GC3-NGA AUG 2011

GC3-Natural Gas Analysis Advanced User Guide

Manual Information

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Changes to User Guide

Summarize requested modifications to this user guide in an e-mail and/or annotate the PDF file and e-mail change requests to ***techdoc@iodp.tamu.edu.***

User Guide Contents

Introduction

Overview

The absolute quantity of hydrocarbons combined with the potential for trapping and accumulating hydrocarbons is the primary safety risk during shipboard operations. Gas monitoring via gas chromatography (GC) analysis is a means of quanitfying the risk posed by these factors. ***Figure 1*** depicts the safe ranges for gas concentrations (C_1/C_2) vs. temperature.

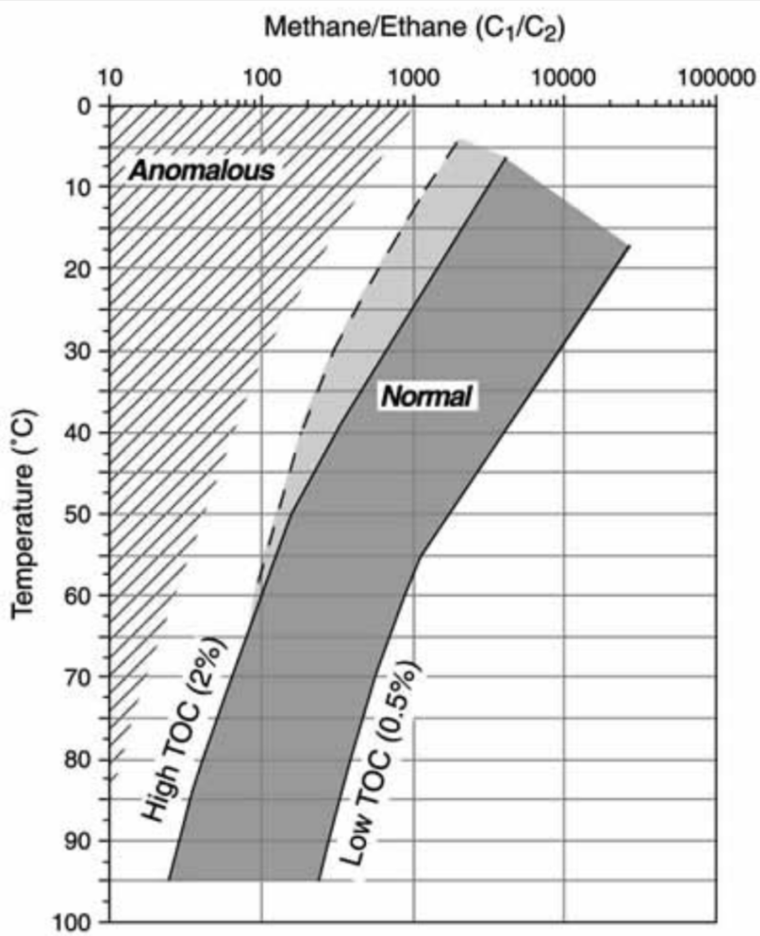

Figure 1. Risk Assessment for Drilling Safety (IODP).

Hydrogen sulfide (H_2S) is another significant risk factor for individuals working in the area. Early detection of H_2S is accomplished by emergency monitors on the drill floor, and later quantification is performed on the natural gas analyzer (NGA).

Hydrocarbon Generation

Hydrocarbon generation in sediments results from thermal decomposition (maturation) of biogenic organic matter (e.g., Tissot and Welte, 1984). C_1–C_4 hydrocarbons may be generated in significant quantities in sediment via two processes:

- **Biogenic:** biogenic hydrocarbons are typically characterized by methane. They are produced in a sulfate-free environment via the reduction of dissolved bicarbonate.
- **Thermogenic:** thermogenic hydrocarbons are produced in sediments in direct proportion to temperature. C_5 and other heavy hydrocarbons almost always result from thermal generation of hydrogen-rich organic matter. Typically, a temperature of ~100°C or greater is required for these products to become significant.

The evolution of sedimentary biogenic organic matter under increasing burial depth and consequent temperature rise is divided into three stages:

- Diagenesis
- Catagenesis
- Metagenesis

Diagenesis

Diagenesis refers to the biological, physical, and chemical alteration of sedimentary organic matter that occurs at low temperature (<50°C) in relatively recently deposited sediments (Peters et al., 2005).

Catagenesis

Catagenesis, the principal zone of oil formation, refers to a temperature range of 50°C~150°C. Liquid and gaseous hydrocarbons together with organic compounds with heteroatoms (oxygen, sulfur, and nitrogen) are released from the kerogen (***Figure*** 2), so the catagenesis stage is called the "oil window."

Metagenesis

The last stage of sedimentary organic matter alteration is metagenesis. Dry gases (mainly methane) are derived from liquid hydrocarbon accumulation in the crust (***Figure 3***). C_1–C_4 hydrocarbons may be generated in significant quantities in sediment via biogenic and thermogenic processes.

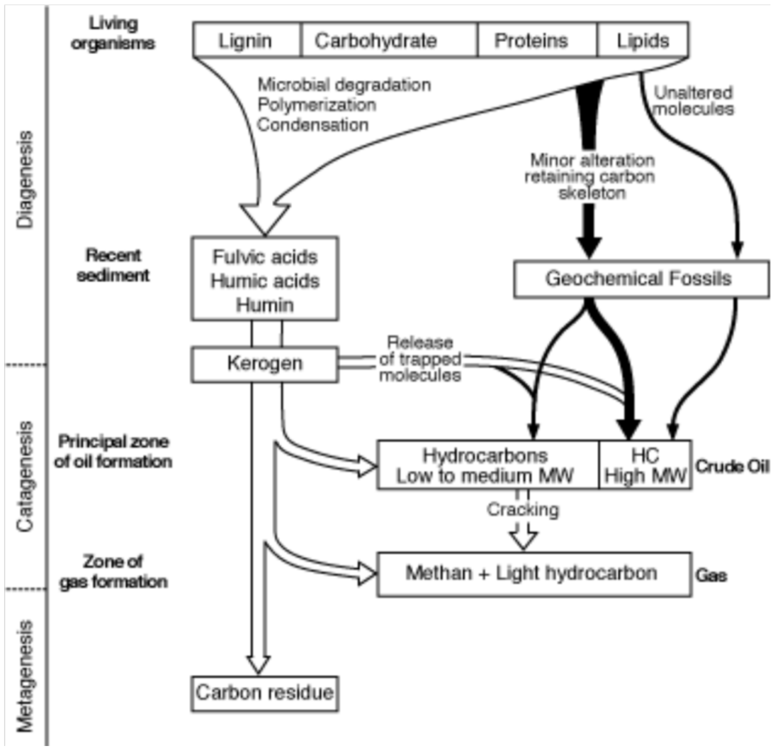

Figure 2. Hydrocarbon Formation Pathways in Geological Situations (Rullkotter, 1993).

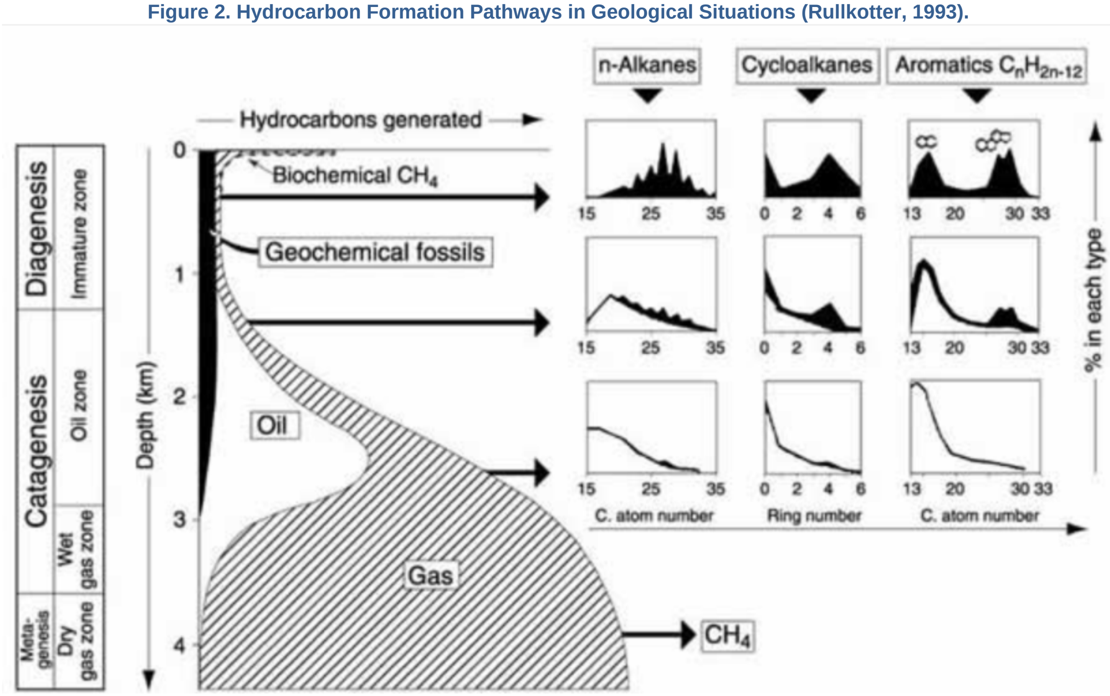

Figure 3. Hydrocarbon Generation Resulting from Burial of Organic Matter during Geologic Time.

Hydrogen Sulfide

Sulfate reducing bacteria produce H_2S in euxinic sediments (Raiswell and Berner, 1985). Biogenic alteration of organic matter may occur in a relatively shallow part of the sediment. Thermochemical sulfate reduction of sulfate by hydrocarbons in reservoirs occurs under high temperature (>127°C ~ 140°C) (e.g., Orr, 1974; Worden et al., 1995).

References

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Theory of Method

The hydrocarbon monitoring system consists of two instruments that monitor gases in core headspace and core void samples:

- **GC3:** Agilent 6890 gas chromatograph (GC) with a flame ionization detector (FID). This instrument measures C_1–C_6 hydrocarbons.
- **NGA:** Agilent 6890 GC with an FID and a thermal conductivity detector (TCD). This instrument measures C_1–C_6 hydrocarbons as well as N_2, O_2, CO_2, CS_2, and H_2S gases.

GC3

The GC3 is used to determine the concentrations of the following light hydrocarbon gases:

- Methane (CH_4)
- Ethene (C_2H_4)
- Ethane (C_2H_6)
- Propene (C_3H_6)
- Propane (C_3H_8)

The GC3 instrument has a 1/6-inch VALCO union injector with a 2 µm stainless steel screen and a 10 port VALCO valve that is electrically switched (***Figure 4***). An 80/100 mesh 8 ft HaySep "R" packed column (2.0 mm ID x 1/8 inch OD) is installed in the oven.

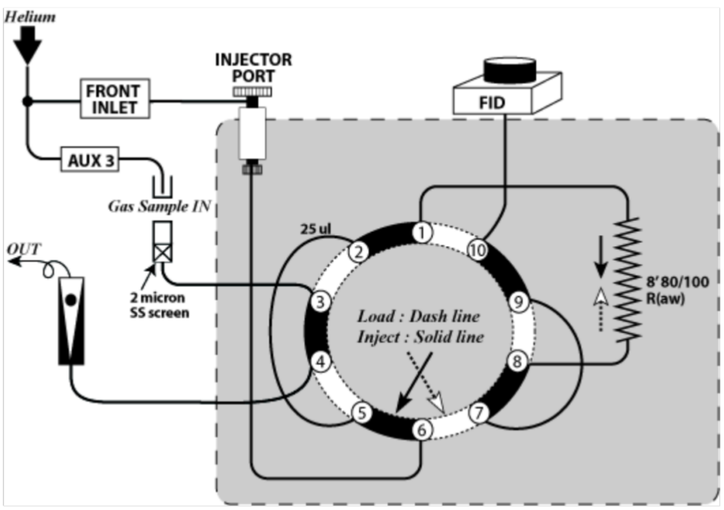

Figure 4. Schematic of Sample Gas Line in the GC3.

NGA

The NGA is used to determine the concentrations of nonhydrocarbon gases along with hydrocarbons from C_1 to C_7. The analytes measured on this instrument are:

- Nonhydrocarbons
- Nitrogen (N_2)
- Oxygen (O_2)
- Carbon dioxide (CO_2)
- Carbon disulfide (CS_2)
- Hydrogen sulfide (H_2S)
- Hydrocarbons
- Methane (CH_4)
- Ethene (C_2H_4) + Ethane (C_2H_6)
- Propene (C_3H_6) + Propane (C_3H_8)
- n-Butane (C_4H_{10})
- iso-Butane ($CH_3–C_3H_7$)
- n-Pentane (C_5H_{12})
- iso-Pentane ($CH_3–C_4H_9$)
- n-Hexane (C_6H_{14})
- iso-Hexane ($CH_3–C_5H_{11}$)
- n-Heptane (C_7H_{16})
- iso-Heptane ($CH_3–C_6H_{13}$)

The TCD flow path contains the following columns (***Figure 5***):

- 6 ft x 2.0 mm ID stainless steel column packed with Poropak T (50/80 mesh)
- 3 ft x 2.0 mm ID stainless steel column packed with molecular sieve 13x (60/80 mesh)
- 6 ft x 2.0 mm ID stainless steel column packed with 80/100 mesh HaySep R (acid wash)

The FID flow path has a 60 m x 0.25 mm ID with 0.25 µm film thickness DB-1 capillary column.

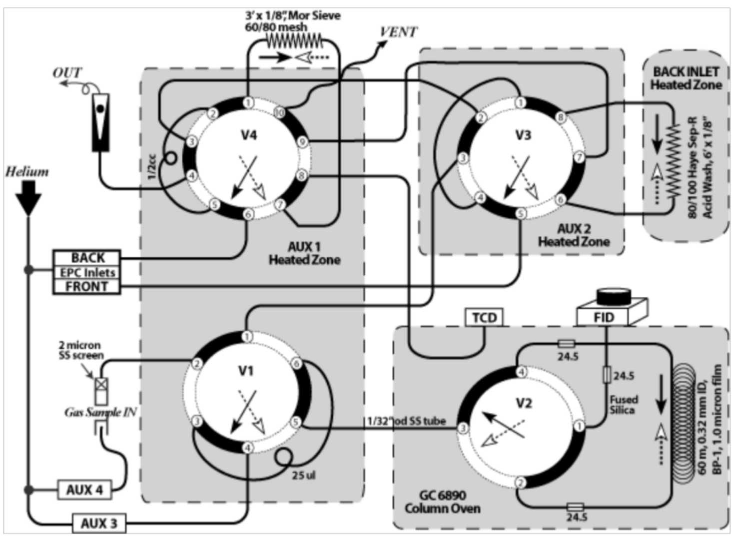

Figure 5. Schematic of a Sample Gas Line in the GC-NGA.

Instrument Installation & Setup

Agilent 6890 GC Specifications

Maximum temperature	450°C
Temperature program	Up to 6 ramps

Maximum run time	999.99 min
Temperature ramp rate	0°–120°C/min
Dimensions	50 cm × 58.5 cm × 50 cm
Weight	112 lb (50 kg)
Heat dissipation	7681 BTU/hr max
Operating temperature	20°–27°C
Operating humidity	50%–60%

Gases

The GC requires that hydrogen and air are connected to the marked fittings on the back of the instrument. The type of makeup gas must be identified in the method file.

- Air, compressed (Zero-Air +): >50 psi
- Helium, compressed (99.9995% +): >50 psi
- Hydrogen, compressed (99.9995% +): >50 psi

GC3 Method: GC390FR.M

h7.Injector

- Injection source: manual
- Injection location: front

h7.Oven

- Initial temperature: 90°C
- Maximum temp: 250°C
- Initial time: 0.50 min
- Equilibration time: 1.00 min
- Port temp: 100°C
- Post time: 0.00 min
- Run time: 8.60 min (run time will automatically be changed based on ramp setting)
- Temperature program:

1	30.00	100	0.00
2	15.00	110	4.50
3	50.00	150	1.80
4	0.00		

h7.Front Inlet

- Initial temp: 120°C
- Flow: 30.6 mL/min
- Gas type: helium

h7.Column 1

- Packed column (model #: Restek PC3970)
- HaySep "R" 80/100, 2.00 mm ID x 1/8 inch OD, 6 ft
- Max temperature: 225°C
- Mode: ramped flow
- Initial flow: 30.0 mL/min
- Initial time: 2.70 min
- Post flow: 0.0 mL/min
- Inlet: front
- Outlet: front detector
- Outlet pressure: ambient
- Temperature program:

1	100.00	40	3.00
2	10.00	30	0.30

3	100.00	60	0.00

h7.Column 2 (not installed)
h7.Front detector (FID)

- Temperature: 250°C (always on)
- Hydrogen (H_2) flow: 40.0 mL/min (on if FID temperature is >150°C, auto control)
- Air flow 400.0 mL/min (on if FID temperature is >150 °C, auto control)
- Mode*: constant makeup flow
- Makeup flow*: 25.0 mL/min
- Makeup gas type*: nitrogen (*Makeup flow: none; makeup gas: none)
- Flame: on (auto on when FID temperature reaches 150°C)
- Electrometer: on
- Lit offset: 1.0

h7.Back detector: no detector
h7.Signal 1

- Data rate: 5 Hz
- Type: front detector
- Save data: On
- Zero: 0.0
- Range: 0
- Fast peaks: off
- Attenuation: 0

h7.Signal 2

- Data rate: 20 Hz
- Type: front detector
- Save data: Off
- Zero: 0.0
- Range: 0
- Fast peaks: off
- Attenuation: 0

h7.Column comp 1 & 2

- Derive from front detector

h7.Auxillary pressure 3, 4, & 5

- Gas type: helium
- Initial pressure: 0.00 psi

h7.Valves

- Valve 5 switching off

h7.Post run time: 0.00 min
h7.Time table for valve control

- 0.01 min—Valve 5 on; 6.00 min—Valve 5 off

GC3 Sample Flow Schematics

Standby Mode

Green line shows helium carrier gas flow when GC3 is in standby mode.

- Inlet—injector port—V6—V7—V9—V8—column—V1—V10—FID

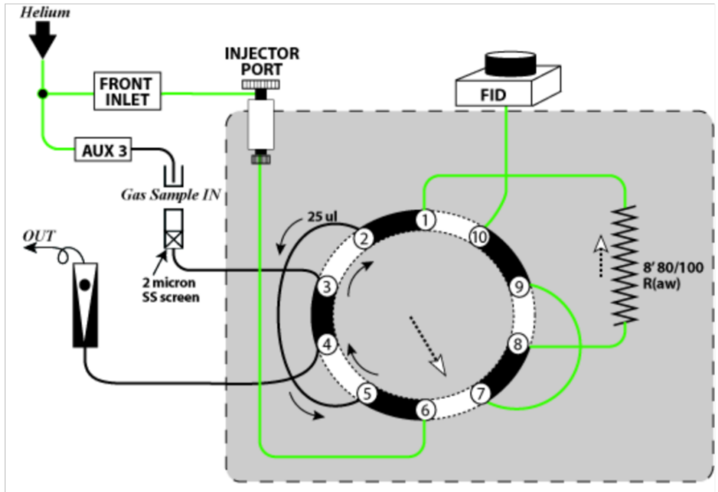

Figure 6. GC3 in Standby Mode.

Injection Mode

He carrier (green) and sample (red) gas flows during injection mode. Sample gas fills the 25 µL sample loop.

- Sample gas: injector—V3—V2—V5—V4—vent
- Carrier gas: Inlet—injector port—V6—V7—V9—V8—column—V1—V10—FID

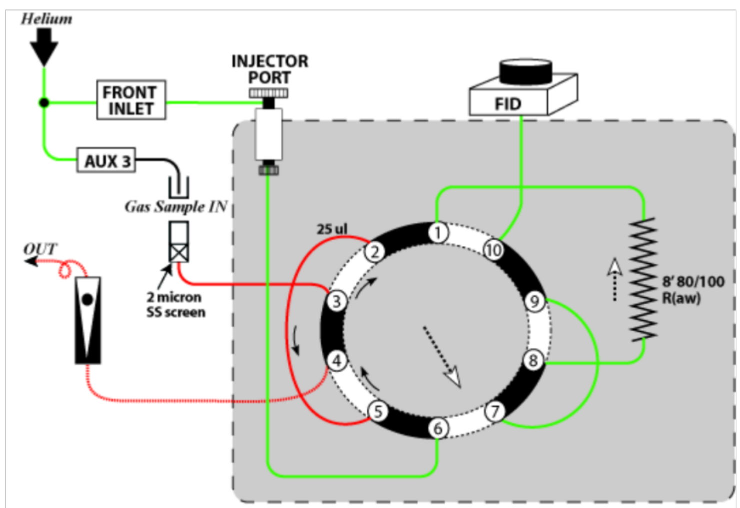

Figure 7. GC3 in Injection Mode.

Run Mode

He carrier (green) and sample (red) gas flows during the sample run. When the valve is turned, helium coming from the inlet pushes the sample gas trapped in the sample loop.

- Sample gas: column—FID
- Carrier gas: V5—V2—V1—column—V8—V7—V9—V10—FID

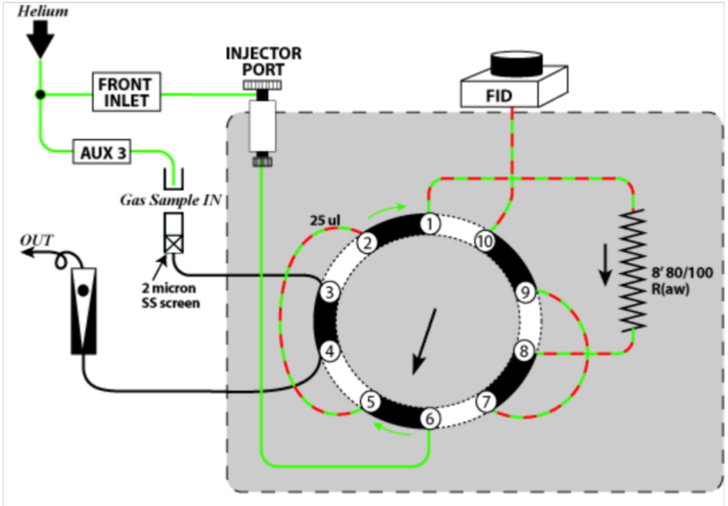

Figure 8. GC3 in Run Mode.

NGA Method: NGA_CS.M

Injector

- Injection source: manual
- Injection location: front

Oven

- Initial temp: 50°C
- Maximum temp: 300°C
- Initial time: 2.00 min
- Equilibration time: 1.00 min
- Port temp: 50°C
- Post time: 0.00 min
- Run time: 14.80 min (run time will be changed based on ramp setting)
- Temperature program:

Ramp	Rate (°C/min)	Final Temperature (°C)	Final Time (s)
1	8.00	70	0.00
2	25.00	200	5.10
3	0.00	NA	NA

Front Inlet

- Flow: 21.0 mL/min
- Gas type: helium

Back inlet

- Initial temp: 80°C
- Initial time: 0.00 min
- Cryo: off
- Cryo type: compressed air
- Pressure: 20.75 psi (On)
- Gas type: helium
- Temperature program:

Ramp	Rate (°C/min)	Final Temperature (°C)	Final Time (s)
1	0.00	NA	NA

Column 1: Not installed

Column 2

- Capillary column (model #: Agilent 122-1062)
- Agilent DB-1 (dimethylpolysiloxane) 60.0 m x 0.25 mm diameter x 0.25 µm film thickness
- Max temperature: 325°C

- Mode: constant flow, 2.0 mL/min
- Inlet: back inlet
- Outlet: back detector
- Outlet pressure: ambient

Front detector (FID)

- Temperature: 250°C (always on)
- Hydrogen (H_2) flow: 40.0 mL/min
- Air flow 400.0 mL/min
- Mode*: constant makeup flow
- Makeup flow*: 50.0 mL/min
- Makeup gas type*: helium
- Flame: On
- Electrometer: on
- Lit offset: 2.0

Back detector (TCD)

- Temperature: 200°C (always on)
- Reference flow: 45.0 mL/min
- Mode: constant makeup flow
- Makeup flow: 3.0 mL/min
- Makeup gas type: helium
- Filament: on
- Negative polarity: off

Signal 1

- Data rate: 5 Hz
- Type: back detector
- Save data: on
- Zero: 0.0
- Range: 0
- Fast peaks: off
- Attenuation: 0

Signal 2

- Data rate: 5 Hz
- Type: front detector
- Save data: on
- Zero: 0.0
- Range: 0
- Fast peaks: off
- Attenuation: 0

Column comp 1

- Derive from front detector

Column comp 2

- Derive from back detector

Thermal AUX 1 & 2

- Use: valve box heater
- Initial temp: 110°C
- Initial time: 0.00 min

Ramp	Rate (°C/min)	Final Temperature (°C)	Final Time (s)
1	0.00	NA	NA

AUX pressure 3

- Gas type: helium
- Initial time: 4.50 min

Ramp	Rate (°C/min)	Final Temperature (°C)	Final Time (s)
1	30.00	22.20	0.00
2	1.10	27.50	0.00
3	0.00	NA	NA

Aux pressure 4 & 5

- Gas type: helium
- Initial pressure: 0.00 psi

Valves (1 to 4, initial): Switching off

- Valve control time program

Time (min)	Valve control
0.00	valve 1: off
	valve 2: off
	valve 3: off
	valve 4: off
0.01	valve 4: on
0.07	valve 1: on
	valve 2: on
1.80	valve 3: on
1.83	valve 4: off
8.50	valve 3: off
9.10	valve 1: off
	valve 2: off

NGA Sample Flow Schematics

Standby Mode

He gas flow for standby mode (green lines).

- Line 1: Aux-3—V1-4—V2-5—V2-3—capillary column—V2-4—V2-1—FID
- Line 2: Aux-4—sample inlet—V1-2—V1-3—V1-6—V1-1—V3-3—V3-4—V3-1—V4-3—V4-2—V4-5—V4-4—Vent
- Line 3: Front inlet—V3-5—V3-6—HaySep R column—V3-8—V3-7—V4-9—V4-8—TCD
- Line 4: Back inlet—V4-6—V4-7—MolSieve column—V4-1—V4-10—Vent

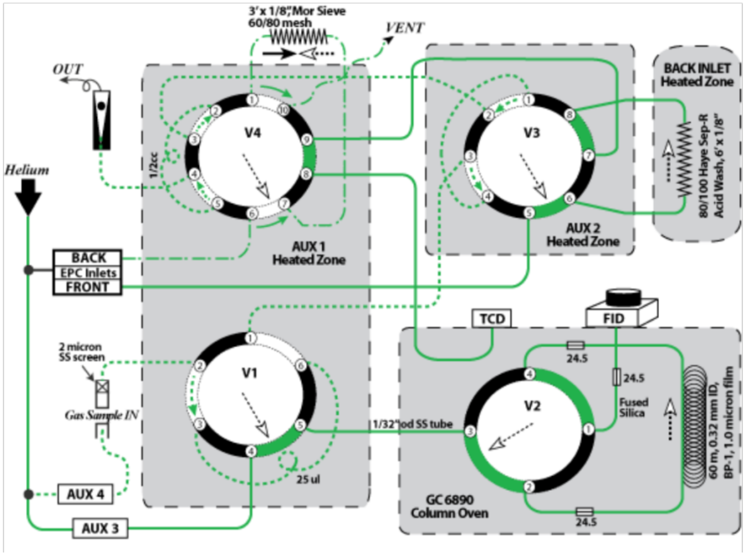

Figure 9. NGA in Standby Mode.

Injection mode

He carrier gas (green line) and sample gas (red line) flows in the NGA in injection mode. Sample gas fills the sample loops connected to V1 (25 µL), V3 (1 cm^3), and V4 (0.5 cm^3). He flushes the separation columns.
He gas flow (green):

- Line 1: Aux-3—V1-4—V1-5—V2-3—V2-2—capillary column—V2-4—V2-1—FID
- Line 3: Front inlet—V3-5—V3-6—HaySep R column—V3-8—V3-7—V4-9—V4-8—TCD
- Line 4: Back inlet—V4-6—V4-7—MolSieve column—V4-1—V4-10—Vent

Sample gas flow (purge; red):

- Sample inlet—V1-2—V1-3—V1-6—V1-1—V3-3—V3-4—V3-1—V3-2—V4-3—V4-2—V4-5—V4-4—Vent

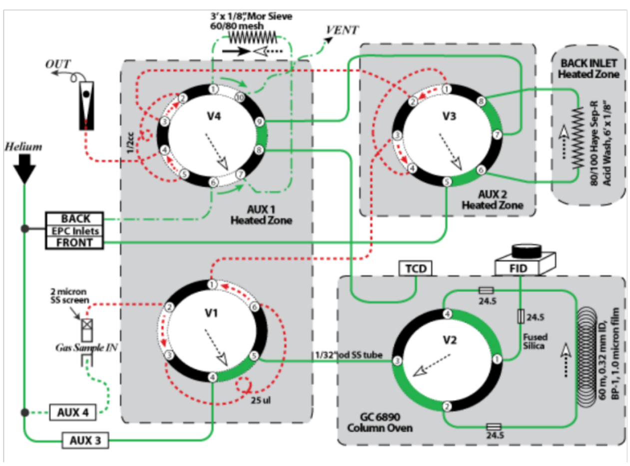

Figure 10. NGA in Injection Mode.

Run Mode at 0.01 min (open Valve V4)

He (green) and sample gas (red) flows in the NGA 0.01 min after start of run. Sample gas remains in the sample loop connected to V1 (25 µL) and V3 (1 cm^3). After V4 opens, He returning from the back inlet pushes the sample gas out of the sample loop and into the molecular sieve column. Separated elements are detected by TCD.
He gas flow:

- Line 1: Aux-3—V1-4—V1-5—V2-3—V2-2—capillary column—V2-4—V2-1—FID
- Line 2: Aux-4—V1-2
- Line 3: Front inlet—V3-5—V3-6—HayeSep R column—V3-8—V3-7—V4-9—V4-10—Vent
- Line 4: Back inlet—V4-6—V4-5

Sample gas flow (purge):

- V1-2—V1-3—V1-6—V1-1—V3-3—V3-4—V3-1—V3-2—V4-3—V4-4—out

Sample gas flow with He:

- V4-5—V4-2—V4-1—MolSieve column—V4-7—V4-8—TCD

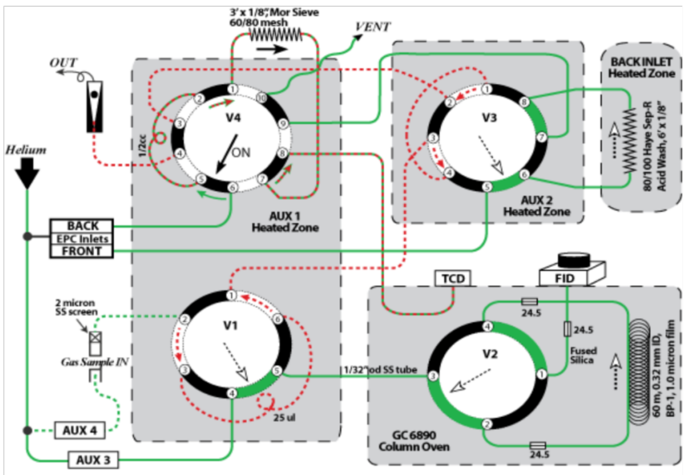

Figure 11. NGA in Run Mode: 0.01 min after starting run.

Run Mode at 0.07 min (open Valves V1 and V2)

He (green) and sample gas (red) flows in the NGA 0.07–1.79 min after start of run. Sample gas remains in the sample loop connected to V3 (1 cm^3). After V1 and V2 open, He from Aux-3 pushes the sample gas out of the sample loop connected to V1 (25 µL) and into the capillary column (60 m) through V2, where it passes into the FID.
He gas flow:

- Line 1: Aux-3—V1-4
- Line 2: Aux-4—V1-2
- Line 3: Front inlet—V3-5—V3-6—HaySep R column—V3-8—V3-7—V4-9—V4-10—vent
- Line 4: Back inlet—V4-6—V4-5—V4-2—V4-1—MolSieve column—V4-7—V4-8

Sample gas flow (purge):

- V3-4—V3-1—V3-2—V4-3—V4-4—out

Sample gas flow with He:

- V4-8—TCD
- V1-3—V1-6—V1-5—V2-3—V2-4—capillary column—V2-2—V2-1—FID
- V1-1—V3-3

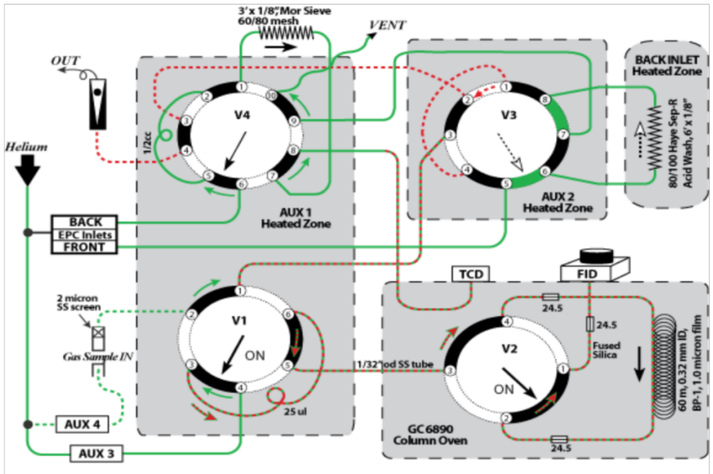
Figure 12. NGA in Run Mode: 0.07–1.79 min after starting run.

Run Mode at 1.80 min (open Valve V3)

He (green) and sample gas (red) flows in the NGA 1.80–1.82 min after start of run. After V3 opens, He from the front inlet pushes the sample gas out of the 1 cm^3 sample loop into the HaySep column.
He gas flow:

- Line 1: Aux-3—V1-4—V1-3—V1-6—V1-5—V2-3—V2-4
- Line 2: Aux-4—V1-2—V1-1—V3-3—V3-2—V4-3—V4-4—out
- Line 3: Front inlet—V3-5—V3-4
- Line 4: Back inlet—V4-6—V4-5—V4-2—V4-1—MolSieve column—V4-7—V4-8—TCD

Sample gas flow with He:

- Capillary column—V2-2—V2-1—FID
- B3-4—V3-1—V3-8—HaySep R column—V3-6—V3-7

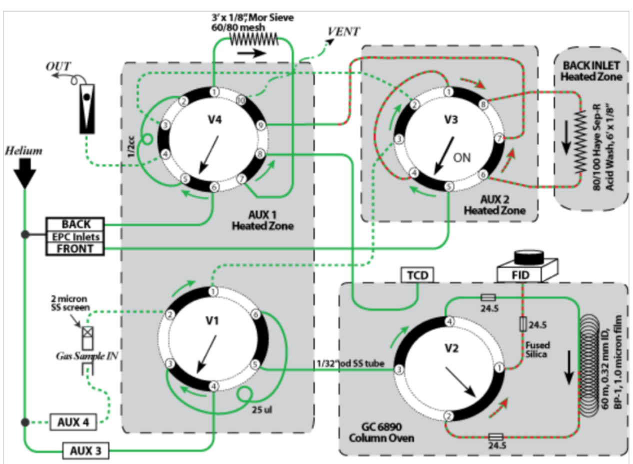
Figure 13. NGA in Run Mode: 1.80–1.82 min after starting run.

Run Mode at 1.83 min (close Valve V4)

He (green) and sample gas (red) flows in the NGA 1.83–8.49 min after start of run. After V4 closes, He from the back inlet flushes the molecular sieve column (backflush). Gas samples separated by the HaySep column enter the TCD through V4.
Helium gas flow:

- Line 1: Aux-3—V1-4—V1-3—V1-6—V1-5—V2-3—V2-4—capillary column—V2-2—V2-1—FID
- Line 2: Aux-4—V1-2—V1-1—V3-3—V3-2—V4-3—V4-2—V4-5—V4-4—out
- Line 3: Front inlet—V3-5—V3-4—V3-1—V3-8

Sample gas flow with He:

- HaySep R column—V3-6—V3-7—V4-9—V4-8—TCD

Backflush:

- Line 4: Back inlet—V4-6—V4-7—MolSieve column—V4-1—V4-10—vent

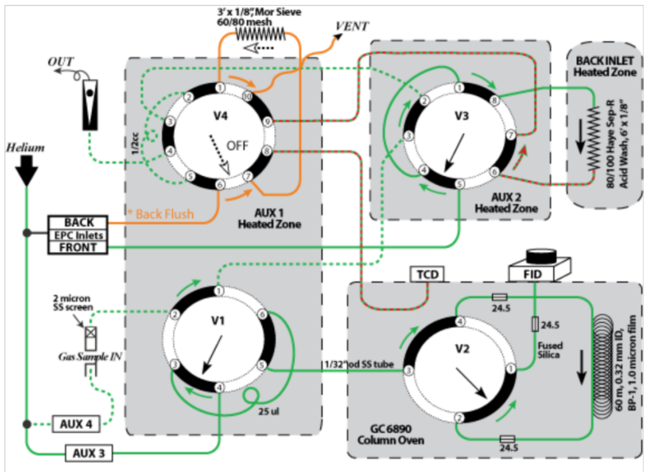

Figure 14. NGA in Run Mode: 1.83–8.49 min after starting run.

Run Mode at 8.50 min (close Valve V3)

He gas (green) and sample gas (red) flows in the NGA 8.50–9.09 min after start of run. After V3 closes, He from the front inlet flushes the HaySep column and the line leading to the TCD (backflush).
He gas flow:

- Line 1: Aux-3—V1-4—V1-3—V1-6—V1-5—V2-3—V2-4—capillary column—V2-2—V2-1—FID
- Line 2: Aux-4—V1-2—V1-1—V3-3—V3-4—V3-1—V3-2—V4-3—V4-2—V4-5—V4-4—out
- Line 3: Back inlet—V4-6—V4-7—MolSieve column—V4-1—V4-10—vent

Backflush:

- Line 3: Front inlet—V3-5—V3-6—HaySep R column—V3-8—V3-7—V4-9—V4-8—TCD

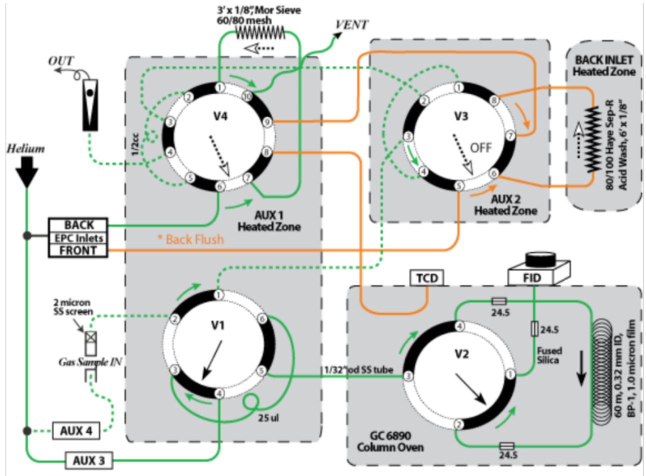

Figure 15. NGA in Run Mode: 8.50–9.09 min after starting run.

Run Mode at 10.0 min (close Valves V1 and V2)

He (green) and sample gas (red) flows in the NGA 9.09–10.0 min after start of run. After V1 and V2 close, He flow returns to standby mode.
He gas flow:

- Line 1: Aux-3—V1-4—V1-5—V2-3—V2-2—capillary column—V2-4—V2-1—FID
- Line 2: Aux-4—V1-2—V1-3—V1-6—V1-1—V3-3—V3-4—V3-1—V3-2—V4-3—V4-2—V4-5—V4-4—out
- Line 3: Front inlet—V3-5—V3-6—HaySep R column—V3-8—V3-7—V4-9—V4-8—TCD
- Line 4: Back inlet—V4-6—V4-7—MolSieve column—V4-1—V4-10—vent

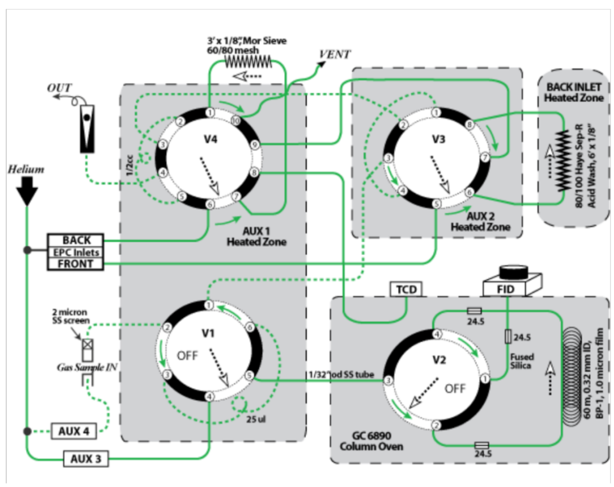

Figure 16. NGA in Run Mode: 9.09–10.0 min after starting run.

GC3 & NGA Startup

Overview

The chromatography application *ChemStation* controls GC data acquisition and processing. It can be run either online or offline. Offline mode can be run without communication with the GCs, so it is useful for reintegrating or reprocessing chromatograms. Online mode requires communication with the GC.

Starting up GC3/GC-NGA and ChemStation

	Start *ChemStation* software and load the appropriate method for the analysis (see ***Starting up ChemStation and GC Ovens***).
	Condition the GC (see ***Conditioning the GC***). If the GC has been turned off for longer than a week, then bake the column for 8 hr with gas flowing (manually set the oven temperature to 175°C for GC3 or 275°C for NGA).
	Run a calibration curve (see *GC3/NGA User Guide*).
	Run a calibration verification standard (see *GC3/NGA User Guide*)
	Run a test sample (see *GC3/NGA User Guide*)

Starting up ChemStation and GC Ovens

	Turn on the GC. **WARNING:** Before turning on the GC, make sure the gas lines are open. The 6890 GC performs a comprehensive self-evaluation and shows real-time diagnostics on the screen. *Warning*, *Fault*, or *Bad Main Board & Fatal Error* messages require troubleshooting before moving to the next step (see ***Maintenance & Troubleshooting (HP6890GC)***).
	Turn on the PC.
	Click the **GC3 Online** or **NGA Online** icon to start *ChemStation*. The *Method and Run Control* window opens. At startup, *ChemStation* uses the method last used (shown on the main screen). In addition, the GC LCD shows the loaded settings from *ChemStation*. Settings changed on the GC using the GC control panel are also made to *ChemStation*, and parameter changes entered into *ChemStation* are made to the GC. *ChemStation* will prompt to save changes.
	To load a different method in *Chemstation*: • Click **Method > Load Method**, select the method from the list, and press **OK** or • Click the **Method** tab on the left side of the window and select a method to load
	The system automatically loads the new method selected in *ChemStation* to the appropriate GC. Oven and detector temperatures may increase immediately after a new method is loaded, and the FID will ignite when the detector temperature reaches 150°C. Sometimes, the GC beeps because the FID flame is out, especially after a long idle period. See ***Maintenance & Troubleshooting (HP6890GC)***.

GC3 Methods

Method Title	Definition
GC390FR.M	Standard operation method since November 2007.
def_gc.m	Default for ChemStation. This method must be kept in the Method folder permanently.
cbt.m	Default method for training.
estd_ex.m	Default method for training.
istd_ex.m	Method created onshore to make conditions for GC3.

NGA Methods

Method title	Definition
NGA_CS	Standard operation method since November 2007.
NGA_308	Method for IODP phase 1, Expedition 308.
def_gc.m	Default for ChemStation. This method must be kept in the Method folder permanently.
cbt.m	Default method for training.
estd_ex.m	Default method for training.
istd_ex.m	Method created onshore to make conditions for GC3.

Conditioning the GC

	To condition the GCs, in the Main menu click **RunControl > Sample Info**.

	Fill in the following fields: • Operator name: your last name • Sample name: "cond" for conditioning • Comment: "Conditioning" Click **OK** to close window and save information.
	Prepare laboratory air (5000 µL) and inject it into the GC when the *ChemStation* software shows *Ready*.
	Press the **Start** button on the GC control panel to start the run.
	Confirm the chromatogram on the screen shows no peaks. If peaks are present, the system contamination must be found (injector, detector, sample loop, etc.).

LIMS Data Upload

Overview

Data is uploaded from the GC3 and NGA in one of two modes:

- **Automatic mode:** files are uploaded as soon as the run completes
- **Manual mode:** the user selects **upload** from the menu

Automatic Upload

Data is uploaded from the GC3 and NGA via a multi-step process:

1. When the run is complete, a macro (**GC3_LIMS.MAC** or **NGA_LIMS.MAC**) is automatically called, as configured in the method file. The macro copies information from the method directory to **C:\LIMS\NGA\data** or **C:\LIMS\GC3\data**.
2. An in-house program called MegaUploadaTron (**MUT)** monitors the data folder locations and when a file is copied in initiates the next steps of the upload process.

- The file is opened and read, and data points are uploaded to LIMS
- The data files are compressed (zipped) and uploaded as well
- LIMS analysis codes are **GC3**, **NGAFID**, and **NGATCD**

1. After the upload to LIMS is complete, **MUT** moves the data files to an archive directory at **C:\DATA\GC3\archive** or **C:\DATA\NGA\archive**.
2. If an upload error occurs, the files are not archived and **MUT** will report the error in the main window (only).

Manual Upload

If **MUT** is not running when the GC finishes, files will queue in the data directory for manual upload.

Maintenance & Troubleshooting (HP6890GC)

Overview

Use the **Status** and **Info** keys on the GC keypad as a first check when something goes wrong.

Leak Checking

When checking for leaks, check both parts of the system:

- **External leaks:** gas cylinders, gas purifiers/traps, regulator fittings, supply shutoff valves, GC supply fittings.
- **GC leaks:** inlets, purge vents; column connections to inlets, detectors, valves, splitters, adapters, and unions.

For safe leak-checking and flow measurement:

- Purge flowmeters with inert gas after measuring a flammable gas (such as hydrogen).
- Measure gases individually.
- Turn off detectors while measuring gas flows.

Column Size and Carrier Gas Flow Rate

Column type	Column ID	Carrier gas flow rate (mL /min)	
		Hydrogen	Helium
Packed	1/8 inch		30
	1/4 inch		60
Capillary	50 µm	0.5	0.4
	100 µm	1.0	0.8
	200 µm	2.0	1.6
	250 µm	2.5	2.0
	320 µm	3.2	2.6
	530 µm	5.3	4.2
These flow rates at normal temperature and pressure (25°C and 1 atm) are recommended for all column temperatures. For capillary columns, flow rates are proportional to column diameter and are 20% lower for helium than for hydrogen.			

6890GC Messages

Message	Description/Cause	Troubleshooting
Not Ready	"Not Ready" LED lights (a component of the GC is not ready to begin a run)	• Press **Status** key for explanation • Check for leaks in gas lines • Check gas supply delivery pressure • Check that oven, inlet, and detector temperatures are not too far apart
Method Mismatch	A loaded method contains parameters that do not match the GC's current configuration • If the parameter is set from the keyboard, method will overwrite current parameter and display a message that the parameter has been replaced • If the parameter depends on hardware, the method will be ignored and the current setpoints will remain. A message will indicate the method parameter is being ignored.	Follow *ChemStation* instructions After method update, open **Method parameter** to check new setting; edit method if needed
Warning	A serious problem exists. • GC will not stop or prevent a run • GC emits 1 beep and displays warning message • Warning appears at run start • Warning is not recorded in run log	Press **Status** key to view explanation
Shutdown	Shutdown occurs/numbered error message is displayed	Pop-up message briefly explains the nature of the problem
Faults	Hardware problem requires user intervention • GC emits no beep or a single beep • Ready LED does not light • Error message appears	Press **Status** button for more information
Bad Main Board & Fatal Errors	Main board has malfunctioned; must be replaced	See ***Bad Mainboard/***Fatal Error Messages

Common Chromatography Problems

Problem	Cause	Troubleshooting
No peaks on chromatogram	Acquisition aborted	Confirm the method is correct
	Bad cable or connection	Check cables between GC and PC, detectors and GC
	Leak in sample line	• Purge test injectors and detectors • Check sample loop and columns for leaks
	FID flame out	See FID flame out/will not light
	TCD filament break	Measure TCD filament resistance (~10 ohm)
	Column break	Check column installation
Retention times inconsistent	Column flow has changed	• Check for leaks at inlet, liner, column connections • Check carrier gas supply pressure • Check column installation • Check method
	FID jet contaminated	Remove jet and clean
	Injector port temperature wrong	Check method
	Oven temp program changed	Check method
	Column overload	Inject less sample
Extra peaks on chromatogram	Contamination in system	• Clean sample loops and injector port with solvent • Check gas trap indicators and expiration dates • Verify carrier/detector gas purity • Check gas supply tubing and fittings • Click **Start** on the control panel of GC without injection to confirm column contamination
	Contaminated syringe	• Clean syringe and vials with solvent • Click **Start** on the control panel of GC without injection, then inject laboratory air
Noisy baseline/random spiking	Leaks	• Check for leaks at column fittings
	Contamination	• Verify purity of carrier/detector gases • Inspect the jet for contamination • Verify column has been conditioned
	Electrical problem	• Column is installed too high in detector • Electronic interference in laboratory

Common Hardware Problems

Problem	Cause	Troubleshooting
FID flame out/will not light	Detector gas flow incorrect	• Check that gas lines are open • Check the gas system for leaks • Check air/hydrogen flow rates/mix • Check column flow rate • Check column/detector fitting for leaks
	FID temperature too low	Wait 15–20 min for conditioning Press **Front Detector** on the GC control panel and light the flame manually

FID flame out/will not light	Bad igniter	Remove heater/sensor assembly from the FID and measure resistance of heater and sensor. Replace ignitor if resistance is too high or too low: • Heater resistance = ~22 ohm • Sensor resistance = ~109 ohm
	Jet dirty or partially plugged	Remove jet and clean
	Flame will not stay lit	Check dessicant state in the hydrogen generator
Oven cannot attain or maintain setpoint temperature	• Oven flaps • Oven fan • Oven heater • Oven temperature sensor	Contact service representative

Bad Mainboard/Fatal Error Messages

Message	**Comment**
Main FPGA Failure	Contact vendor representative
Static RAM Failure	
Boot ROM Checksum	
ROM #2 or #3 Checksum	EEPROM 2 or 3 malfunction
Incorrect ROM #2 or #3	EEPROM 2 or 3 installed in wrong position
ROM #2 or #3 wrong version	EEPROM 2 or 3 does not match other EEPROMs
DMA FPGA Failure	Contact vendor representative
DRAM Failure	
Exception Vector	
Bus Error	
Address Error	
Illegal Instruction	
Divide by Zero	
No 512Hz Interrupt	

Shutdown Messages

Message number	Message	Explanation/Troubleshooting
1	Oven shut off	• Oven flap malfunction • Thermal leaks (missing insulation) • Excessive oven load • Heater electronics malfunction
2	Oven cryo shutdown	Timeout

3, 5	Inlet pressure shutdown	Inlet does not reach setpoint
4, 6	Inlet flow shutdown	
5, 8	Front detector fuel gas shutdown	Gas unable to reach/maintain setpoint in time allowed
6, 9	Front detector air/ref shutdown	
7, 10	Front detector makeup shutdown	
8, 9, 10	Pres aux shutdown	Pneumatics aux module cannot maintain setpoint
9	Multiposition valve not switching	• Valve disconnected • Valve stuck • Switching time too short
10	Can't reach setpoint of multiposition valve	• Valve position incorrect • Invert BCD setpoint incorrect
11, 12	Inlet cryo shutdown	Timeout
12, 14	Aux cryo shutdown	
13, 14	Inlet heating too slowly	• Temperature sensor failure • Zone heater defective

Warning Messages

Message number	Message	Explanation/Troubleshooting
100	Oven sensor missing	
101, 102	Invalid heater power	Invalid heater power for front detector, inlet, or aux 1 or 2
103, 104	Signal buffer full	• PC network down • PC cable disconnected • PC turned off • PC entered power saver mode • PC data collection stopped • GC hardware problem
105	Analog out data loss	Possible data loss
106	Signal data loss	
107, 108	Detector config changed	Correct method to match hardware
109, 110	Inlet config changed	
111, 112	Column config changed	
113, 114, 115	Aux method changed	
116	Log overflow	Capacity = 50 entries
117, 118	Inlet calibration deleted	Returned to default calibration
119, 120	Detector calibration deleted	
121	Aux calib deleted	
122	Comm data overrun	Possible data loss
123	Comm data error	
124	Comm abnormal break	Check connection
125	Sampler data overrun	Possible data loss
126	Sampler data error	

127	Sampler abnormal break	Check connection
128, 129	Inlet flow calibration fail	Contact vendor representative
130, 131	Aux cryo disabled	Reconfigure cryo
132–137	Sampling end problem	Setpoint conflicts with sampling end time parameter

Fault Messages

Message number	Message	Comments
200, 201	Faulty pneumatics board	
202	Hydrogen shutdown	• Check gas supply pressure • Check for leaks • Check for broken column • Check for stuck valve stuck
203–207	Signal DSP fault	
208–211	Out signal path test failed	
212, 213	Detector electrometer out of spec	
214, 215	Detector flame out	• Check hydrogen/air flow rates • Check for leaks at detector/column fitting • Check jet
216–219	TCD filament open or shorted	• Check wire connections • Check cell temperature sensor • Replace TCD cell
220, 221	Thermal shutdown	• Check electrical supply to GC • Check zone control electronics • Possible shorted temperature sensor • Possible shorted heater
222–224	Oven temperature fault	
225–228	Detector temperature fault	
229–232	Inlet temperature fault	
233–236	Aux temperature fault	
237, 238	Line interrupt fault	
239, 240	Mux ADC thermal shutdown	
241	Invalid line sense	
242–244	Pneu aux module invalid constants	
245–249	Obsolete EEPROM	
250–254	Wrong module	
255–258	Invalid module	
259, 260	Detector module/board mismatch	
261	MIO board defective	
262, 264	RS232 defective	
263	GPIB defective	

265–269	Invalid pids	
270–274	Invalid checksum	
275–279	Invalid constants from factory calibration	
280–284	I/O failure	
285, 286	Detector offset adjustment failed	