Agilent 8355 S Sulfur and 8255 S Nitrogen Chemiluminescence Detectors

User Manual

Agilent Technologies

Notices

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CAUTION

A **CAUTION** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

WARNING

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

Contents

1
Getting Started

This chapter introduces the Agilent 8355 S Sulfur Chemiluminescence Detector (SCD) and the Agilent 8255 S Nitrogen Chemiluminescence Detector (NCD), and provides details about where to find helpful information and tools, such as GC manuals, flow calculators, and so forth.

Manuals, Information, Tools and Where to Find Them

This manual describes how to operate the Agilent 8355 S Sulfur Chemiluminescence Detector (SCD) and the Agilent 8255 S Nitrogen Chemiluminescence Detector (NCD). This manual also provides operating recommendations, maintenance procedures, and troubleshooting. For installation instructions, see “Overview of Installation and First Startup” on page 14. To prepare the installation site for a new SCD or NCD, see the Agilent *Site Preparation Guide*.

In addition, Agilent provides other manuals, familiarization information, and help systems for self-paced learning about it’s current line of GCs. You will need to reference this general GC information for installation and operation of the detector. The sections below describe this information and where to find it.

Safety Information

Before proceeding, read the important safety and regulatory information found in the *Agilent Safety and Regulatory Information manual for the 8355, 8355 S and 8255, 8255 S Chemiluminescence Detectors*.

Online help

In addition to hardware manuals, your GC data system also includes an extensive online help system with detailed information, common tasks, and video tutorials on using the software.

Education Opportunities

Agilent has designed customer courses to help you learn how to use your GC to maximize your productivity while learning about all of the great features of your new system:

For course details and education opportunities, visit **http://www.agilent.com/chem/education**, or call your local Agilent sales representative.

Overview of the 8355 S SCD and 8255 S NCD

Figure 1 through Figure 5 show the controls, parts, and components of the 8355 S SCD and 8255 S NCD used or accessed during installation, operation, and maintenance.

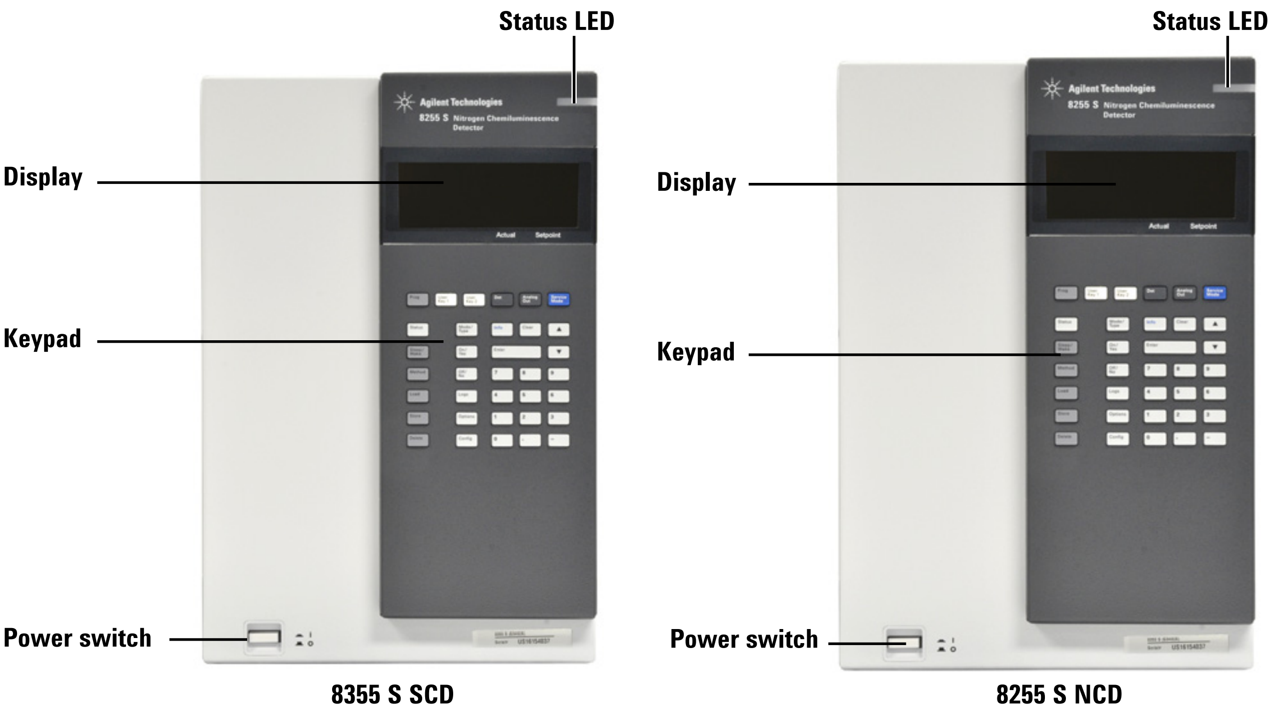


Figure 1 Front view, Standalone detectors (SCD and NCD)

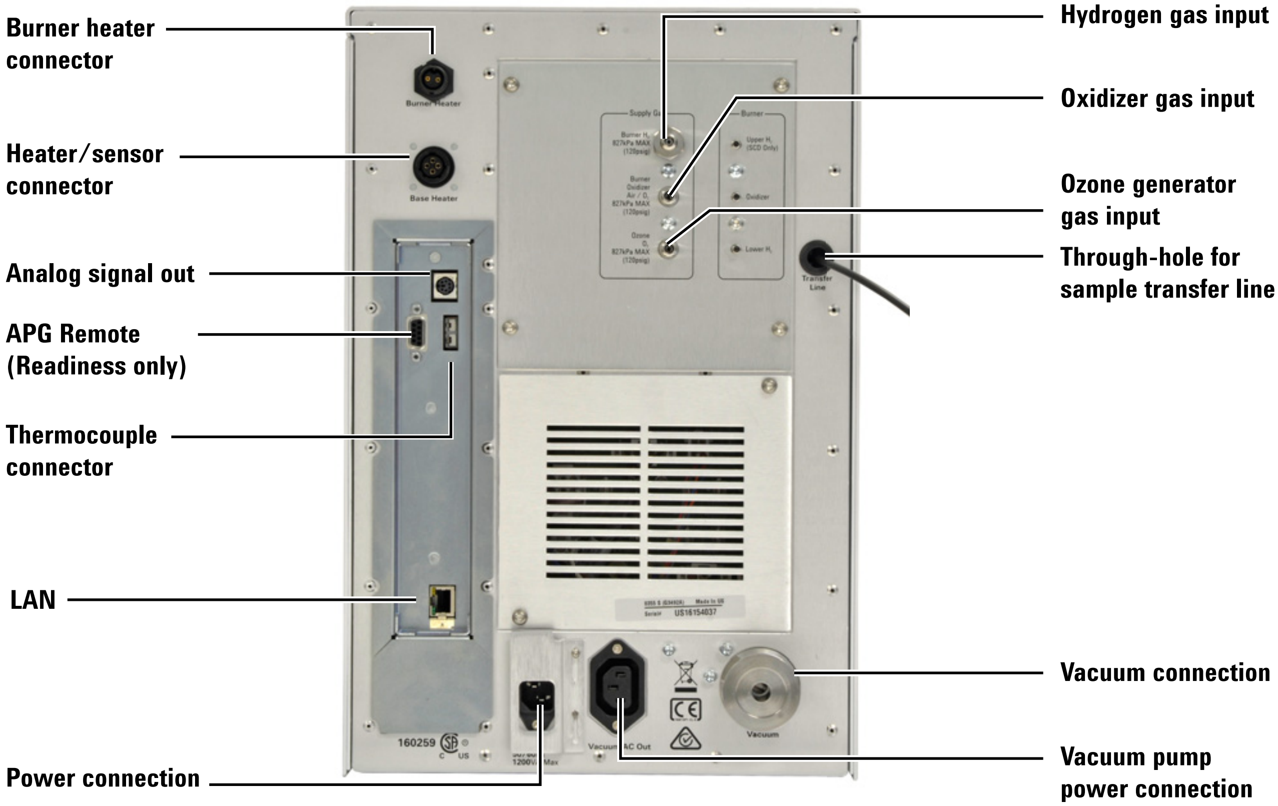


Figure 2 Standalone detector back view

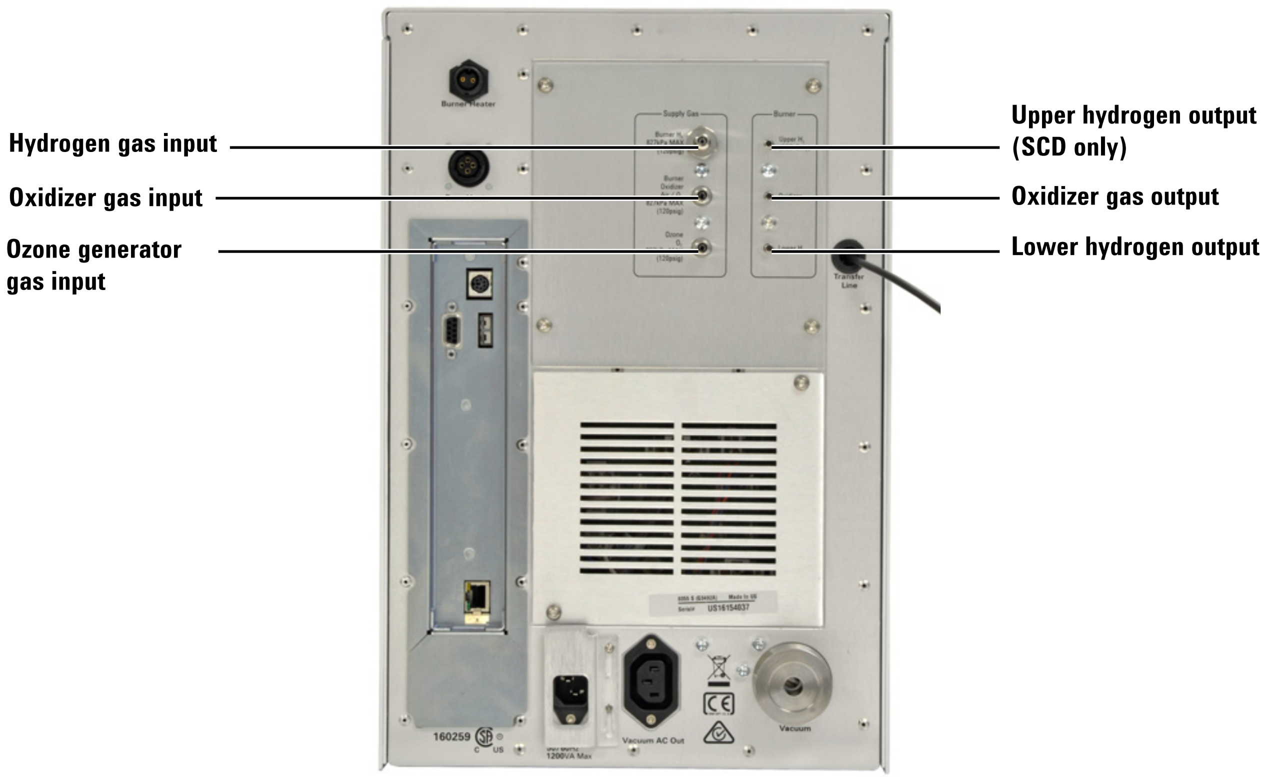


Figure 3 Standalone detector gas connections

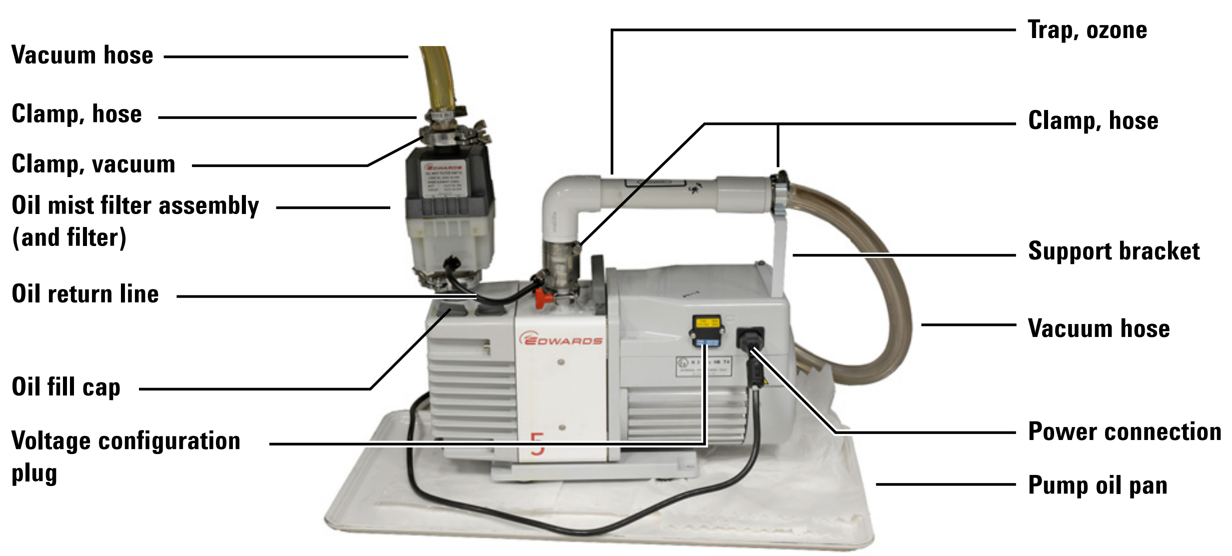


Figure 4 RV5 Vacuum pump

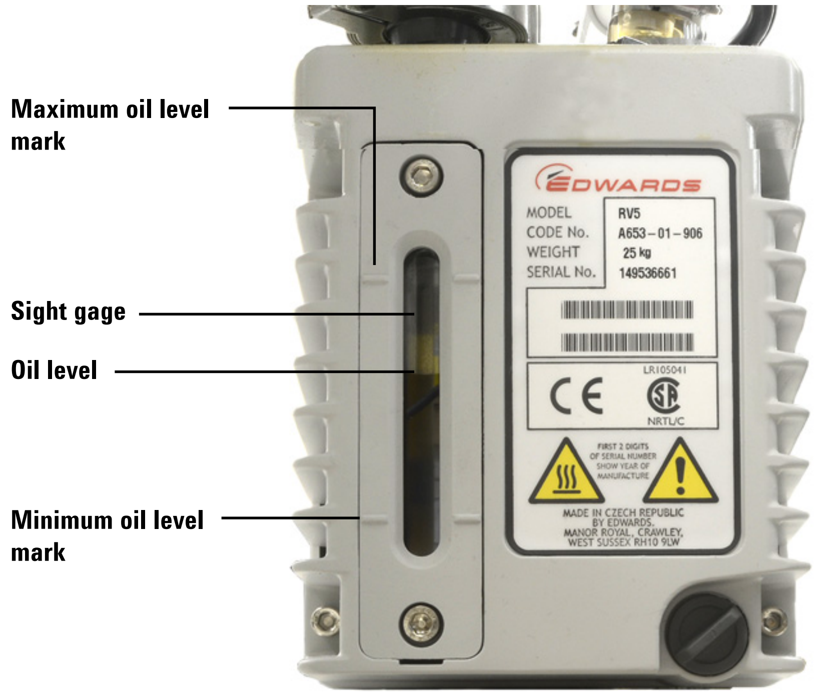


Figure 5 RV5 Vacuum pump oil sight gage

Overview of Installation and First Startup

Below is an overview of the installation process. Installation and service of the detector should only be performed by Agilent-trained service personnel.

1. Place the detector on the bench. Remove protective caps.
2. Prepare the detector mounting location.
3. Unpack the vacuum pump. Remove plugs. Install oil coalescing filter and ballast.
4. Install the vacuum pump.
5. Prepare the detector.
6. Cool the GC oven, inlet, and other heated zones to a safe handling temperature (< 40 °C). Turn off the GC.
7. Verify the power configuration.
8. Install the burner assembly.
9. Connect the supply gases.
10. Connect the detector gases.
11. Connect the detector cables and wires.
12. Connect cables to the GC and detector.
13. Connect to power.
14. Install the column.
15. Turn the power on to the detector.
16. Configure the detector.
17. Create a checkout method and verify performance.

2
System Description

This chapter provides typical performance specifications, and describes the theory of operation for the 8355 S SCD and 8255 S NCD Detectors.

Specifications

This section lists the published specifications for a new detector, installed on a new Agilent GC, when used in a typical laboratory environment. The specifications apply to the Agilent checkout sample.

8355 S SCD

Specification	
Minimum Detection Limit (MDL), typical	< 0.5 pg (S)/s (2x Agilent data system ASTM noise)
Linearity	> 10^4
Selectivity	> 2 x 10^7 response S/response C^2
Precision* and stability	< 2 % RSD over 2 hours < 5 % RSD over 24 hours
Typical time to reach 800 °C from ambient	10 min

* Typically, based on one run per 30 minutes, collected over 24 hours. For example, a 24 hour time span will contain approximately 48 replicate runs.

SCD specifications apply only when using air as the oxidizer gas.

8255 S NCD

Specification	
Minimum Detection Limit, typical	< 3 pg (N)/s (2x Agilent data system ASTM noise)
Linearity	> 10^4
Selectivity	> 2 x 10^7 response N/response C
Area repeatability	< 1.5 % RSD over 8 hours < 2 % RSD over 18 hours
Typical time to reach 900 °C from ambient	10 min

MDL calculations

The MDL specifications are defined using the Agilent checkout standard for SCD or NCD.

Sensitivity is typically reported as:

$$\text{Sensitivity} = \frac{\text{peak area}}{\text{amount}}$$

Calculate a minimum detection limit (MDL) from the following formula:

$$\text{MDL} = \frac{2 \text{ x noise}}{\text{sensitivity}}$$

where the noise is the ASTM noise reported by the Agilent data system.

Theory of Operation

The SCD and NCD chemiluminescence detectors detect target molecules by chemically transforming them in several steps to an excited species that emits light. The light from this emission is converted to an electrical signal by a photomultiplier tube (PMT). For each detector, samples undergo preliminary reaction(s) with an oxidizer (air or oxygen for SCD, oxygen for NCD) and hydrogen in a very hot reaction zone (the burner) at reduced pressure to form either SO or NO in addition to other products such as H_2O and CO_2. The reaction products then flow to a reaction cell in a separate detector module. In this cell, they mix with ozone (O_3) produced from oxygen using an ozone generator. The O_3 reacts with SO or NO to generate SO_2^* and NO_2^* respectively. This reaction cell operates at a pressure of about 4-7 Torr. These high energy species return to ground state by chemiluminescence. The emitted light is filtered and then is detected by a PMT. The electrical signal produced is proportional to the amount of SO_2^* or NO_2^* formed in the reaction cell. The sample exits the reaction cell, passes through an ozone destruction trap, and then passes through a vacuum pump and out to vent.

SCD

The SCD uses the chemiluminescence (light-producing reaction) from the reaction of ozone with sulfur monoxide (SO) produced from combustion of the analyte:

$$\text{Sulfur compound (analyte)} \rightarrow SO + H_2O + \text{other products}$$

$$SO + O_3 \rightarrow SO_2 + O_2 + h\nu\ (< 300\text{-}400\ \text{nm})$$

The pressure differential produced by a vacuum pump transfers the combustion products into a reaction cell, where excess ozone is added. Light ($h\nu$) produced from the subsequent reaction is optically filtered and detected with a blue-sensitive photomultiplier tube, and the signal is amplified for display or output to a data system.

NCD

The NCD uses the chemiluminescence of ozone with nitric oxide formed from combustion. Reacting nitric oxide with ozone results in the formation of electronically excited nitrogen dioxide. The excited nitrogen dioxide emits light, a chemiluminescence reaction, in the red and infrared region of the spectrum. The light emitted is directly proportionally to the amount of nitrogen in the sample:

$$NO + O_3 \longrightarrow NO_2 + O_2$$

$$NO + O_3 \longrightarrow NO_2 + O_2 + h\nu \text{ (~600 to 3,000 nm)}$$

The light (hν) emitted by the chemical reaction is optically filtered and detected by a PMT. A chiller cools the PMT to reduce thermal noise and help measure infrared light. The signal from the PMT is amplified for display or output to a data system.

Description of Major Components

Burner assembly

The burner assembly mounts on top of the GC in a detector location, and contains the column connection.

For the SCD, the burner provides two heated zones, one at the base and one farther up the assembly. In the burner base region, the column effluent mixes with the lower hydrogen flow and air or oxygen at high temperature. The resulting hydrogen flame combusts the effluent. Low concentration components burn to form the usual combustion products, including SO_2 for compounds containing sulfur. The products are drawn upward through a ceramic tube, where at even higher temperature the upper hydrogen flow mixes with the combustion products, causing the SO_2 to reduce into SO.

Figure 6 shows the flow paths for the SCD burner assembly.

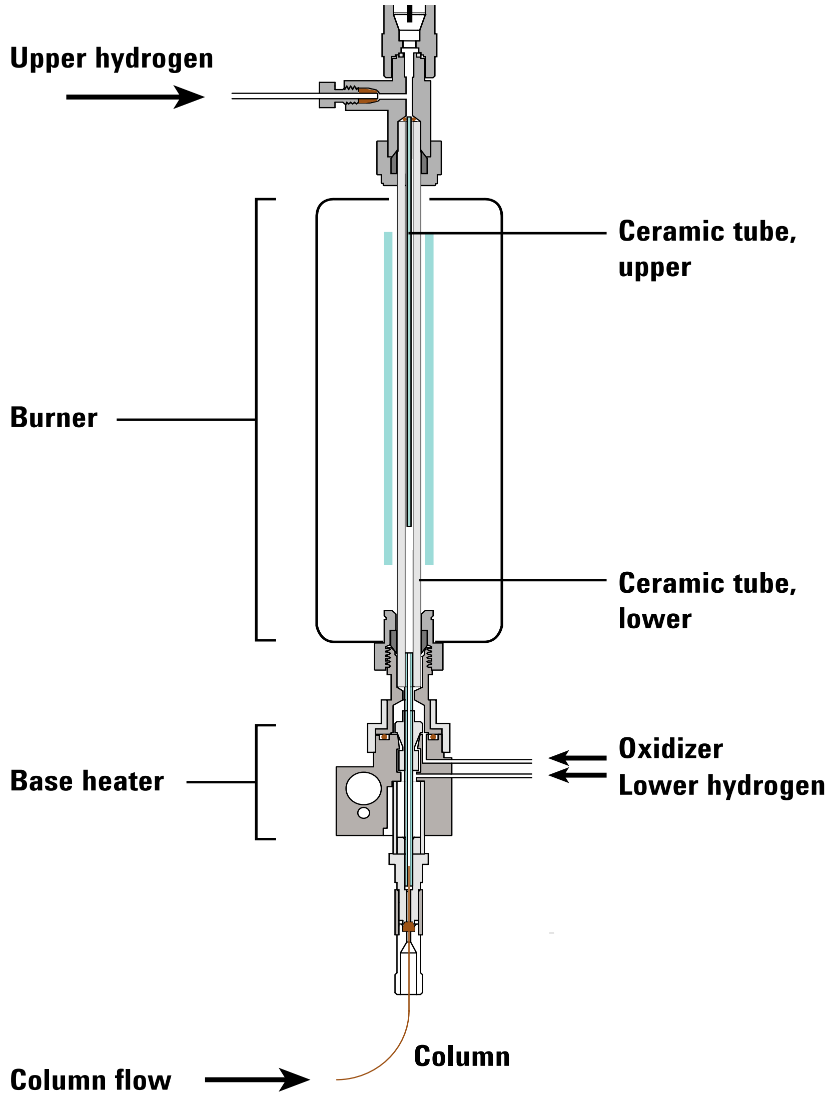


Figure 6 SCD flows

For the NCD, the burner provides two heated zones, one at the base and one farther up the assembly. In the burner base region, the column effluent mixes with hydrogen and air at high temperature. The resulting hydrogen flame combusts the effluent. Low concentration components will burn to form the usual combustion products, including NO_2 for compounds containing nitrogen. The products are drawn upward through a quartz tube and catalyst, where a high temperature will convert NO_2 into NO.

Figure 7 shows the flow paths for the NCD burner assembly.

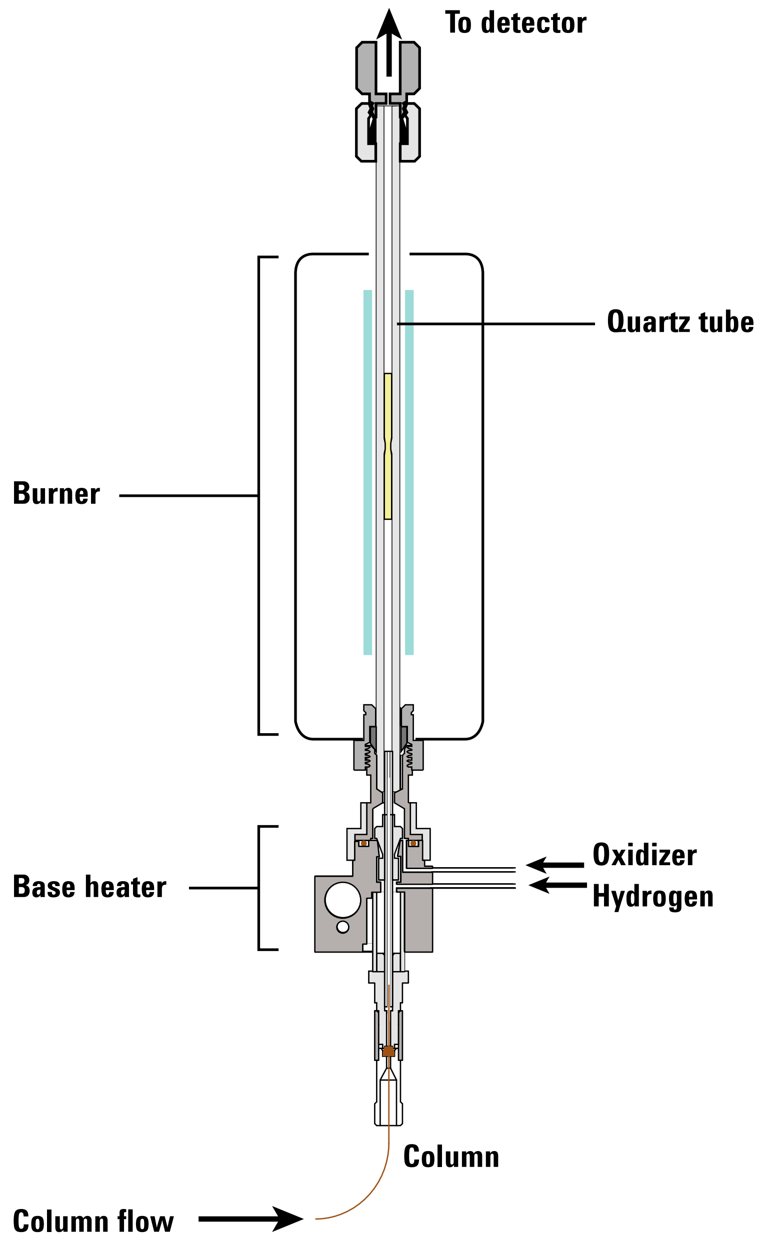


Figure 7 NCD Flows

Ozone generator

The ozone generator provides ozone that reacts with any SO or NO in the reaction cell to generate SO_2* and NO_2* respectively. These high energy species return to ground state by chemiluminescence.

The detector regulates the ozone gas supply pressure to maintain a fixed flow from the ozone generator.

Reaction cell and photomultiplier tube (PMT)

The ozone generator discharges ozone into the reaction cell. This ozone reacts with any SO or NO to generate SO_2^* and NO_2^* respectively. As the species return to ground state though chemiluminescence, the photomultiplier tube produces a current proportional to the intensity of emitted light. A bandpass filter is used to optimize the detector for either sulfur or nitrogen detection.

EPC modules

The detector controls hydrogen, oxidizer (air or oxygen), and ozone supply (oxygen) gas flows using two electronic pressure control modules.

Vacuum pump

A two-stage, oil-sealed rotary vacuum pump provides an operating pressure between 3 and 10 Torr in the reaction cell. This vacuum helps transfer combustion gases from the burner to the reaction cell, as well as transferring the ozone from the ozone generator into the reaction cell. The vacuum pump also reduces non-radiative collisional quenching of the emitting species in the reaction cell.

Ozone destruction trap

A chemical trap between the detector exhaust and the vacuum pump destroys ozone, converting it to diatomic oxygen. Unconverted ozone reduces pump life.

Oil coalescing filter

The oil-sealed rotary vacuum pump uses a partially-open gas ballast to aid in the elimination of water produced in the burner and transferred to the pump. As a result of the open gas ballast and the relatively high flow rates of gases, oil vaporized in the pump can escape through the pump exhaust. To minimize oil loss, the pump includes an oil coalescing filter on the pump exhaust to trap vaporized oil and to return this oil to the vacuum pump oil reservoir.

FID adapter (optional)

The SCD burner normally mounts onto the GC oven directly as a stand-alone detector. However, some applications also require simultaneous detection of hydrocarbon components using a single column without splitting. For this reason, Agilent offers an optional FID adapter to mount the burner assembly onto an FID for the simultaneous collection of FID and SCD chromatograms. During dedicated SCD operation, 100 % of the column effluent passes through the burner to the detector. During simultaneous detection, approximately 10 % of the FID exhaust gases are drawn into the burner through a restrictor, which reduces SCD sensitivity to approximately 1/10 of the signal observed in a dedicated SCD burner.

NCD chiller

For NCD, the detector uses a Peltier cooler to lower the PMT temperature, which in turn reduces noise. This chiller cools the PMT relative to the current ambient temperature. Higher laboratory ambient temperatures may result in higher PMT temperatures. Fluctuations in ambient temperature may result in fluctuations in PMT temperature.

Since noise and response determine the MDL, the efficiency of the chiller can influence the MDL. Depending on the ambient temperature, the chiller may not be able to maintain a sufficiently cool temperature in the PMT, and XCD noise will increase, therefore increasing the MDL.

Because chiller efficiency depends on the ambient temperature inside the detector and in the laboratory, the chiller setpoint does not impact detector readiness. A GC run can start regardless of whether or not the chiller has cooled to its setpoint.

3
Keypad Operation

This section describes the basic operation of the Agilent 8355 S SCD and 8255 S NCD Detectors keypad.

The Detector Key

These keys are used to set the temperatures, pressures, flows, and other method operating parameters for the detector.

To display the current settings, press [**Det**] or [**Analog Out**]. More than three lines of information may be available. Use the scroll keys to view additional lines, if necessary.

To change settings, scroll to the line of interest, enter the change, and press [**Enter**].

For context-sensitive help, press [**Info**]. For example, if you press [**Info**] on a setpoint entry, the help provided would be similar to: Enter a value between 0 and 350.

[**Det**]	Controls detector operating conditions.
[**Analog Out**]	Controls range attenuation and zero offset.

The Status Key

Displays **Ready**, **Not ready**, and **Fault** information.

The order in which items appear in the scrolling display window for [**Status**] can be modified. You may, for example, want to display the things you most frequently check in the top three lines so that you do not need to scroll to see them. To change the order of the **Status** display:

Press [**Config**]. Scroll to **Status** and press [**Enter**].

Scroll to the setpoint you want to appear first and press [**Enter**]. This setpoint will now appear at the top of the list.

Scroll to the setpoint you want to appear second and press [**Enter**]. This setpoint will now be the second item on the list.

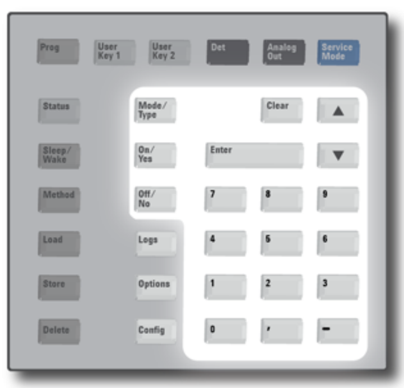
Continue as above until the list is in the order you require.

The Info Key

Provides help for the currently shown parameter. In other cases, [**Info**] will display definitions or actions that need to be performed.

The General Data Entry Keys

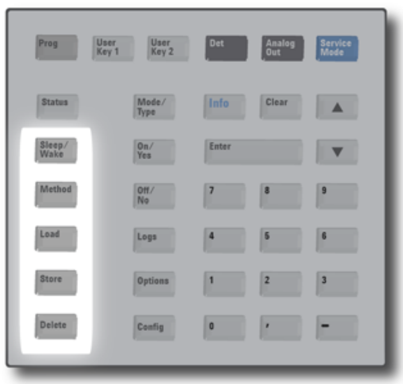
[**Mode/Type**]	Accesses a list of possible parameters associated with a component's nonnumerical settings.
[**Clear**]	Removes a misentered setpoint before pressing [**Enter**]. It can also be used to return to the top line of a multiline display, return to a previous display, cancel a function during a method, or cancel loading or storing methods.
[**Enter**]	Accepts changes you enter or selects an alternate mode.
	Scrolls up and down through the display one line at a time. The < in the display indicates the active line.
Numeric keys	Are used to enter settings for the method parameters. (Press [**Enter**] when you are finished to accept the changes.)
[**On/Yes**] [**Off/No**]	These keys are used to set parameters, such as turning the pump on, high voltage on.



Method Storage Keys

These keys are for loading and storing methods locally on your detector. They cannot be used to access methods stored by your Agilent data system.

[**Load**] [**Method**] [**Store**]	Used together to load and store methods on your detector. For example, to load a method, press [**Load**] [**Method**] and select one from the list of methods stored in the GC.
[**Delete**]	Deletes methods.
[**Sleep/Wake**]	The detector stores a schedule, based on the on-board clock, along with two special methods called SLEEP and WAKE. See "Resource Conservation" on page 49 for additional information.



To load a method

1 Press [**Load**].

2 Press [**Method**].

3 Enter the number of the method to be loaded.

4 Press [**On/Yes**] to load the method and replace the active method. Alternatively, press [**Off/No**] to return to the stored methods list without loading the method.

To store a method

1 Ensure that the proper parameters are set.

2 Press [**Method**].

3 Scroll to the method to store, then press [**Enter**].

4 Press [**On/Yes**] to store the method and replace the active method. Alternatively, press [**Off/No**] to return to the stored methods list without storing the method.

The Service Mode Key

Is used to set up Early Maintenance Feedback and to access inlet leak checks for selected inlet types. This key accesses settings intended for service personnel. Because these advanced settings can cause problems if misused, avoid the service settings unless specifically directed to use them.Is used to access maintenance functions and settings, service counters, and diagnostics for the GC.

To view the ozone flow rate, press [**Service Mode**]. Scroll to **Diagnostics**, press [**Enter**], then select **Diag 03 Generator**.

The Supporting Keys

Three logs are accessible from the keypad: the run log, the maintenance log, and the system event log.

[**Logs**]	Access the Maintenance Log, and the System Event Log. The information in these logs can be used to support Good Laboratory Practices (GLP) standards.
[**Options**]	Accesses the instrument parameters setup options for calibration, communications, and the display. Scroll to the desired line and press [**Enter**] to access the associated entries.
[**Config**]	Accesses the detector for configuration and time settings.
[**Prog**] [**User Key1**] [**User Key2**]	Use to program a series of keystrokes commonly for specific operations. Press [**Prog**] [**User Key 1**] or [**Prog**] [**User Key 2**] to record up to 21 keystrokes as a macro.

Configuration

Ignore Ready=

To ignore an element's readiness, press [**Config**] and scroll to **Detector** and press [**Enter**]. Scroll to **Ignore Ready** and press [**On/Yes**] to set it to **True**.

To set time and date

1 Press [**Config**] and scroll to **Time** and press [**Enter**].

2 Select **Time zone (hhmm)** and enter the local time offset from GMT using a 24 hour format.

3 Select **Time (hhmm)** and enter the local time.

4 Select **Date (ddmmyy)** and enter the date.

Maintenance and System Event Logs

Two logs are accessible from the keypad: the run log and the system event log. To access the logs, press [**Logs**] then scroll to the desired log and press [**Enter**]. The display will indicate the number of entries the log contains. Scroll through the list.

Maintenance logs

The maintenance log contains entries made by the system when any of the user-defined component counters reach a monitored limit. The log entry contains a description of the counter, its current value, the monitored limits, and which of its limits has been reached. In addition, each user task related to the counter is recorded in the log, including resetting, enabling or disabling monitoring, and changing limits or units (cycles or duration).

System event log

The system event log records significant events during the detector’s operation. Some of the events also appear in the run log if they are in effect during a run.

4
Operation

This chapter describes the how to use the 8355 S SCD and 8255 S NCD detectors. This chapter assumes familiarity with detector keypad and display.

Introduction

Standalone version

Use the front keypad on the 8355 S SCD or the 8255 S NCD detector to access the operating parameters. The settings and information provided on the detector keypad include:

- Settings for temperatures, flows, and gas types
- Sequence integration
- Method storage

Setting Parameters

This section lists the parameter ranges for the SCD and NCD. The available setpoints provide a wide range suitable for a large variety of applications as well as for method development. See "Adjusting the Operating Conditions" on page 40 for important details about the relationships between the setpoints.

Parameters and ranges

The table below lists the available parameters for the detector.

Table 1 8355 S SCD and 8255 S NCD parameters and ranges

Parameter	Range, SCD	Range, FID-SCD	Range, NCD
Method			
Base temperature	125 – 400 °C	125 – 400 °C	125 – 400 °C
Burner temperature	100 – 1000 °C	100 – 1000 °C	100 – 1000 °C
Chiller temperature (NCD only)*			On/Off
Lower hydrogen flow	5 – 25 mL/min	—	1 – 25 mL/min
Upper hydrogen flow (SCD only)	25 – 100 mL/min	25 – 100 mL/min	—
Oxidizer flow	25 – 150 mL/min. (Air) 5 – 30 mL/min. (Oxygen)	5 – 100 mL/min, (Air) 5 – 30 mL/min, (Oxygen)	4 – 80 mL/min
O3 Generator flow	On/Off	On/Off	On/Off
O3 Generator power	On/Off	On/Off	On/Off
Vacuum pump	On/Off	On/Off	On/Off

* Chiller (PMT cooler) operation depends on the current detector ambient temperature. The actual chiller temperature does not impact detector readiness. See "NCD chiller" on page 24.

Note that PMT voltage is fixed at 800 V.

Detector Stability and Response

The time required for system stabilization varies depending on the application, system cleanliness, the presence of active sites, and other factors.

- When starting an existing system, typically wait at least 10 minutes before using the system to collect data.
- A new burner or a new set of ceramic tubes may take up to 24 hours to condition. Set the detector to the operating conditions, and monitor the baseline until the baseline becomes stable enough for your application.

Typical Operating Conditions

Table 2 lists the recommended starting conditions for SCD and NCD methods. These conditions should provide acceptable results for a wide variety of applications. However, optimize these conditions as needed to improve the performance of the specific application.

Table 2 Typical operating conditions, SCD and NCD

Parameter	SCD	NCD
Base temperature, °C	250	250
Burner temperature, °C	800	900
Chiller temperature	N/A	On
Upper H_2 flow, mL/min	40	N/A
Lower H_2 flow, mL/min	10	3
Oxidizer flow, mL/min	50 (Air) 10 (Oxygen)	8 (Oxygen)
O3 Generator flow, mL/min	On	On
O3 Generator power	On	On
Vacuum pump	On	On
Burner pressure, Torr, typical reading	<450 Torr, Air <300 Torr, Oxygen	< 120 Torr
Reactor pressure, Torr (read only)	Should be below 7 Torr	Should be below 5 Torr

The checkout methods for the SCD and NCD also provide example parameters for balancing good detection limit, good selectivity, and reasonable ceramic tube life. In any XCD method:

- Always keep oxidizer gas flowing through the burner.
- The firmware will not allow hydrogen to flow into the burner while there is no oxidizer to protect the system.

During startup and shutdown, always turn on the pump first and turn off the pump last to prevent contamination or damage.

Adjusting the Operating Conditions

Table 1 on page 37 lists the range of values for each parameter, as limited by the GC firmware.Table 1 on page 37 lists the range of values for each parameter, as limited by the detector firmware. To provide flexibility during method development for particular application, the range is wider than needed for most applications.

However, hydrogen flows in the SCD need particular attention. Using very high hydrogen flows (both upper and lower), relative to the oxidizer flow, can permanently damage the ceramic tubes. This condition may not be recoverable. See "Hydrogen Poisoning" on page 93.

SCD lower hydrogen flow: Very high flow can damage the ceramic tubes.

NCD lower hydrogen flow: The NCD can operate without hydrogen flow, although this is not recommended. The hydrogen flame/plasma can help burn off solvent and heavy molecules. If operating an NCD without hydrogen flow, plumb the 1/16-inch tubing for the lower hydrogen flow to the oxygen supply. Otherwise, residual hydrogen in the tubing will continue diffuse into the burner and affect stability.

1 Disconnect the **Lower H2** line from the back of the detector and cap off the detector fitting.
2 Install a 1/16-inch Swagelok Tee fitting in the **Oxidizer** output from the detector body.
3 Connect the **Oxidizer** and **Lower H2** lines to the Tee fitting.

You will typically need to adjust the recommended starting conditions to create an optimized method for your application. When optimizing SCD or NCD method parameters, consider the following:

- A higher hydrogen to oxidant ratio may initially show higher response, but later yield a reduced response because of the accumulation of contaminants that reduce detector response, such as soot or other active species.
- Operating the burner at higher temperatures may shorten the useful lifetimes of the heater, thermocouple, and seal materials.

In general, when making any parameter change, allow sufficient time for the system to reach equilibrium. Monitor the baseline until it stabilizes at its new value.

Start-up

How to start the detector depends on whether or not you have created a method for the detector.

If a valid method exists: After you have used the SCD/NCD (at least one valid method exists), start the detector by loading the method. As soon as the method loads, the GC will turn on the vacuum pump and oxidizer flows, and also turn on all other parameters except hydrogen flow. The GC will monitor the temperatures and prevent hydrogen flow until the base temperature reaches 150 °C and the burner temperature reaches 200 °C. Once the detector temperatures reach these minimum limits, the GC turns on the hydrogen flow.As soon as the method loads, the detector will turn on the vacuum pump and oxidizer flows, and also turn on all other parameters except hydrogen flow. The detector will monitor the temperatures and prevent hydrogen flow until the base temperature reaches 150 °C and the burner temperature reaches 200 °C. Once the detector temperatures reach these minimum limits, the detector turns on the hydrogen flow.

During initial startup, or whenever there are no method parameters set for the SCD or NCD, start the detector as follows:

1. Access the method parameters.
   - At the detector front panel, press **[Det]**.
2. Turn on the vacuum pump.
3. Set the oxidizer flow rate and turn on the oxidizer flow.
4. Wait 1–2 minutes for the vacuum pump to purge the system using the oxidizer flow.
5. Set the base temperature and turn it on.
6. Set the burner temperature and turn it on.
7. For NCD only: Set the chiller temperature and turn it on.
8. Set the hydrogen flow and turn it on.
9. Set the ozone generator supply gas flow and turn it on.
10. Turn on the ozone generator power.

The detector will monitor the temperatures and prevent hydrogen flow until the base temperature reaches 150 °C and the burner temperature reaches 200 °C. Once the detector temperatures reach these minimum limits, the detector turns on the hydrogen flow.

Shutdown

When turning off the detector for a long period of time, or to perform maintenance on the GC or detector, shut down the detector as follows:

1 Access the method parameters.
   - At the detector keypad, press **[Det]**.
2 Turn off the ozone generator power.
3 Turn off the ozone generator supply gas flow.
4 Turn off all hydrogen flows.
5 For NCD only: Turn off the chiller.
6 Turn off the burner heater.
7 Turn off the base heater.

NOTE

When shutting down, the detector will keep the vacuum pump and oxidizer flow running until approximately 100 mL of oxidizer gas has purged the system after the hydrogen flow is turned off. This action prevents moisture residual contamination.

8 Turn off the oxidizer flow.
9 Turn off the vacuum pump.
10 Turn off the detector power.
11 If shutting down the GC, turn off the GC.

WARNING

Burn hazard. Many parts of the detector can be dangerously hot. If performing detector maintenance, turn off all heated zones and monitor them until they reach a safe handling temperature before turning off the GC and detector.

Alternately, create a method that turns off all detector components, and load that method to shut down the detector.

Flow and Pressure Sensors

The EPC gas control modules contain flow and/or pressure sensors that are calibrated at the factory. Sensitivity (slope of the curve) is quite stable, but zero offset requires periodic updating.

Flow sensors

The detector uses flow sensors to monitor hydrogen flow. If the **Autoflow zero** feature is on, they are zeroed automatically. This is the recommended way. They can also be zeroed manually (see “Zero a specific flow or pressure sensor” on page 44).

Pressure sensors

All EPC control modules use pressure sensors. The must be zeroed individually. There is no automatic zero for pressure sensors. Pressure sensors should be zeroed once every twelve months.

Configure auto flow zero

A useful calibration option is **Autoflow zero**. When it is **On**, after the end of a run the GC shuts down the flow of gases to an inlet, waits for the flow to drop to zero, measures and stores the flow sensor output, and turns the gas back on. This takes about two seconds. The zero offset is used to correct future flow measurements. Agilent recommends setting the detector to automatically zero flow sensors to reduce drift.

To activate:

1. On the detector keypad, press **[Options]**.
2. Scroll to **Calibration** and press **[Enter]**.
3. Scroll to **Autoflow zero (H2 Lower)** and press [**On/Yes**]. (To turn off autozero, instead press [**Off/No**].

Conditions for zeroing a sensor

Flow sensors are zeroed with the carrier gas connected and flowing.

Pressure sensors are zeroed with the supply gas line disconnected from the gas control module.

Zero a specific flow or pressure sensor

1 **Press [Options]**, scroll to **Calibration**, and press **[Enter]**.

2 Scroll to the sensor to be zeroed and press **[Enter]**.

3 Set the flow or pressure:

- **Flow sensors**. Verify that the gas is connected and flowing (turned on).
- **Pressure sensors**. Disconnect the gas supply line at the back of the GC. Turning it off is not adequate; the valve may leak.

4 Scroll to the desired zero line.

5 Press [**On/Yes**] to zero or [**Clear**] to cancel.

6 Reconnect any gas line disconnected in step 3 and restore operating flows.

Signal Output

The 8355 S and 8255 S detectors provides output data as an analog signal. Analog output ranges are 0 to 1 V, 0 to 10 V, and 0 to 1 mV. To use the detector with Agilent GCs (6850, 6890, 7820, and 7890), an Analog to Digital converter, such as the Agilent 35900E A/D Converter, or Analog Input Board (AIB) is required.

6850 Series GCs

- A single channel detector can connect to an AIB (accessory G3375A, or option 206), or external A/D converter.
- For a tandem FID/XCD, use an external A/D converter (such as the 35900E).

6890 Series GCs

- A single channel detector can connect to an AIB (part number G1556A, or option 206), or external A/D converter.
- A tandem configuration can use an AIB to collect the XCD signal, but the 6890 only supports two detectors total. A configuration such as dual FID-XCD, or an FID-XCD paired with any other detector requires an external A/D converter for the XCD.

7820 Series GCs

- Since an AIB is not supported by the 7820, you must use an A/D converter to collect the XCD signal.

7890 Series GCs

- A single channel detector can connect to an AIB (accessory G3456A, or option 206), or external A/D converter.

Intuvo 9000 GC

- A single channel detector can connect to an AIB (accessory G3456A, or option 206), or external A/D converter.

Signal Types

When assigning detector signals, use the [**Mode/Type**] and choose from the Signal Type parameter list, or press a key or combination of keys.

The nondetector signal is test plot. Access the test plot by pressing [**Mode/Type**]. Diagnostic signals are for use by your service representative and are not described in detail here.

Value

Value on the sginal parameter list is the same as **Detector** on the detector parameter list.

Analog Signals

If you use an analog recorder, you may need to adjust the signal to make it more usable. **Zero** and **Range** in the signal parameter list do this.

Analog zero

Zero subtracts value entered from baseline. Press [**On/Yes**] to set to current Value or [**Off/No**] to cancel.

This is used to correct baseline elevation or offsets. A common application is to correct a baseline shift that occurs as the result of a valve operation. After zeroing, the analog output signal is equal to the Value line of the parameter list minus the Zero setpoint.

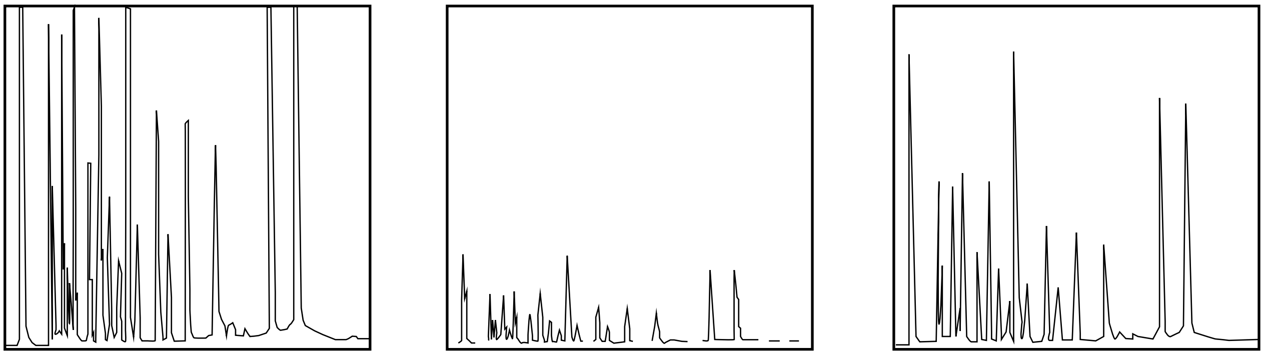
1 Verify that thew dectector is on and in a ready state.
2 Press [**Analog Out**].
3 Scroll to **Zero**.
4 Press [**On/Yes**] to set **Zero** at the current signal value, or enter a number between -500000 and +500000. A value smaller than the current **Zero** shifts baseline up.

Analog range

Range scales data coming from the detector.

Range is also referred to as gain, scaling, or sizing. It sizes the data coming from the detector to the analog signal circuits to avoid overloading the circuits (clamping). Range scales all analog signals.

Valid setpoints are from 0 to 13 and represent 2^0 (=1) to 2^{13} (=8192). Changing a setpoint by 1 changes the height of the chromatogram by a factor of 2. The following chromatograms illustrate this. Use the smallest possible value to minimize integration error.



A: Range = 0 B: Range = 3 C: Range = 1

When developing a method, first set the analog range to 9, if using an AIB, or to 10 if using an Agilent 35900E A to D. Adjust as necessary to view your data.

Analog data rates

Your integrator or recorder must be fast enough to process data coming from the GC. If it cannot keep up with the GC, the data may be damaged. This usually shows up as broadened peaks and loss of resolution.

Speed is measured in terms of bandwidth. Your recorder or integrator should have a bandwidth twice that of the signal you are measuring.

Methods

To load a method

1 Press [**Load**].
2 Press [**Method**].
3 Enter the number of the method to be loaded.
4 Press [**On/Yes**] to load the method and replace the active method. Alternatively, press [**Off/No**] to return to the stored methods list without loading the method.

To store a method

1 Ensure that the proper parameters are set.
2 Press [**Method**].
3 Scroll to the method to store, then press [**Enter**].
4 Press [**On/Yes**] to store the method and replace the active method. Alternatively, press [**Off/No**] to return to the stored methods list without storing the method.

Resource Conservation

For optimal performance and precision, leave the detector at operating conditions. The SCD achieves maximum precision when maintained at operating conditions for a long period of time. If your application requires the highest precision, you may prefer to not use the resource conservation features with an SCD. The NCD can more recover precision more quickly. With either detector, consider the cost savings for the resources against the recovery time for precision.

WARNING **Never put the detector into sleep mode while the GC is operating normally.**

Schedule

The detector uses a schedule, based on the on-board clock, along with two special methods called **SLEEP** and **WAKE**. A **SLEEP** method sets low flows and temperatures to conserve resources. A **WAKE** method sets new flows and temperatures, typically to restore operating conditions.

Load the sleep method at a specified time during the day to reduce flows and temperatures. Load the wake method or analytical method to restore settings before using the detector again. For example, load the sleep method at the end of each day or work week, then load the wake before arriving to work the next day. Experiment to find the best time to load the wake method.

Sleep Methods

Consider the following:

- Do not turn off the vacuum pump. The pump needs to be on to prevent shutdown conditions.
- Do not turn off the oxidizer flow.
- To conserve oxygen, turn off the ozone generator. This setting also turns off the oxygen flow.

For SCD:

The main concern is to avoid heating the ceramic tube without all gases flowing. Turning off the ozone generator will conserve oxygen without changing the flows through the ceramic tube. If you must reduce temperature or gas flow further, then you can reduce the oxidizer flow, turn off the hydrogen flows, and most importantly, reduce the burner temperature to about 250 C. The base temperature can be reduced also.

For NCD:

Turn off the ozone generator. If needed to further conserve energy and gases, reduce (but do not turn off) the oxygen and turn off the hydrogen flow. Reduce the burner temperature to about 250 °C and turn off the chiller.

To Set the Detector to Conserve Resources

Set the detector to conserve resources by selecting and using an **Instrument Schedule**:

1 Decide how to restore flows. The choices from the **Schedule Function** are:
   - **Go to Sleep with SLEEP method**. This method should reduce flows and temperatures. (While it is good practice to create these methods, you do not need them if you only wake the detector to the last active method.)
   - **Wake with the current method**. At the specified time, the detector will restore the last active method used before it went to sleep.
   - **Wake with WAKE method**. At the specified time, the detector will load the wake method, and remain at those settings.
2 Create a **SLEEP** method. This method should reduce flows and temperatures.
3 Program the **WAKE** method, as needed. (While it is good practice to create these methods, you do not need them if you only wake the detector to the last active method.)
4 Create the **Sleep/Wake Schedule**.
   a Press [**Sleep/Wake**].
   b Scroll to **Edit the Instrument Schedule?** and press [**Enter**].
   c Press [**Mode/Type**] to create a new schedule item.
   d When prompted, scroll to the desired day of the week and press [**Enter**].
   e When prompted, scroll to **Go to Sleep with SLEEP method** and press [**Enter**] then input the event time. Press [**Enter**]

f If desired, set the wake function while still viewing the schedule. Press [**Mode/Type**] to create a new schedule item.

g When prompted scroll to the desired day of the week and press [**Enter**].

h When prompted, scroll to **Wake with the current method** or **Wake to Wake method** and press [**Enter**], then input the event time. Press [**Enter**]

i Repeat steps b through g as needed for any other days of the week.

You do not have to program events for every day. For example, you can program the detector to sleep on Friday evening, then wake on Monday morning, keeping it continuously at operating conditions during weekdays.

To create or edit an instrument schedule

To create a new schedule or edit an existing schedule, delete unwanted items, then add new items as desired.

1 Create the **Sleep/Wake Schedule**:

a Press [**Sleep/Wake**].

b Scroll to **Edit the Instrument Schedule?** and press [**Enter**].

c Press [**Mode/Type**] to create a new schedule item.

d When prompted, scroll to the desired day of the week and press [**Enter**].

e When prompted, scroll to **Go to Sleep with SLEEP method** and press [**Enter**] then input the event time. Press [**Enter**]

f If desired, set the wake function while still viewing the schedule. Press [**Mode/Type**] to create a new schedule item.

g When prompted scroll to the desired day of the week and press [**Enter**].

h When prompted, scroll to **Wake with the current method** or **Wake to Wake method** and press [**Enter**], then input the event time. Press [**Enter**]

i Repeat steps b through g as needed for any other days of the week.

2 Edit the **Sleep/Wake Schedule**:

   a Load a method with similar setpoints.

   b Edit the method setpoints. The detector only allows for setting relevant parameters.

   - For a **SLEEP** method, the detector sets the initial detector temperature and flow rates.
   - For a **WAKE** method, the detector sets the same parameters as the sleep method.

   c Press [**Method**], scroll to the method to store (**SLEEP** or **WAKE**), and press [**Store**]. press [**Enter**].

   d If prompted to overwrite, press [**On/Yes**] to overwrite the existing method or [**Off/No**] to cancel.

To put the detector to sleep now

1 Press [Sleep/Wake].

2 Select **Go to sleep now**, then press [**Enter**].

To wake the detector now

1 Press [Sleep/Wake].

2 Select the desired wake choice, then press [**Enter**].

   - When prompted, scroll to **Wake with the current method** and press [**Enter**]. Exit sleep mode by loading the last active method used before going to sleep.
   - **Wake to Wake method** and press [**Enter**]. Exit sleep mode by loading the **WAKE** method.

5
Maintenance

This chapter describes the routine maintenance procedures needed for normal use of the SCD and NCD.

Maintenance Schedule

To maintain optimum performance of the Agilent 8355 S SCD and 8255 S NCD, routinely replace the ozone trap, oil coalescing filter, and vacuum pump oil. Refer to Table 3 for the expected life span of each item.

Table 3 Recommended Edwards RV5 vacuum pump maintenance schedule

Component	Operating life*
Ozone destruction trap (converts O_3 to O_2)	~ 6 months
Oil coalescing filter	~ 3 months
Oil odor filter	~ 3 months if needed
Pump oil†	~ 3 months
Oil level	Check weekly

* The operating life is based on the total time logged during operation of the detector with the burner and the ozone generator On.

† Pump oil can be purchased from a supplier or directly from Agilent: SAE 10W-30, Multiviscosity Synthetic Motor Oil such as, MOBIL 1 or AMSOIL.

Tracking Detector Sensitivity

Sensitivity reflects the performance characteristics of a given system, and decreased sensitivity may indicate the need for routine detector maintenance.

The MDL specifications are defined using the Agilent checkout standard for SCD or NCD.

Sensitivity is typically reported as:

$$\text{Sensitivity} = \frac{\text{peak area}}{\text{amount}}$$

Calculate a minimum detection limit (MDL) from the following formula:

$$\text{MDL} = \frac{2 \text{ x noise}}{\text{sensitivity}}$$

where the noise is the ASTM noise reported by the Agilent data system.

Consumables and Replacement Parts

See the Agilent catalog for consumables and supplies for a more complete listing, or visit the Agilent Web site for the latest information (http://www.chem.agilent.com/store).

Table 4 Consumables and parts for the SCD and NCD

Description/quantity	Part number
Detector parts	
Ceramic tube, inner, small (SCD) (pack of 3) (See Exploded Parts View of the SCD)	G3488-60008
Quartz tube (NCD)	G6600-80063
Ferrule, 1/4-inch, graphite, straight, 10/pk for SCD outer ceramic tube and NCD quartz tube	0100-1324
Column installation tool (See Exploded Parts View of the SCD)	G3488-81302
Sulfur chemiluminescence test sample	5190-7003
Nitrogen chemiluminescence test sample	5190-7002
Vacuum pump parts	
RV5 pump – 110 V/230 V – Inland	G6600-64042
Pump tray, RV5 pump	G1946-00034
PM Kit, RV5 oil pump	G6600-67007
Oil mist filter for RV5 pump, for SCD/NCD	G6600-80043
Replacement oil coalescing filter, RV5 pump	G6600-80044
Replacement odor filtration element	G6600-80045
Ozone destruction trap	G6600-85000
Oil return line, RV5 pump	3162-1057
Oil, synthetic, Mobil 1	G6600-85001
NW 20/25 clamping ring (for oil mist filter)	0100-0549
NW 20/25 clamping ring (for exhaust hose)	0100-1398
Tools	
Funnel	9301-6461
Wrench, Allen, 5-mm	8710-1838
Screwdriver, flat-bladed	8710-1020
Gloves, chemical resistant, lint-free	9300-1751

Table 5 Filters for the SCD and NCD

Description/quantity	Part number
Gas Clean filter, sulfur (filters sulfur and moisture)	CP17989
Gas Clean Filter SCD Kit, for sulfur chemiluminescence detectors	CP17990

Table 6 Nuts, ferrules, and hardware for capillary columns

Column id (mm)	Description	Typical use	Part number/quantity
.53	Ferrule, graphite, 1.0-mm id	0.53-mm capillary columns	5080-8773 (10/pk)
	Ferrule, graphite, 0.8-mm id	0.53-mm capillary columns	500-2118 (10/pk)
	Column nut, finger-tight (for 0.53-mm columns)	Connect column to inlet or detector	5020-8293
.45	Ferrule, graphite, 0.8-mm id	0.45-mm capillary columns	500-2118 (10/pk)
.32	Ferrule, graphite, 0.5-mm id	0.1-mm, 0.2-mm, 0.25-mm, and 0.32-mm capillary columns	5080-8853 (10/pk)
	Column nut, finger-tight (for .100- to .320-mm columns)	Connect column to inlet or detector	5020-8292
.1 – .25	Ferrule, graphite, 0.4-mm id	0.1-mm, 0.2-mm, 0.25-mm, and 0.32-mm capillary columns	500-2114 (10/pk)
	Column nut, finger-tight (for .100- to .320-mm columns)	Connect column to inlet or detector	5020-8292
All	Ferrule, no-hole	Testing	5181-3308 (10/pk)
	Capillary column blanking nut	Testing–use with any ferrule	5020-8294
	Column nut, universal	Connect column to inlet or detector	5181-8830 (2/pk)
	Column cutter, ceramic wafer	Cutting capillary columns	5181-8836 (4/pk)
	Ferrule tool kit	Ferrule installation	440-1000

Exploded Parts View of the SCD

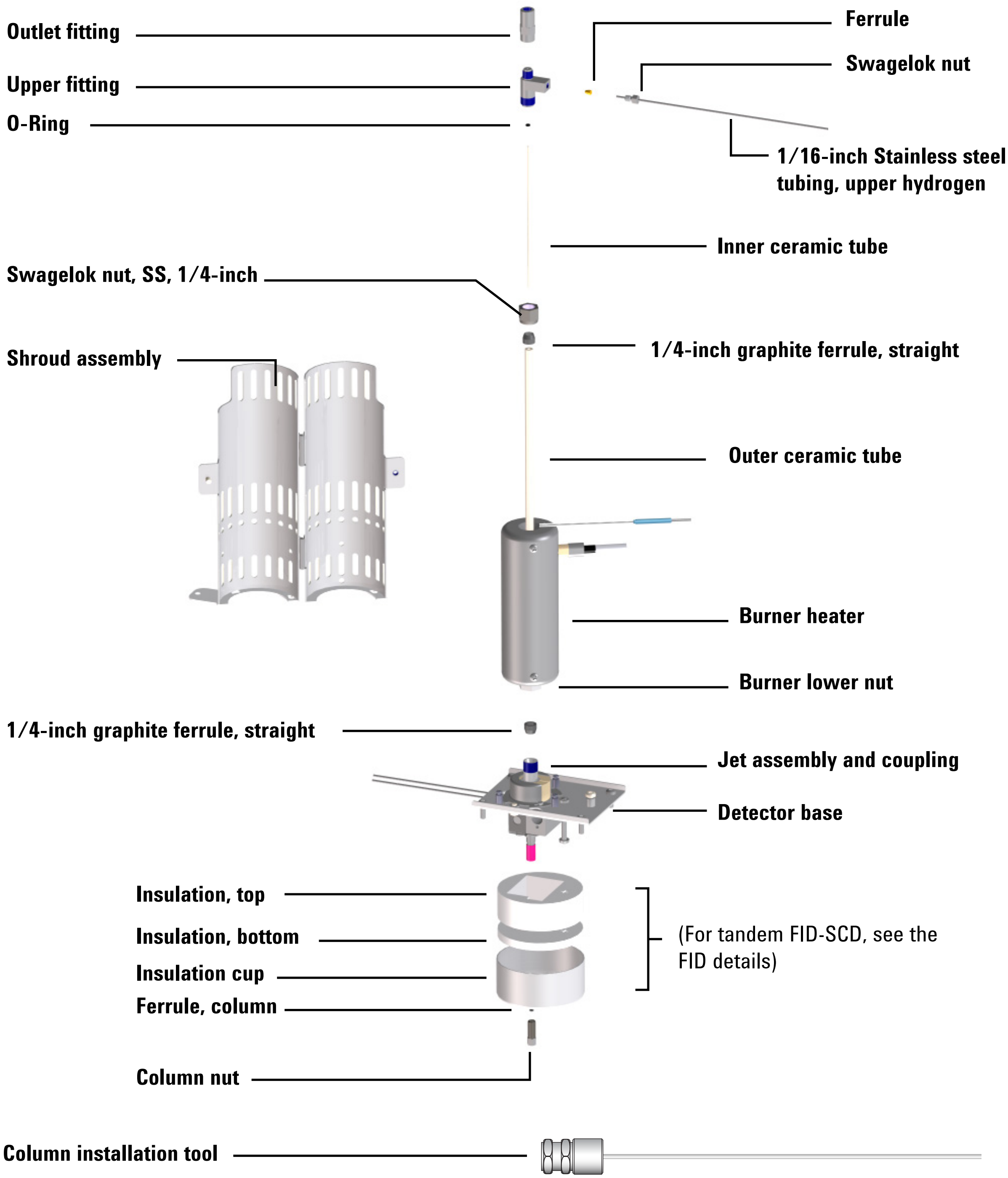


Figure 8 SCD Exploded parts view

Exploded Parts View of the NCD

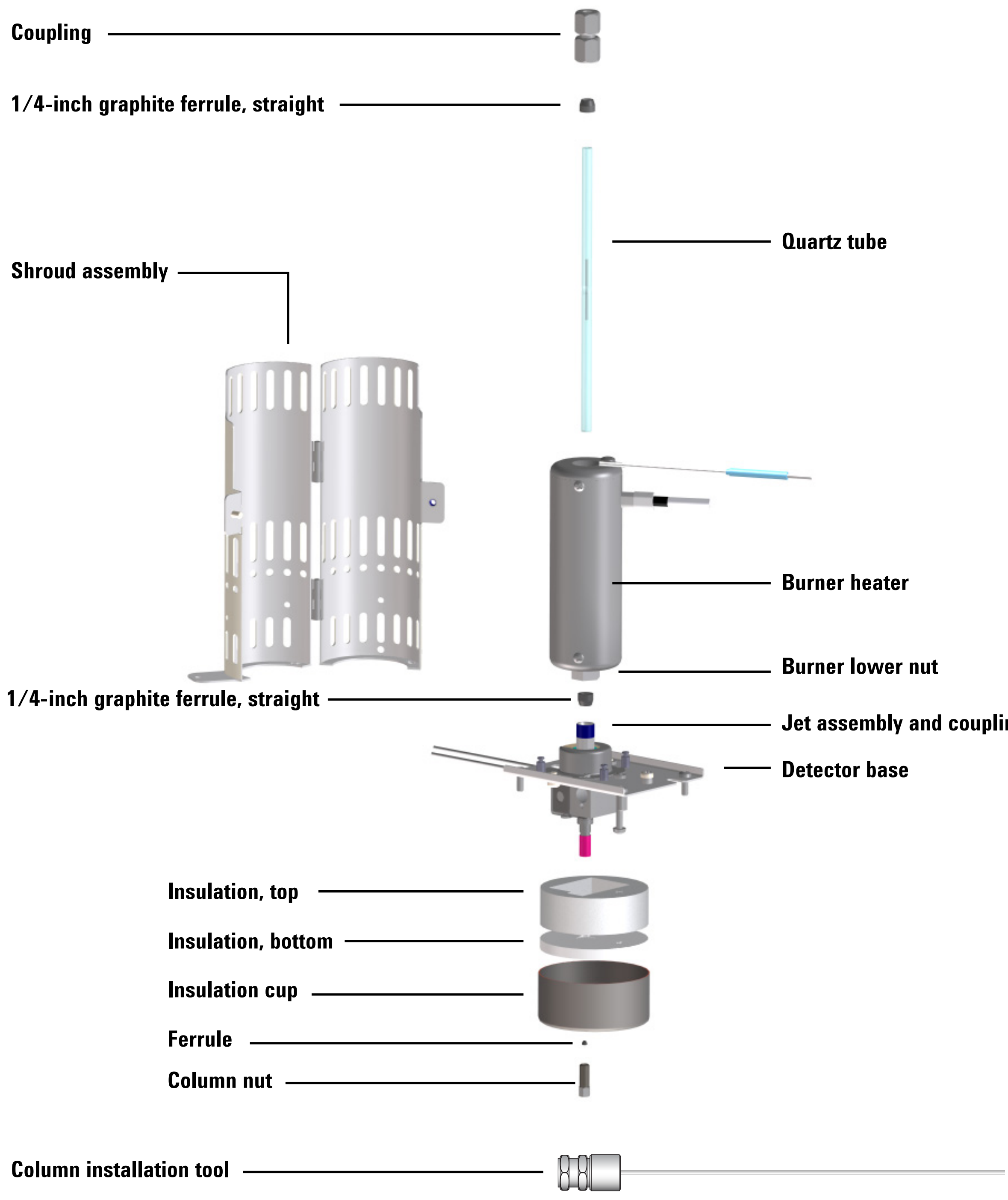


Figure 9 NCD exploded parts view

Detector Maintenance Method

It is good practice to create a maintenance method for the GC that prepares the GC and detector for maintenance. Load this method before performing maintenance.

A maintenance method for the SCD or NCD should do the following:

1 Turn off the heater and burner to allow them to cool.
2 Turn off all hydrogen flows.
3 Leave on the oxidizer and ozone generator supply gases.
4 Turn off the ozone generator.
5 Leave on the vacuum pump.
6 Keep (helium) carrier gas flowing.
7 Set the oven to 30 °C to minimize column bleed.

In addition, cool any other parts of the detector as needed. Allow heated zones to cool to < 40 °C for safe handling.

Attach a Column to the Detector

(Does not apply to the Intuvo 9000 GC.)

NOTE

This procedure describes how to attach a column directly to an XCD. In a tandem FID-XCD installation, install the column into the FID as described in the FID instructions. See the GC documentation.

1 Gather the following materials (see "Consumables and parts for the SCD and NCD" on page 56):
    - Column installation tool for SCD/NCD (G3488-81302)
    - Column
    - Ferrule (for column)
    - Column nut
    - Column cutter
    - 1/4-inch open-end wrench
    - Septum
    - Isopropanol
    - Lab tissue
    - Lint-free gloves
    - Magnifying loupe

WARNING

The oven, inlet, or detector may be hot enough to cause burns. If the oven, inlet, or detector is hot, wear heat-resistant gloves to protect your hands.

WARNING

Wear safety glasses to protect your eyes from flying particles while handling, cutting, or installing glass or fused silica capillary columns. Use care in handling these columns to prevent puncture wounds.

CAUTION

Wear clean, lint-free gloves to prevent contamination of parts with dirt and skin oils.

2 Cool the GC oven, inlet, and other heated zones to a safe handling temperature (< 40 °C).

3 Prepare the detector for maintenance.

   a Load the maintenance method and wait for the detector to become ready. (See “Detector Maintenance Method” on page 60.) Wait until the detectors, burner assembly, and detector base cool to a safe handling temperature (< 40 °C).

   b Turn off all hydrogen flows. (Leave on the oxidizer and ozone supply gases.)

   c Turn off the ozone generator.

WARNING **Hydrogen gas is flammable. Turn off all detector (and column) hydrogen gas flows before performing maintenance on the detector.**

4 Place a septum, capillary column nut, and ferrule on the column.

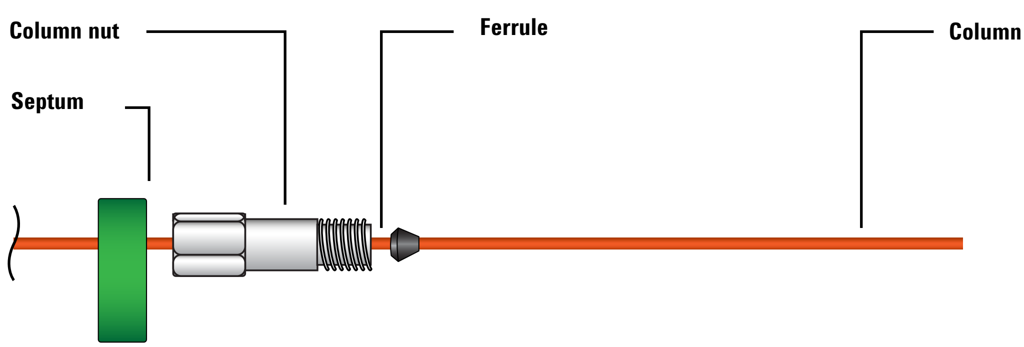


Figure 10 Place septum, column nut, and ferrule on the column

5 Insert the end of the column through the column measuring tool so that the end protrudes beyond the tool.

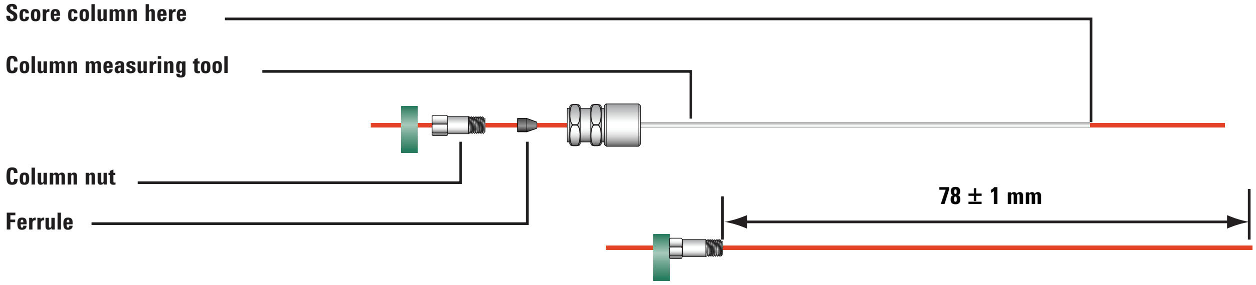
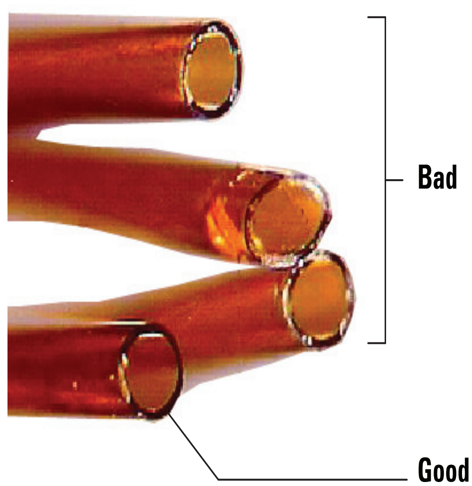


Figure 11 Set column length and swage ferrule using column measuring tool

6 Tighten the column nut into the column measuring tool until the column nut grips the column. Tighten the nut an additional 1/8- to 1/4-turn with a pair of wrenches. Snug the septum against the base of the column nut.

7 Use a column cutting wafer at 45° to score the column.

8 Snap off the column end. The column may protrude about 1 mm beyond the end of the tool. Inspect the end with a magnifying loupe to make certain that there are no burrs or jagged edges.



9 Remove the column, nut, and swaged ferrule from the tool.

10 Wipe the column walls with a tissue dampened with isopropanol to remove fingerprints and dust.

11 Carefully thread the swaged column into the detector fitting. Finger-tighten the column nut, then use a wrench to tighten an additional 1/8 turn.

Replace the Inner Ceramic Tube (SCD)

To replace the inner ceramic tube:

WARNING **The oven, inlets, and detectors can be hot enough to cause burns. Cool these areas to a safe handling temperature before beginning.**

CAUTION Wear clean, lint-free gloves to prevent contamination of parts with dirt and skin oils.

CAUTION Most steps in this procedure require the use of two wrenches, one to hold the burner steady and the other to loosen a part. Always use two wrenches to avoid over-torquing or bending the burner assembly.

1 Gather the following:
   - Two 7/16-inch open-end wrenches
   - 3/8-inch open-end wrench
   - New O-ring
   - New ceramic tube
   - Tweezers
   - 1/8-inch cap for transfer line
   - T20 Torx driver
2 Cool the GC oven, inlet, and other heated zones to a safe handling temperature (< 40 °C).
3 Prepare the detector for maintenance.
   a Load the maintenance method and wait for the detector to become ready. (See "Detector Maintenance Method" on page 60.) Wait until the detectors, burner assembly, and detector base cool to a safe handling temperature (< 40 °C).
   b Turn off all hydrogen flows. (Leave on the oxidizer and ozone supply gases.)
   c Turn off the ozone generator.

WARNING **Hydrogen gas is flammable. Turn off all detector (and column) hydrogen gas flows before performing maintenance on the detector.**

4 Disconnect the transfer line and quickly cover the open end with the 1/8-inch cap. Use a 3/8-inch wrench on the transfer line and a 7/16-inch wrench on the upper fitting to hold the burner assembly steady.

5 Using two 7/16-inch wrenches, remove the outlet fitting from the upper fitting.

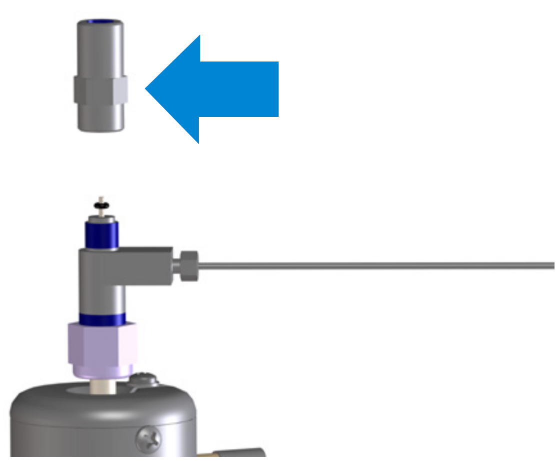
6 If the old O-ring is stuck to the bottom of the outlet fitting, use tweezers or similar to gently pry it loose from the fitting.

7 Remove the old inner ceramic tube.

8 Place a new O-ring over the end of the new inner ceramic tube, and slide the O-ring about 7 mm down the tube. (This dimension is not critical.)

9 Gently insert the tube and O-ring assembly into the burner until it rests on the O-ring.

10 Orient the outlet fitting so the hex flats are closer to the upper fitting, as shown, and install over the ceramic tube. Tightening the outlet fitting will automatically adjust the O-ring and ceramic tube positions. Tighten until snug (finger-tight). Do not overtighten.



11 Reinstall the transfer line onto the outlet fitting. Tighten until snug (finger-tight). Do not overtighten.

12 Restore the detector gas flows.

13 Check for leaks at the upper hydrogen fitting. Correct a leak as needed.

14 Restore the remaining detector operating conditions.

Replace the Quartz Tube (NCD)

To replace the NCD quartz tube:

WARNING **The oven, inlets, and detectors can be hot enough to cause burns. Cool these areas to a safe handling temperature before beginning.**

CAUTION Wear clean, lint-free gloves to prevent contamination of parts with dirt and skin oils.

CAUTION Most steps in this procedure require the use of two wrenches, one to hold the burner steady and the other to loosen a part. Always use two wrenches to avoid over-torquing or bending the burner assembly.

1 Gather the following:
   - Two 7/16-inch open-end wrenches
   - 3/8-inch open-end wrench
   - 5/8-inch open-end wrench
   - New quartz tube
   - Tweezers
   - 1/8-inch cap for transfer line
   - T20 Torx driver
   - Dental tool or similar tool for graphite ferrule removal
   - 2 New graphite ferrules
2 Cool the GC oven, inlet, and other heated zones to a safe handling temperature (< 40 °C).
3 Prepare the detector for maintenance.
   a Load the GC maintenance method and wait for the GC to become ready. (See “Detector Maintenance Method” on page 60.) Wait until the detectors, burner assembly, and detector base cool to a safe handling temperature (< 40 °C).
   b Turn off all hydrogen flows. (Leave on the oxidizer and ozone supply gases.)

c Turn off the ozone generator.

WARNING **Hydrogen gas is flammable. Turn off all detector (and column) hydrogen gas flows before performing maintenance on the detector.**

4 Remove the protective shroud. Remove the two T20 Torx screws, twist the shroud counter-clockwise to remove it from the mounting posts, then lift. Set the shroud and screws aside for later use.

5 Disconnect the transfer line and quickly cover the open end with the 1/8-inch cap. Use a 3/8-inch wrench on the transfer line and a 7/16-inch wrench of the upper fitting to hold the burner assembly steady.

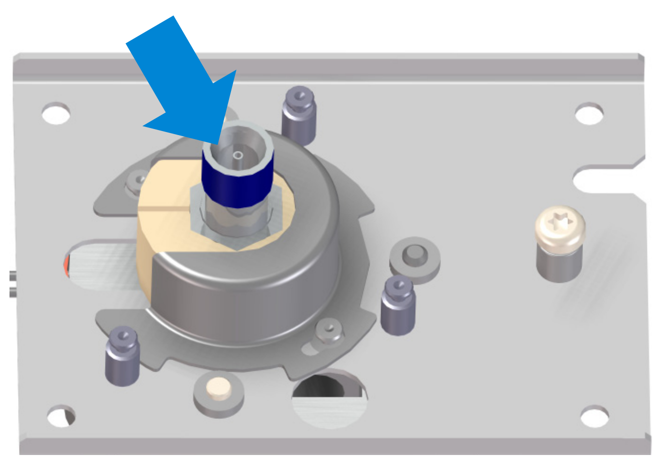
6 Using two 7/16-inch wrenches, remove the outlet fitting from the nut on the top of the quartz tube.

7 Gently slide the nut and its ferrule up and off of the quartz tube.

CAUTION The quartz tube is fragile and can be chipped or cracked. To avoid damaging the quartz tubes, handle carefully.

8 Using 5/8-inch and 9/16-inch wrenches, remove the burner assembly and tube from the coupling in the detector base assembly.

9 Inspect the area around the jet in the coupling. If there are broken bits of tube present, remove with tweezers or similar tool.

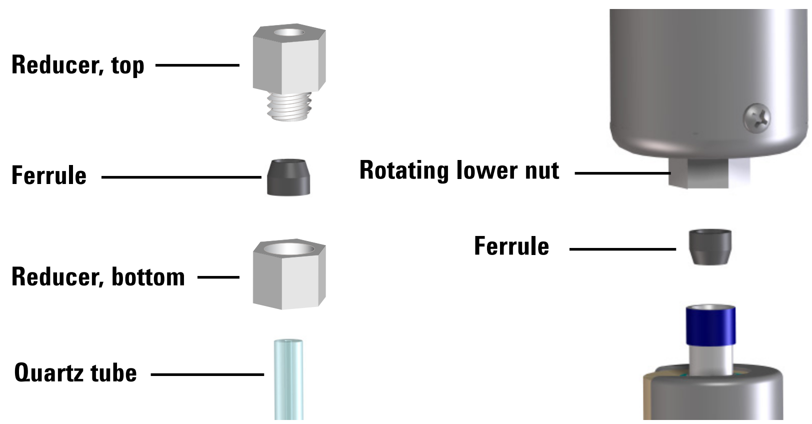


10 Gently pull the quartz tube up through the burner assembly to remove. The graphite ferrule should remain in the rotating nut in the burner base.

11 Use a dental tool or similar to remove the old graphite ferrule from the rotating nut in the burner base.

12 Use two wrenches to disassemble the reducer, then remove the old ferrule.

13 Install new graphite ferrules. In both cases, the tapered end of the ferrule faces out, away from the burner.



14 Reassemble the reducer. Tighten with two wrenches until snug.

15 Slide the new quartz tube down through the burner assembly until it protrudes from the base about 1 cm. (This dimension is not critical. The tube position will adjust as you tighten the lower nut onto the coupling.)

CAUTION When tightening the graphite ferrules onto a quartz tube, tighten only until snug. Over tightening can damage the ferrules or quartz tubes.

16 Carefully lower the burner assembly onto the detector base and thread the nut onto the detector base assembly by hand. Tighten finger-tight, then snug in place using a wrench. Do not overtighten.

17 Place the nut and ferrule over the open end of the quartz tube so that the open end of the nut faces up.

18 Install the nut into the outlet fitting and tighten with two wrenches just until snug.

19 Reinstall the transfer line onto the outlet fitting. Tighten until snug (finger-tight). Do not overtighten.

20 Reinstall the protective shroud.

21 Restore the detector operating conditions.

Check the Vacuum Pump Oil

CAUTION Never add or replace the foreline pump oil while the pump is on.

Check the level and color of the pump oil weekly.

1 Check the oil level in the window of the foreline pump. The oil level should be between the marks for Max and Min.

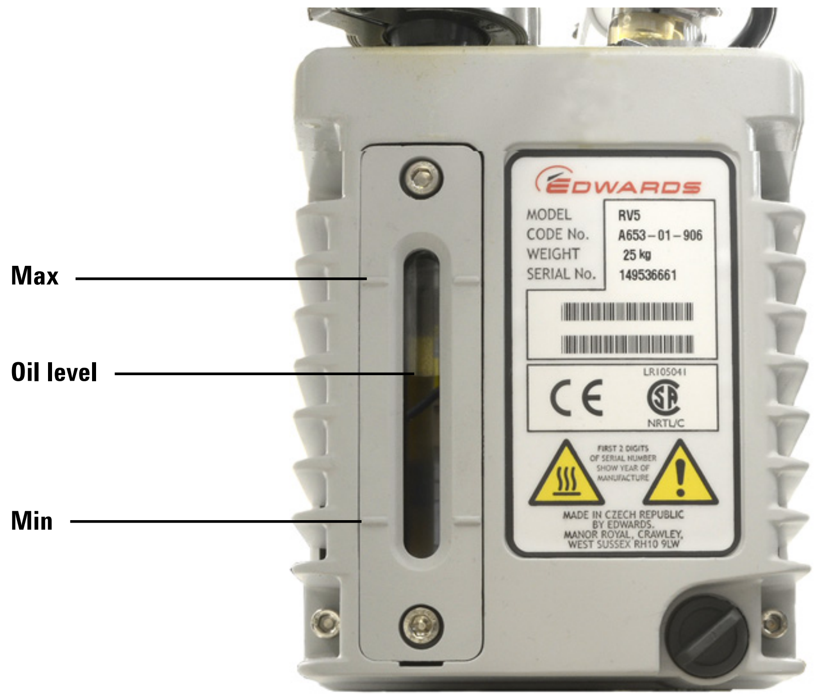


Figure 12 Checking the oil level

2 Check that the color of the pump oil is clear or almost clear with few suspended particles. If the pump oil is dark or full of suspended particles, replace it.

3 Record the maintenance in the maintenance logbook.

Add Vacuum Pump Oil

Add pump oil when the pump oil level is low.

Materials needed

- Funnel (9301-6461)
- 5-mm Allen wrench (8710-1838)
- Gloves, chemical resistant, clean, lint free (9300-1751)
- Oil, synthetic, Mobile 1 (G6600-85001)
- Safety glasses (goggles)

WARNING **Never add pump oil while the pump is on.**

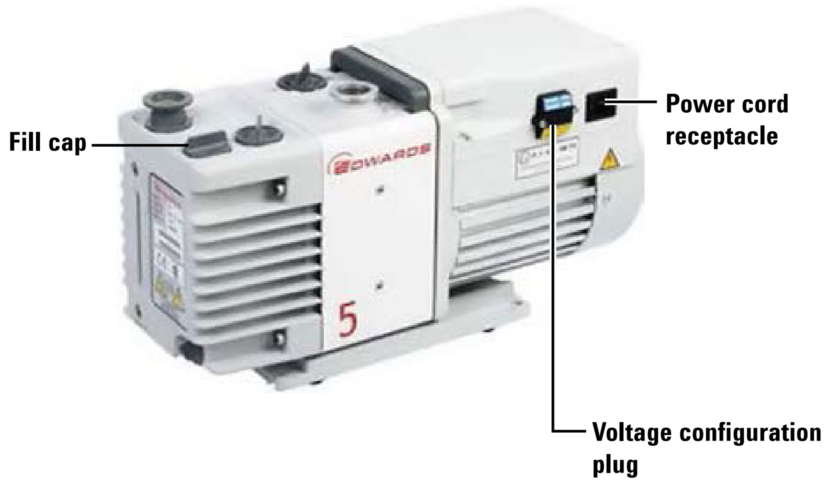
WARNING **The fill cap and pump may be dangerously hot. Check that the fill cup and pump are cool before you touch them.**

CAUTION Use only synthetic 10W30 oil, such as Mobil 1. Any other oil can substantially reduce pump life and invalidates the pump warranty.

Procedure

1. Shut down the detector and wait for the pump to turn off. See “Shutdown” on page 42.
2. Turn off the detector and unplug the pump power cord at the pump.

3 Remove the fill cap on the vacuum pump.



4 Add new pump oil until the oil level is near, but not over the maximum mark beside the oil level window. See Figure 12 on page 71.

5 Reinstall the fill cap.

6 Wipe off all excess oil around and underneath the pump.

7 Reconnect the pump power cord.

8 Turn on the detector and restore operating conditions. See “Start-up” on page 41.

9 Record the maintenance in the maintenance logbook.

Replace the Vacuum Pump Oil

Replace the pump oil every three months or sooner if the oil appears dark or cloudy.

Materials needed

- Container for catching used pump oil
- Funnel (9301-6461), 5-mm Allen wrench (8710-1838)
- Gloves, chemical resistant, clean, lint free (9300-1751)
- Oil, synthetic, Mobile 1 (G6600-85001)
- Safety glasses (goggles)
- Screwdriver, flat-bladed, large (8710-1029)

WARNING **Never add pump oil while the pump is on.**

WARNING **The fill cap and pump may be dangerously hot. Check that the fill cup and pump are cool before you touch them.**

WARNING **Do not touch the oil. The residues from some samples are toxic. Properly dispose of the oil.**

CAUTION Use only synthetic 10W30 oil, such as Mobil 1. Any other oil can substantially reduce pump life and invalidates the pump warranty.

Procedure

1. Shut down the detector and wait for the pump to turn off. See “Shutdown” on page 42.
2. Turn off the detector and unplug the pump power cord at the pump.

3 Place a container under the drain plug of the vacuum pump.

4 Remove the fill cap, then open the drain plug. Drain the oil completely by raising the motor end of the pump.

5 Reinstall the drain plug.

6 Add new pump oil until the oil level is near, but not over the maximum mark beside the oil level window. See Figure 12 on page 71.

7 Reinstall the fill cap.

8 Wipe off all excess oil around and underneath the pump.

9 Reconnect the pump power cord.

10 Turn on the detector and restore operating conditions. See "Start-up" on page 41.

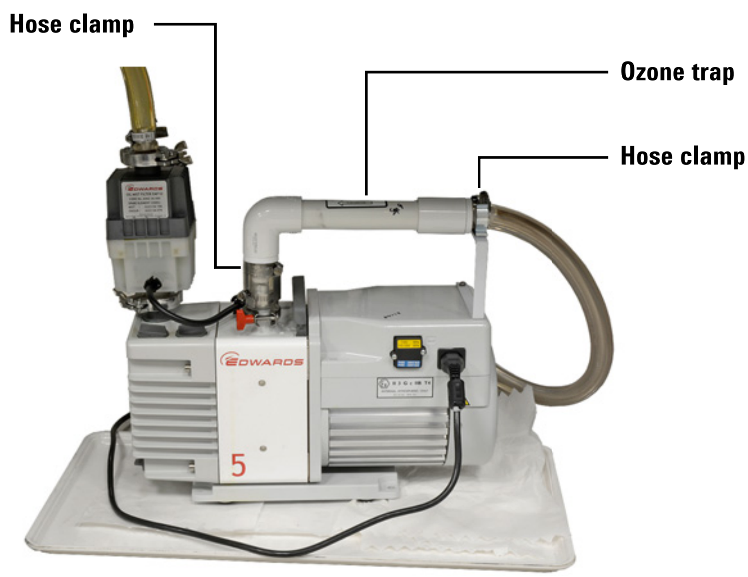
11 Record the maintenance in the maintenance logbook.

12 Check the pump for leaks after about 30 minutes, and check again after 24 hours.

Replace the Ozone Trap

To replace the ozone trap:

1 Load a method to cool the detector, turn off the heaters, and turn off the hydrogen flow.
   - Turn off the heaters and allow the burner to cool.
   - Leave the oxidizer flow on.
   - Turn off the hydrogen flow.
   - Turn off the vacuum pump.
   - Set the GC oven to 30 °C (or off) to minimize column bleed.
   - Leave (helium) carrier gas flowing.
2 Allow the vacuum pump to cool to a safe handling temperature.
3 Remove the trap assembly and vacuum hose from the support bracket.
4 Loosen the two hose clamps that secure the old ozone trap in place.



5 Remove the trap from the pump intake hose. (If needed, loosen the clamp at the pump intake.)
6 Lift the old trap from the support bracket, then remove the detector vacuum hose from the barbed fitting on the old trap.
7 Install the new trap. Make sure that the flow direction arrow on the new trap points towards the intake fitting. (The trap elbow must be nearest the pump intake.) If you removed the short connector hose from the pump intake, reinstall it.

Change the Oil Mist Filter

The oil mist filter on the RV5 pump has two components: the charcoal odor filter and the oil coalescing filter element. To replace the filters, disassemble the oil mist filter assembly with the 4 mm long-handled hex wrench (provided). The smaller charcoal odor filter sits on top of the larger oil coalescing filter element. While it is recommended to replace the oil coalescing filter element after 90 days of continuous use, replacement of the charcoal odor filter is optional. After replacing the filter, re-assemble the filter assembly and attach it to the pump flange.

Clean the Detector Exterior

WARNING **Burn hazard. The burner assembly can be hot enough to cause burns. Before touching, cool to a safe handling temperature (< 40 °C).**

WARNING **Shock hazard. Before cleaning the detector, turn it off and unplug its power cord.**

Before cleaning the detector, shut it down, turn it off, and unplug the detector power cord. Clean the detector with a **damp** cloth using water. Do not spray liquids directly onto the detector. Wipe dry with a clean, soft cloth. Do not allow cleaning fluids to drip into the detector or GC because liquids can damage the detector or GC electronics.

Do not use cleaning agents on the burner assembly that could cause a hazard on the burner.

Calibrate the Flow and Pressure Sensors

The 8355 S SCD and 8255 S NCD use electronic pressure control modules. Typically, set the detector to use automatic flow zeroing. See "Configure auto flow zero" on page 43. Calibration is generally not required. However, if needed the flow and pressure sensors can be manually zeroed. See "Zero a specific flow or pressure sensor" on page 44 for details.

6
Troubleshooting

This chapter describes how to troubleshoot and resolve typical issues encountered while using an Agilent 8355 S SCD or 8255 S NCD detector.

Solving Detector Problems

A basic understanding of the detector helps to diagnose and solve detector problems. Review the basic detector theory found in “Theory of Operation” on page 18. Also, please note that this section is intended to troubleshoot problems in a detector that has previously been performing acceptably. If trying to optimize a new detector application, see “Adjusting the Operating Conditions” on page 40 for recommendations on adjusting method setpoints to obtain better results.

Many symptoms may be caused by more than one problem or by poor chromatographic technique. Analysis of sulfur or nitrogen compounds has traditionally been very difficult because of the inherent reactivity and instability of the compounds themselves. Often, problems initially blamed on the detector actually originate from either poor chromatographic technique or other system failures (for example, a leak at the column inlet fitting). Therefore, the first step in troubleshooting is to isolate the problem to the GC (inlet, injector, or column), to the burner assembly, or to the detector (ozone generator, vacuum pump, photomultiplier tube, or electronics). When diagnosing a problem in a system that had been working, a good first step is to restore the default typical operating conditions. The response under these conditions can help determine whether or not the method settings are causing the problem.

Table 7 in the next section lists many common problems, their most probable causes, and corrective action that should be taken.

Troubleshooting Table

Table 7 Troubleshooting Detector Issues

Problem	Possible cause	Diagnosis	Corrective action
Detector problems			
No response	No ozone	Little or no difference in output signal between ozone On and Off.	See “No ozone”.
	Incorrect range setting for analog out (standalone detector only)		Edit the range to better scale the data. See “Signal Output” on page 45.
No ozone	High voltage transformer and/or ozone generator inoperative.	No difference in output signal between ozone On and Off even though flow through the ozone generator is normal.	Contact Agilent for service. See “The Service Mode Key” on page 32.
	Restriction in ozone supply to the reactor cell.		Contact Agilent for service.
No Response	Broken ceramic or quartz tube.		Replace the ceramic tube. See “Replace the Inner Ceramic Tube (SCD)” on page 64 or “Replace the Quartz Tube (NCD)” on page 67.
Low Response	Inappropriate hydrogen and oxidizer flow rates.	Check flow rates.	Adjust flow rates.
	Leak in the detector.		Check for leaks in the detector and repair any leaks. See “Leaks” on page 88.
	Contaminated ceramic or quartz tubes.	If there does not appear to be a leak, then inspect the ceramic tube. Contamination can result from column bleed, samples which contain volatile metal complexes, and large injections of coke-forming hydrocarbons.	Replace the ceramic tube. See “Replace the Inner Ceramic Tube (SCD)” on page 64 or “Replace the Quartz Tube (NCD)” on page 67.
	Input voltage does not match configuration plug.		Contact Agilent for service.

Table 7 Troubleshooting Detector Issues

Problem	Possible cause	Diagnosis	Corrective action
Wandering Baseline	Contamination in one of the detector gases.	Check the difference in the output signal between ozone On and Off.	Check inline supply traps and replace. Change detector gases.
Excessive response	Incorrect range setting for analog out (standalone detector only)		Edit the range to better scale the data. See "Signal Output" on page 45.
	Leak in the oxidizer lines.		Check for leaks in the detector and repair any leaks. See "Leaks" on page 88.
	Leak in the hydrogen supply lines.		Check for leaks in the detector and repair any leaks. See "Leaks" on page 88.
Tailing peaks with non-equimolar response	Severe contamination of detector gases.	High background signal compared against ozone Off.	Check inline supply traps and replace. Change detector gases.
Tailing peaks	Poor column connection.	Verify column position at inlet and outlet. Look for discoloration of column at detector fitting which indicates column in combustion zone.	Reinstall column. See "Attach a Column to the Detector" on page 61.
	Cracked tubes.	Confirm pressure and vacuum ranges. Inspect columns and ferrules.	Replace the ceramic tube. See "Replace the Inner Ceramic Tube (SCD)" on page 64 or "Replace the Quartz Tube (NCD)" on page 67.
Detector thermal shutdown	Thermocouple open.		Contact Agilent for service.
Burner pressure exceeds 760 Torr.	Restriction caused by upper ceramic tube inner diameter.	If performing maintenance, complete it. Recheck again during normal operation.	
Burner pressure excessively high.	Cracked quartz or outer ceramic tube. Leaking or disconnected 1/16-inch stainless steel hydrogen or oxidizer line.		Replace the ceramic tube. See "Replace the Inner Ceramic Tube (SCD)" on page 64 or "Replace the Quartz Tube (NCD)" on page 67. Check connection. Check line for leaks. Contact Agilent for service if the line is cracked.

Table 7 Troubleshooting Detector Issues

Problem	Possible cause	Diagnosis	Corrective action
	Leak in burner.		Check for leaks in the detector and repair any leaks. See "Leaks" on page 88.
Burner pressure lower than expected and poor response	Cracked or misaligned inner ceramic tube.	Check that the inner ceramic tube is positioned correctly and has not dropped down into the outer tube.	See "Replace the Inner Ceramic Tube (SCD)" on page 64.

Table 8 Troubleshooting Pump Issues

Problem	Possible Cause	Diagnosis	Corrective Action
Vacuum pump problems			
Pump does not start	Pump switch off or power cord disconnected.		Turn On pump power switch. Check pump power cord.
Fuses blow on startup	Emulsified oil.	Inspect oil for integrity.	Change pump oil, and plug unit into wall to run for 10–15 minutes. Contact Agilent to replace blown fuses.
Water in pump	Cracked coalescing filter.	Milky yellow oil in the pump window.	Change coalescing filter and pump oil.
Reaction cell pressure high	Ozone destruction trap clogged.	Remove ozone destruction trap from the vacuum line and re-check expected pressure readings.	Change chemical trap.
	Burner disconnected from reaction cell.	Check connections.	
	Vacuum pump defective.		Replace vacuum pump.
Pump loses oil gurgle sound	Ballast open.	Oil level drops.	Reset ballast. See pump-specific sections.
High level of oil in coalescing filter	Plugged oil return restrictor.	No visible movement of oil in the return line.	Change filter and clear restrictor.

Status Indicator LED

Use the detector status LED to quickly determine the status and readiness of the detector. The LED changes color depending on the current state of the detector.

- **Green**: Indicates that power is available for the heaters, chiller (NCD), vacuum pump, and ozone generator.
- **Yellow**: Indicates that the detector is not ready for operation. Power is on and available, but not all parameters have reached operating setpoints. A warning or other message may exist.
- **Red**: Indicates a fault or other serious condition. A fault or other message may exist. The detector cannot be used until the fault condition is resolved.

Detector Messages

Check the detector status display for detector messages. The detector keypad will display and status and error messages that occur during operation, as well as log detector maintenance and error messages in the detector log files.

Leaks

Ozone leaks

WARNING **Ozone is a hazardous gas and a strong oxidant. Exposure to ozone should be minimized by using the instrument in a well-ventilated area and by venting the exhaust of the vacuum pump to a fume hood. The ozone generator should be turned off when the instrument is not in use.**

If you suspect an ozone leak, shut down the detector. Do not open the detector mainframe. Contact Agilent for service.

Hydrogen leaks

WARNING **Do not measure hydrogen together with air or oxygen. This can create explosive mixtures that may be ignited by the high burner temperature. To avoid this hazard: 1. Cool the burner before you begin. 2. Always measure gases separately.**

Check all external connections for leaks. See "Checking for hydrogen and oxidizer leaks" on page 89. Check the supply connections to the detector mainframe and between the detector mainframe and the burner assembly. If you suspect a leak inside the detector mainframe, contact Agilent for service. Do not open the detector mainframe.

Oxidizer leaks

WARNING **Oxygen rich environments can promote combustion and even result in spontaneous combustion under conditions of high pressure and exposure to contamination. Use only oxygen rated components and ensure that components are oxygen clean prior to use with pure oxygen.**

Check the oxidizer supply connections to the detector mainframe and between the detector mainframe and the burner assembly. See "Checking for hydrogen and oxidizer leaks" on

page 89. If you suspect a leak inside the detector mainframe, contact Agilent for service. Do not open the detector mainframe.

Checking for hydrogen and oxidizer leaks

To check for a leak in the hydrogen or oxidizer flow paths, do the following:

1. Check all external fittings for leaks. Correct any leaks (tighten or remake connections as appropriate).
2. If you still suspect a leak, establish the typical flow checkout conditions (see Table 10 on page 100 for SCD or Table 11 on page 106 for NCD).
3. Maintain these conditions for several minutes. If the detector cannot maintain these flow rates, contact Agilent for service.
4. If the detector was able to maintain the flow rates, turn off all gas flows using the detector keypad.
5. Monitor the pressure readings on the detector display. (Press [**Det**].) With the vacuum pump running, the reaction cell pressure should fall to approximately 0 (zero). The burner pressure should fall to a value considerably lower than the typical operating pressure. This process will take time due to the internal configuration of the burner assembly. If the burner pressure remains high or at normal pressures, contact Agilent for service.

Power Problems

The power supplied to the SCD/NCD heaters, NCD chiller, vacuum pump, and ozone generator comes from the detector mainframe and is controlled by the detector power switch.

No power

If the detector does not appear to have power—if the vacuum pump does not run and the heaters will not turn on—check the following:

- Check that the power switch is on.
- Check that the power cord is properly connected.
- Check the building power supply.

If the cord if connected properly, and the building circuit for the detector is operating normally, contact Agilent.

Ozone Generation Problems

Before troubleshooting the ozone generator, first verify that the other components of the system operate normally. For example, check for leaks in the detector external connections, check for leaks in the inlet and inlet column fitting, check that the vacuum pump operates normally, check that the inlet and ALS are operating normally, and so on.

Troubleshoot ozone generation as follows:

1 On the display, note the detector output signal.
2 Leave the vacuum pump on and ozone supply gas flowing.
3 Turn off the ozone generator.
4 Observe the detector output signal.
5 Turn on the ozone generator and check the detector output signal again.

A properly operating detector will typically display a difference in background signal between ozone supply gas voltage on and off. If no change is observed, contact Agilent service.

Coking

Contamination from some sample matrices can reduce sensitivity. For example, crude oils containing volatile metal complexes may contaminate the ceramic tubes. In addition, incomplete combustion of certain hydrocarbon-containing compounds leaves behind coke deposits on the ceramic tubes. Coke deposits may be removed from the burner by reducing the hydrogen flow rate.

Hydrogen Poisoning

Hydrogen poisoning of the SCD ceramic tubes can occur when the relative oxidizer flow is very much lower than the hydrogen flow. Whether this state occurs due to inappropriate method setpoints or due to a problem with the oxidizer flow, this condition results in extremely reduced or no response. If you suspect hydrogen poisoning:

- Check for and resolve any flow shutdown.
- Check for restrictions in the oxidizer supply line to the burner assembly.
- Load the checkout method or other method that uses more balanced relative flow rates.

If the response does not recover, replace the inner ceramic tube. If response still does not recover, replace the outer ceramic tube. The ceramic tubes cannot be reconditioned.

Contaminated Gases

Agilent recommends the use of clean gases that meet the requirements in the *Site Preparation Guide*. In addition, Agilent highly recommends the use of high quality traps to eliminate as much possible contamination as possible. The use of clean gases is essential for optimal performance. Otherwise, sulfur and other contaminants from gases may accumulate in the column and bleed out over time, desensitizing the ceramic tubes and causing elevated baselines.

Moisture in the ozone generator supply line can lead to the formation of acids that can damage or destroy the ozone generator and other detector components. Agilent highly recommends the use of a high-quality moisture trap, such as the Gas Clean Filter System with a moisture trap, for the ozone supply gas. See "Consumables and Replacement Parts" on page 56.

7
Performance Verification

This chapter describes how to verify that the detector is operating normally.

About Chromatographic Checkout

The tests described in this section provide basic confirmation that the detector can perform comparably to factory condition. However, as detectors and the other parts of the GC age, detector performance can change. The results presented here represent typical outputs for typical operating conditions and are not specifications.

The tests assume the following:

- Use of an automatic liquid sampler. If not available, use a suitable manual syringe instead of the syringe listed.
- Use of a 10-µL syringe in most cases. However, a 5-µL syringe is an acceptable substitute.
- Use of the septa and other hardware (liners, adapters, and so forth) described. If you substitute other hardware, performance can vary.

Prepare for Chromatographic Checkout

Because of the differences in chromatographic performance associated with different consumables, Agilent strongly recommends using the parts listed here for all checkout tests. Agilent also recommends installing new consumable parts whenever the quality of the installed ones is not known. For example, installing a new liner and septum ensures that they will not contribute any contamination to the results.

NOTE The parts listed here apply to Agilent GCs. For other GC types, match part properties as appropriate.

1 Check the indicators/dates on the gas supply traps. Replace expended traps.
2 Install new consumable parts for the inlet and prepare the correct injector syringe (and needle, as needed).

Table 9 Recommended parts for checkout by inlet type

Recommended part for checkout	Part number
Split splitless inlet	
Syringe, 10-µL	5181-1267
Liner O-ring	5188-5365
Septum, bleed and temperature optimized (BTO), non-stick	5183-4757
Liner, Ultra Inert, splitless, single taper, glass wool	5190-2293
Gold plated inlet seal, with washer	5188-5367
Multimode inlet	
Syringe, 10-µL	5181-1267
Liner O-ring	5188-5365
Septum	5183-4757
Liner, Ultra Inert, splitless, single taper, glass wool	5190-2293
Cool On-column inlet	
Septum	5183-4758
Septum nut	19245-80521
Syringe, 5-µL on-column	5182-0836

Table 9 Recommended parts for checkout by inlet type (continued)

Recommended part for checkout	Part number
0.32-mm needle for 5-µL syringe	5182-0831
7693A ALS: Needle support insert, COC	G4513-40529
7683B ALS: Needle support assembly for 0.25/0.32 mm injections	G2913-60977
Insert, fused silica, 0.32-mm id	19245-20525

Prepare sample vials

Performance verification requires a 1 uL injection.

WARNING **When handling checkout standards, always follow the handling precautions recommended with its packaging.**

1. Open the sample box.
2. Snap the top off of one checkout sample ampoule.
3. Transfer the contents to a 2-mL ALS sample vial and cap the vial.

Check SCD Performance

1 Gather the following:
   - Evaluation column, DB-1 30 m × 0.32 mm × 1.0 µm (part number 123-1033)
   - SCD performance evaluation (checkout) sample (5190-7003): 0.7 ± 0.002 mg/L diethyl disulfide and 1.0 ± 0.003 mg/L tert-butyldisulfide in isooctane.
   - Chromatographic-grade isooctane
   - 4-mL solvent and waste bottles or equivalent for autoinjector
   - 2-mL sample vials or equivalent for sample
   - Inlet and injector hardware (See “Prepare for Chromatographic Checkout” on page 97.)
2 Verify the following:
   - Chromatographic-grade gases plumbed and configured: helium as carrier gas, air as oxidizer, and oxygen as ozone supply gas.
   - Ozone supply gas moisture trap and other traps are in date.
   - Empty waste vials loaded in sample turret.
   - 4-mL solvent vial with diffusion cap filled with isooctane and inserted in Solvent A injector position.
   - 4-mL solvent vial with diffusion cap filled with isooctane and inserted in Solvent B injector position.
3 Replace consumable parts (liner, septum, traps, syringe, and so forth) as needed for the checkout. See “Prepare for Chromatographic Checkout” on page 97.
4 Install the evaluation column. (See the procedure for in the GC *Maintaining Your GC* manual.)
   - Bake out the evaluation column for at least 30 minutes at 180 °C. (See the procedure for in the GC *Maintaining Your GC* manual.)
   - Configure the column.
5 Check the detector baseline output. The output should be below 150 pA and relatively stable, assuming a well-stabilized system with the column oven at 50 °C.

   However, a new burner (or a burner with a new ceramic tube) can have a very high baseline after first ignition. In this case, the baseline should gradually decrease, depending on burner cleanliness. Noise will also greatly decrease over time. For a well-stabilized system, noise measured by Agilent OpenLAB CDS should be approximately 5 display units or less.

Checkout can continue before the baseline becomes completely stable.

6 Set the analog range to 9, if using an AIB. If using an Agilent 35900E A to D set to 10, or set to 12 if testing for linearity. This starting point may require adjustment.

7 Create or load a method with the parameter values listed in Table 10.

Table 10 SCD Checkout conditions

Column and sample	
Type	DB-1, 30 m × 0.32 mm × 1 µm (123-1033)
Sample	SCD checkout 5190-7003
Column flow	2 mL/min helium
Column mode	Constant flow
Split/splitless inlet	
Temperature	250 °C
Mode	Splitless
Purge flow	40 mL/min
Purge time	0.7 min
Septum purge	3 mL/min
Gas saver	Off
Multimode inlet	
Mode	Splitless
Inlet temperature	250 °C
Purge time	0.7 min
Purge flow	80 mL/min
Septum purge	3 mL/min
Gas saver	Off
Cool on-column inlet	
Temperature	Oven Track

Table 10 SCD Checkout conditions (continued)

Septum purge	15 mL/min
Detector	
Base temperature	280°C
Burner temperature	800 °C
Upper H_2 flow	38 mL/min
Lower H_2 flow	8 mL/min
Oxidizer flow	50 mL/min, Air
O3 Generator flow	On
O3 Generator power	On
Vacuum pump	On
FID-SCD Tandem settings	
FID temp	350 °C
FID hydrogen flow	35 mL/min
FID air flow	300 mL/min
FID N_2 makeup flow	20 mL/min
SCD oxidizer flow	5 mL/min, Air
SCD upper H_2 flow	40 mL/min
Lower H_2 flow	Not applicable for FID-SCD
Oven	
Initial temp	50 °C
Initial time	3.0 min
Rate 1	25 °C/min
Ramp 1 temp	160 °C
Ramp 1 hold time	2 min
Post run temp	50 °C
ALS settings (if installed)	
Sample washes	2
Sample pumps	6
Sample wash volume	8 µL (maximum)
Injection volume	1 µL
Syringe size	10 µL
Solvent A pre washes	0

Table 10 SCD Checkout conditions (continued)

Solvent A post washes	3
Solvent A wash volume	8 µL (maximum)
Solvent B pre washes	0
Solvent B post washes	3
Solvent B wash volume	8 µL (maximum)
Injection mode (7693A)	Normal
Airgap Volume (7693A)	0
Viscosity delay	0
Solvent Wash Draw Speed (7693A)	150
Solvent Wash Dispense Speed (7693A)	1500
Sample Wash Draw Speed (7693A)	150
Sample Wash Dispense Speed (7693A)	1500
Inject Dispense Speed (7693A)	3000
Plunger speed (7683)	Fast, for all inlets except COC.
PreInjection dwell	0
PostInjection dwell	0
Manual injection	
Injection volume	1 µL
Data system	
Data rate	5 Hz (Front detector, SCD)

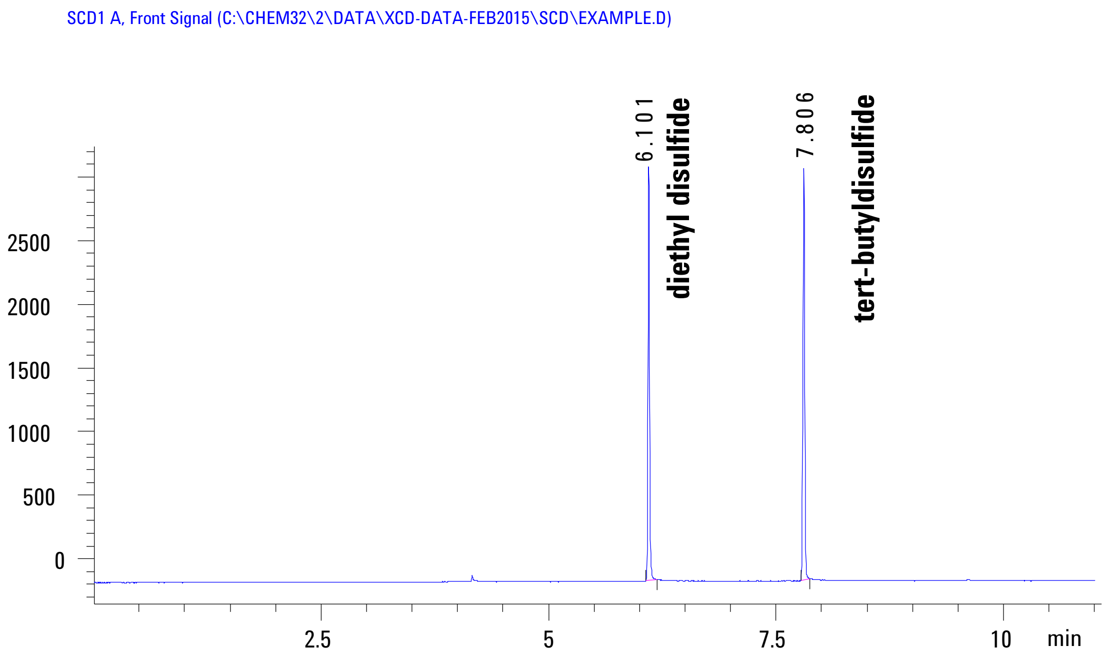
8 If using a data system, prepare the data system to perform one run using the loaded checkout method. Make sure that the data system will output a chromatogram. If not using a data system, create a one sample sequence using the GC keypad.

9 Start the run. If performing an injection using an autosampler, start the run using the data system or press **[Start]** on the GC. If performing a manual injection (with or without a data system):

   a Press **[Prep Run]** to prepare the inlet for splitless injection.

   b When the GC becomes ready, inject 1 µL of the checkout sample and press **[Start]** on the GC.

The following chromatogram shows typical results for a new detector with new consumable parts installed. Note that the response for a tandem FID-SCD installation will be approximately 1/10 of the response shown in this example due to the reduced amount of sample that reaches the SCD.



For a new installation, calculate the MDL of sulfur for the detector. Calculate MDL as:

$$\text{MDL} = \frac{2 \text{ x noise}}{\text{sensitivity}}$$

where

noise is the ASTM noise calculated by the Agilent data system

Sensitivity is calculated as:

$$\text{Sensitivity} = \frac{\text{peak area}}{\text{amount}}$$

Based on the 1 µL injection of the checkout standard, typical results of the MDL calculations are shown in the table below.

Compound	Amount injected (mg/L)	Sulfur content	S amount injected (pg/µL)	Typical ASTM noise	Typical Sensitivity (Area/pg*s)	Typical MDL (pg S/µL)
Diethyl disulfide	0.700	52.50 %	367.500	2.453	9.504	0.469
tert-Butyldisulfide	1.000	36.00 %	360.000	2.453	10.993	0.446

Check NCD Performance

1 Gather the following:
   - Evaluation column, HP-5 30 m × 0.32 mm × 0.25 µm (part number 19091J-413)
   - NCD performance evaluation (checkout) sample (5190-7002): 3-methylindole 10.0 ± 0.1 mg/L, 9-methylcarbazole 14.1 ± 0.1 mg/L, and nitrobenzene 9.51 ± 0.05 mg/L in isooctane.
   - Chromatographic-grade isooctane
   - 4-mL solvent and waste bottles or equivalent for autoinjector
   - 2-mL sample vials or equivalent for sample
   - Inlet and injector hardware (See "Prepare for Chromatographic Checkout" on page 97.)

2 Verify the following:
   - Chromatographic-grade gases plumbed and configured: helium as carrier gas, oxygen as oxidizer and ozone supply gas.
   - Ozone supply gas moisture trap and other traps are in date.
   - Empty waste vials loaded in sample turret.
   - 4-mL solvent vial with diffusion cap filled with isooctane and inserted in Solvent A injector position.
   - 4-mL solvent vial with diffusion cap filled with isooctane and inserted in Solvent B injector position.

3 Replace consumable parts (liner, septum, traps, syringe, and so forth) as needed for the checkout. See "Prepare for Chromatographic Checkout" on page 97.

4 Install the evaluation column. (See the procedure for in the GC *Maintaining Your GC* manual.)
   - Bake out the evaluation column for at least 30 minutes at 270 °C. (See the procedure for in the GC *Maintaining Your GC* manual.)
   - Configure the column.

5 Check the detector baseline output. The output should be below 150 pA and relatively stable, assuming a well-stabilized system with the column oven at 50 °C. (A negative baseline can be acceptable.).

However, a new burner (or a burner with a new quartz tube) can have a very high baseline after first ignition. In this case, the baseline should gradually decrease, depending on burner cleanliness. Noise will also greatly decrease over time. For a well-stabilized system, noise measured by Agilent OpenLAB CDS should be approximately 4 display units or less.

Checkout can continue before the baseline becomes completely stable.

6 Set the analog range to 9, if using an AIB. If using an Agilent 35900E A to D set to 10, or set to 12 if testing for linearity. This starting point may require adjustment.

7 Create or load a method with the parameter values listed in Table 11.

Table 11 NCD Checkout conditions

Column and sample	
Type	HP-5, 30 m × 0.32 mm × 0.25 µm (19091J-413)
Sample	NCD checkout 5190-7002
Column flow	2.2 mL/min
Column mode	Constant flow
Split/splitless inlet	
Temperature	250 °C
Mode	Splitless
Purge flow	80 mL/min
Purge time	0.8 min
Septum purge	3 mL/min
Gas saver	Off
Multimode inlet	
Mode	Splitless
Inlet temperature	250 °C
Initial time	0 min
Purge time	0.8 min
Purge flow	80 mL/min
Septum purge	3 mL/min
Gas saver	Off

Table 11 NCD Checkout conditions (continued)

Cool on-column inlet	
Temperature	Oven Track
Septum purge	15 mL/min
Detector	
Base temperature	280°C
Burner temperature	900 °C
Chiller temperature	On
H_2 flow	3 mL/min
Oxidizer flow	8 mL/min, Oxygen
O3 Generator flow	On
O3 Generator power	On
Vacuum pump	On
Oven	
Initial temp	50 °C
Initial time	3.0 min
Rate 1	25 °C/min
Ramp 1 temp	250 °C
Ramp 1 hold time	2 min
Post run temp	50 °C
ALS settings (if installed)	
Sample washes	2
Sample pumps	6
Sample wash volume	8 µL (maximum)
Injection volume	1 µL
Syringe size	10 µL
Solvent A pre washes	0
Solvent A post washes	3
Solvent A wash volume	8 µL (maximum)
Solvent B pre washes	0
Solvent B post washes	3
Solvent B wash volume	8 µL (maximum)
Injection mode (7693A)	Normal

Table 11 NCD Checkout conditions (continued)

Airgap Volume (7693A)	0
Viscosity delay	0
Solvent Wash Draw Speed (7693A)	150
Solvent Wash Dispense Speed (7693A)	1500
Sample Wash Draw Speed (7693A)	150
Sample Wash Dispense Speed (7693A)	1500
Inject Dispense Speed (7693A)	3000
Plunger speed (7683)	Fast, for all inlets except COC.
PreInjection dwell	0
PostInjection dwell	0
Manual injection	
Injection volume	1 µL
Data system	
Data rate	5 Hz (Front detector, NCD)

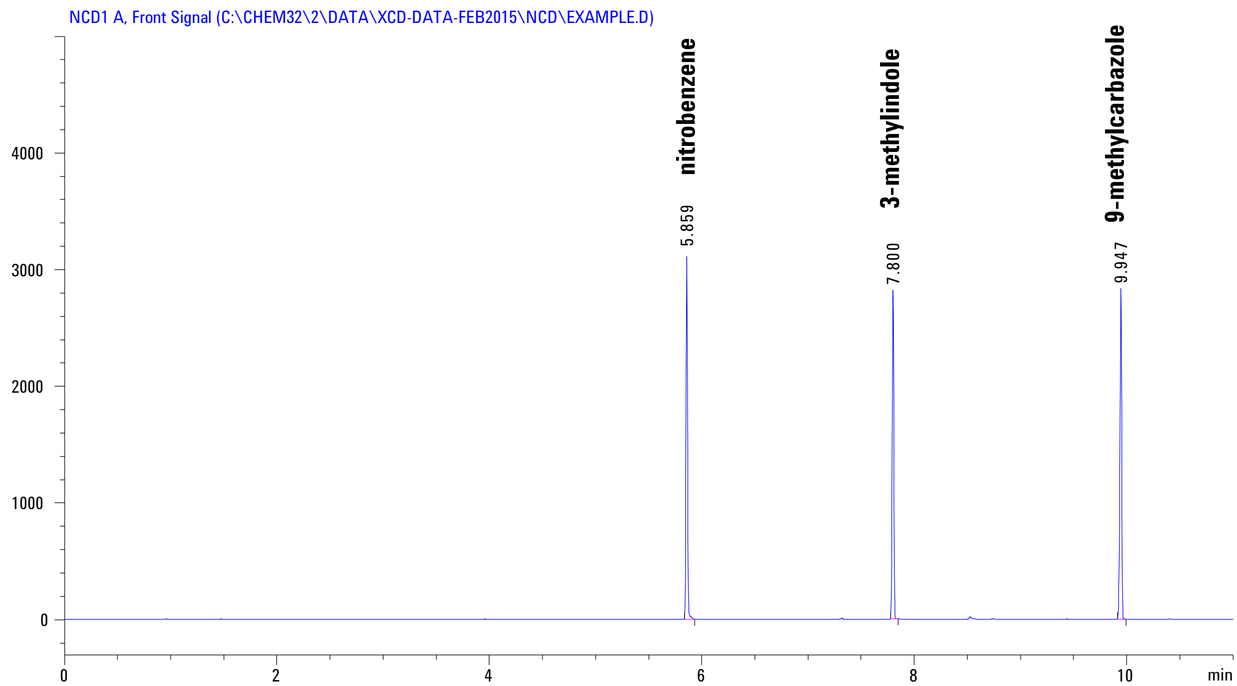
8 If using a data system, prepare the data system to perform one run using the loaded checkout method. Make sure that the data system will output a chromatogram. If not using a data system, create a one sample sequence using the GC keypad.

9 Start the run. If performing an injection using an autosampler, start the run using the data system or press **[Start]** on the GC.If performing a manual injection (with or without a data system):

   a Press **[Prep Run]** to prepare the inlet for splitless injection.

   b When the GC becomes ready, inject 1 µL of the checkout sample and press **[Start]** on the GC.

The following chromatogram shows typical results for a new detector with new consumable parts installed.



For a new installation, calculate the MDL of nitrogen for the detector. Calculate MDL as:

$$\text{MDL} = \frac{\text{2 x noise}}{\text{sensitivity}}$$

where

noise is the ASTM noise calculated by the Agilent data system

Sensitivity is calculated as:

$$\text{Sensitivity} = \frac{\text{peak area}}{\text{amount}}$$

Based on the 1 µL injection of the checkout standard, typical results of the MDL calculations are shown in the table below.

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Compound	Amount injected (mg/L)	Nitrogen content	N amount injected (pg/µL)	Typical ASTM noise	Typical Sensitivity (Area/pg*s)	Typical MDL (pg N/µL)
Nitrobenzene	9.510	11.37 %	1081.29	1.080	2.813	0.7679
3-Methylindole	10.000	10.67 %	1067.00	1.080	2.668	0.810
9-Methylcarbazole	14.100	7.72 %	1088.52	1.080	2.795	0.773

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