOC methods.

Purge-and-Trap Autosamplers

OI Analytical offers a full range of autosamplers for use with the System VOC, System VPH, and System BTEX including the Model 4551A Water Autosampler, Model 4552 Water/Soil Autosampler, and AMPS six-port, on-line, continuous multipoint sampler.

- Standard system package includes all necessary hardware for analysis: Eclipse Purge-and-Trap Sample Concentrator, Model 4450 Tandem PID/FID, Agilent GC, appropriate columns, and standards
- System VPH includes the capillary column specified in the Massachusetts VPH method
- Tandem PID/FID provides excellent sensitivity and linearity, while eliminating interconnecting transfer lines
- PID hidden-window design prevents lamp fouling
- Third detector capability
- Systems are available for customized requirements or other related methods such as BTEX, GRO, and DRO
- On-line, automated, multipoint water monitoring system is also available
- Automated pH measurement provides confirmation of proper preservation technique



1.	Methanol
2.	<i>n</i> -Pentane
3.	2-Methylpentane
4.	Methyl-tert-butyl ether
	(MTBE)
5.	2,2,4-Trimethylpentane
6.	Benzene
7.	Toluene
8.	<i>n</i> -Nonane
9.	Ethylbenzene
10,	<i>m</i> /p-Xylene
11.	
12.	o-Xylene
13.	<i>n</i> -Decane
14.	1,2,4-Trimethylbenzene
16.	Naphthalene
17.	2,5-Dibromotoluene

General Specifications

Standard System VOC Hardware

- Eclipse Purge-and-Trap Sample Concentrator
- Model 5350 Tandem PID/ELCD System
- Agilent GC
- OI Analytical low-dead-volume injector

Performance Specifications

Trap

- >1,000 °C/minute heating rate to 300 °C; 450 °C maximum
- >240 °C/minute cooling rate (200 °C to 30 °C in <50 sec)

Water Management

- Eliminates all but ~0.25 μL (0.063 μL/minute) of trapped desorb water (>96% water removed)
- Operates at *ambient* temperature
- Water removal at level equivalent to condensation at 4.8 °C

General Specifications

Standard System VPH and System BTEX Hardware

- Eclipse Purge-and-Trap Sample Concentrator
- Model 4450 Tandem PID/FID detector system
- Agilent GC
- OI Analytical low-dead-volume injector or Agilent split/splitless injector
- Method-appropriate GC capillary column

Performance Specifications

Minimum Detectable Mass

• <40 pg benzene

- FID
- 5 pg carbon/second propane

ELCD

- Minimum Detectable Mass
- Halogen: 1 pg lindane
- Maximum Detectable Mass
- Halogen: 5 µg lindane

PID

Dynamic Range

• Greater than 10⁶

Sensitivity

• <40 picograms benzene

Communication Interface

• Ethernet/LAN connection to the sample concentrator

Detector Output

• 1V full-scale analog voltage

Requirements

Power Requirements

- 115 VAC (±10%), 60 Hz
- 230 VAC (±10%), 50 Hz

Benchspace Requirements

• 105.4 linear cm (41.5 inches) for total basic system

Dynamic Range

PID

- >10⁶
- FID
- $\pm 10\%$ over a 10^6 range

Communications Interface

• Ethernet/LAN connection to the sample concentrator and GC

Detector Output

• 1V full-scale analog voltage

Requirements

Power Requirements

- 110 VAC (±10%), 60 Hz
- 220 VAC (±10%), 50 Hz

Benchspace Requirements

• 109.2 linear cm (43 inches) for a standard system

Gas Requirements

• Ultrahigh purity H₂, and He (99.999% purity or better)

Gas Requirements

• Ultrahigh purity H₂, and He (99.999% purity or better)

Options

- Choice of three autosamplers including multipoint sampling for homeland defense applications
- Foam Buster[™], Foam Sensor[™], Sparge Overfill Sensor[™], Infra-Sparge[™] Sample Heater
- pH Express[™] for automated pH measurement
- Choice of capillary GC columns
- Extended warranties and training
- Eclipse Sample Concentrator supports international languages, as well as English

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Several factors including GC, column, electrolyte, and compound class can affect performance.

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S-PRO 3200 GC System for Sulfur Analysis



Superior Selectivity and Sensitivity



The ability to detect and measure sulfur contaminants in gases is critically important for efficient operation of industrial processes and to control product quality. S-PRO 3200 GC systems have proven highly effective in demanding sulfur analysis applications.

- Sulfur content in Liquefied Petroleum Gas (LPG)
- COS in ethylene and propylene feedstock
- Sulfur in natural gas
- Impurities in beverage grade CO₂
- Semiconductor and industrial gas purity
- Quality control in gas production and blending operations

Gas and Liquid Phase Petrochemicals

Carbonyl Sulfide in Propylene (ASTM D5303)

Sulfur Compounds in Natural Gas (ASTM D5504 & D6228)

Ultra-Low sulfur Gasoline (ULSG)

Ultra-Low sulfur Diesel (ULSD)

Thiophene in Benzene (ASTM D4735-02 & D7011)

Sulfur Compounds in Light Petroleum Liquids (ASTM D5623)

Jet Fuel

Naphtha

Crude & Synthetic Oils

Furnace Oil

Light Cycle Oil (LCO)

Ethylene and Propylene Feedstock

Propylene is a co-product from steam cracking of ethylene. Carbonyl sulfide (COS) is a major contaminant in propylene feedstock and can destroy expensive catalyst beds used in polymer production and other processes if not removed. The accompanying chromatograms show the hydrocarbons and COS present in a feedstock gas before separation of propylene and ethylene components, and prior to sulfur scrubbing.



Natural Gas

Natural gas containing hydrogen sulfide or mercaptans is referred to as "sour" gas. The concentration of hydrogen sulfide in natural gas ranges from barely detectable levels to more than 0.30% (3,000 ppm).¹



Sulfur Impurities in CO₂

Early detection and control of H_2S and COS is an important consideration in the production of food-grade CO₂ because the presence of these compounds can impart undesirable odors and flavors to carbonated beverages.



^{1.} - The Chemistry and Technology of Petroleum, Marcel Dekker, Inc., 1991.

Advanced Detection Technology



S-PRO 3200 Gas Chromatograph

The S-PRO 3200 is a custom-configured gas chromatograph for selective, high-sensitivity measurement of sulfur compounds in gas-phase samples and Liquefied Petroleum Gas (LPG) streams such as propylene and ethylene.

The key technology within the S-PRO 3200 system is OI Analytical's patented* Pulsed Flame Photometric Detector (PFPD). Our Model 5383 PFPD has a linear, equimolar response to sulfur allowing selective measurement of individual sulfur species from low ppb to ppm levels, and total sulfur as the sum of individual peaks. The unique capability to obtain simultaneous sulfur and hydrocarbon chromatograms from a single PFPD detector sets it apart from other sulfur detection technologies. Reliable and cost-effective, the 5383 PFPD uses significantly less gas than SCDs or FPDs and requires less maintenance.



Thirteen Light Sulfur Compounds in a Propylene Standard on the S-PRO 3200

PFPD Principle of Operation

A combustible mixture of H_2 and air is introduced and fills the detector body and cap from the bottom up (1). The combustion mixture is ignited in the cap (2). The resulting flame propagates along the pathway consuming the H_2 /air mixture (3). Compounds eluting from the GC column are combusted within a quartz combustor and emit light at element-specific wavelengths (4). The flame is extinguished when it reaches the bottom of the detector, and excited species continue to fluoresce for up to 25 milliseconds. Emissions from the excited species pass along a light pipe, and selected emissions are transmitted through an optical filter to a photomultiplier tube for detection (5). The entire pulsed flame cycle is repeated approximately 3 to 4 times per second.



Gasoline



Diesel Fuel



PFPD - Pulsed Flame Photometric Detector

The PFPD is widely used in laboratory and process gas chromatographs to analyze sulfur species and total sulfur levels in liquid-phase petrochemicals.



The Complete Solution for Sulfur Analysis

Chemically-Inert Components

Volatile sulfur compounds such as H₂S, methyl, and ethyl mercaptan adsorb strongly to the surfaces of untreated metals. The entire sample pathway of the S-PRO 3200 is constructed with Sulfinert[®] or SilcoNert[®] 2000 treated components to prevent adsorption of sulfur compounds that could cause inaccurate results.



Automated Injection System

Automates sample introduction, system calibration, and QC checks

Pulsed Flame Photometric Detector

Produces simultaneous, mutually selective sulfur and hydrocarbon chromatograms

OI Analytical has integrated a number of special design features into the Agilent 7890 GC platform to provide unique analytical and performance capabilities.

Permeation Oven

- Accommodates up to 5 permeation devices
- Pure sulfur compound diffuses across a permeable Teflon[®] barrier at a temperature-dependent rate
- Precise oven temperature control produces a constant diffusion rate
- Controlled, measured flow of dilution gas creates an accurate gas standard for calibration
- Agilent 7890B keypad- or ChemStation-controlled temperature and dilution gas flow



- 4-port sample selection valve enables sample selection from a gas stream, or to deliver calibration and check standards from the permeation oven
- 6-port gas-phase switching valve with sample loop injects samples through the Volatiles Interface into the GC column

Sulfur Detection - PFPD

- Superior sensitivity and increased selectivity compared to conventional FPDs
- Better long-term stability and less maintenance than SCD or XRF
- Quick, easy calibrations
- Self-cleaning design



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Volatiles Interface

• Optimized for ultralow dead volume flow rates, inertness, and ease of column installation



S-PRO 3200 Specifications

Detectivity	Sulfur < 1 pg S/second
Selectivity	At optimum detectivity levels: Sulfur > 10 ⁶ S/C
Permeation Oven	Temperature range: 30 - 75 °C ±0.05 °C
OI Volatiles Interface	Effective split range: Splitless to 150-to-1 Maximum temperature: 325 °C
GC Column	Agilent J&W Select Low Sulfur Column, 80 m x 32 mm ID Maximum temperature: 260 °C
5383 PFPD Specifications	
Detectivity	
Sulfur	<1 pg S/sec
Phosphorus	<100 fg P/sec
Sensitivity	
Sulfur Signal-to-Noise	>300 (at 10 pg S/sec elution rate peak-to-peak noise)
Drift (S or P)	<10x peak-to-peak noise in 20 min
Selectivity (at Optimum Dete	ctivity Levels)
Sulfur	>10 ⁶ S/C
Phosphorus	>10 ⁵ P/C (selectivity is adjustable with a trade-off in detectivity)
Detector Linearity	
Sulfur	Quadratic in response; linear to approximately 2.4 orders of magnitude Detector (nonlinear) dynamic range ~3 orders of magnitude
Phosphorus	First order linear over approximately 5 orders of magnitude
Response Uniformity	Equimolor ±8% (S,P)

<0.2 sec in S and P

O·I·Analytical

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FBA 5320

Laboratory Fluorinated By-products Analyzer Detection of Fluroinated Contaminants in Hydrocarbons



Selective Detection of Fluoride Compounds



FBA 5320 Fluorinated By-products Analyzer

The OI Analytical FBA 5320 is a laboratory fluorinated by-products analyzer (FBA) designed to quantify total fluorinated contaminants in samples from liquid propane or liquid butane process streams. The analyzer is based on an Agilent 7890 gas chromatograph (GC) equipped with an automated liquid sampling valve for sample introduction, a packed-inlet injection port, a stainless steel column, and 5320 Electrolytic Conductivity Detector (ELCD) configured for fluoride detection. The FBA 5320 accurately detects total fluoride concentrations in the low parts-per-million (ppm) range.

Principle of Operation

The automated liquid sampling valve injects a 2-µL aliquot of an LPG sample into the 1/16-inch O.D. x 55-foot long stainless steel column maintained at 100 °C inside the GC oven. Fluoride compounds eluting from the GC column enter the base of a high-temperature reactor installed in a GC detector port. The compounds are catalytically converted to ionizable HF gas inside the Nickel reaction tube. The gaseous reaction products are carried into the detector cell where they are dissolved in a deionized solvent, increasing the electrolytic conductivity of the solution. The detector amplifies this instantaneous change in conductivity, producing a signal proportional to the mass of fluoride in the original sample. The signal is output to a chromatographic data handling system (supplied by the customer) to display and report the total fluoride concentration.



Total Fluoride Measurement in LPG Streams

HF Alkylation

The Hydrofluoric (HF) Alkylation process catalytically combines C3 - C5 olefins with isobutane to produce motor fuel alkylate.¹ There is an increasing demand for alkylate because of its properties as a low-sulfur, high-octane blending component for unleaded and reformulated gasoline. The products of this process, propane, butane, alkylate contain traces of HF and organofluoride by-product compounds.

Elevated levels of residual fluorinated by-products in process streams can cause serious problems in refinery operations including corrosion, catalyst poisoning, down-time, and lost production. The fluoride level in butane process streams can range from 100 - 400 ppm.² Fluorinated by-products are removed by catalytic decomposition of the organofluorides at 177- 220 °C to HF and olefins. The HF released in this process is removed by adsorption onto a bed of alumina forming aluminum fluoride.



¹ Adsorbents for Motor Fuel Alkylation Process, www.uop.com

² Fluoride Removal in the HF Alkylation Process, www.engelhard.com



FBA 5320 Specifications

Operating Principle	Fluorinated compounds eluting from a GC column are catalytically reduced in a high temperature micro- reactor and converted to HF gas. The HF gas dissolves in a deionized solvent increasing the electrolytic conductivity of the mixture. The change in conductivity is directly proportional to the mass of fluoride in the sample.
Measurement Technique	Electrolytic Conductivity Detector (ELCD)
ELCD Reaction Gas	Hydrogen 100 mL/minute
ELCD Reactor Temperature	Range: 800-1100 °C in 100 °C increments Operation: 900 °C (Stability: ± 1 °C)
ELCD Dynamic Range	5 x 10⁵
FBA 5320 Range	0.02-100 + ppm
Selectivity in Fluoride	Fluoride/Hydrocarbon > 10 ⁶ (Other halogens will respond)
Detector Output	0-1 V or 0-10 V full scale analog voltage
Gas Chromatograph (GC)	Agilent 7890 GC equipped with OI Analytical 5320 ELCD detector. Gases EPC flow controlled.
GC Dimensions	49 cm H x 58.4 cm W x 53.3 cm D (19.3" H x 23" W x 20.9" D)
GC Weight	50 kg (110.5 lbs)
GC Column	54.8 feet long x 1/16 inch O.D. stainless steel
Carrier Gas	Helium 8-13 mL/minute
Liquid Sampling Valve	Air actuated
Sample Volume	2 μL
ELCD Controller Dimensions	21 cm H x 12.7 cm W x 30.5 cm D (8.3" H x 5" W x 12" D)
ELCD Controller Weight	3.8 kg (8.4 lbs)
Gas Requirements	Hydrogen, ultrahigh purity > 99.999% Helium, ultrahigh purity > 99.999%
Power Requirements	90-260 VAC (+ 10%), 47-63 Hz, 200W (Surge-protected power supply required)

PFPD - Pulsed Flame Photometric Detector



The PFPD is widely used in laboratory and process gas chromatographs to analyze sulfur species and total sulfur levels in liquid-phase petrochemicals.



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