SRA Advanced User Guide

Purpose

The primary purpose of this guide is to provide essential information to the onboard laboratory specialist to maintain the instrument. This document introduces characteristics, site preparation, and maintenance procedures for the SRA. Instrument set-up and operation procedures are presented in the SR Analyzer User Guide.

Specifications & Installation

Specifications

- Thermal extraction and pyrolysis parameters
- Temperature: 100°C–850°C
- Heating rates: 0.1°–50°C/min
- Detector: 200°C–400°C
- Oven: special high-temperature tungsten alloy (with four heating units)
- Detectors
- Agilent Technologies FID with gas split
- Dual NDIR with sapphire protection
- Sample handling (autosampler)
- Sampler: Agilent autosampler
- Quantity: 100 samples
- Crucibles: 316 SS
- Volume: up to 200 mg (0.40 cm³)
- Instrument dimensions
- Height: 25 in.
- Width: 16 in.
- Depth: 19 in.
- Instrument weight: 56 lb
- Power requirements
- Voltage: 110 VAC
- Frequency: 50/60 Hz
- Current: 15 A
- Software: Humble Instruments Thermal Station acquisition and processor
- Instrument interface: USB v1.1

Gases

- Helium
  - Purpose: carrier gas
  - Quality: GC analytical grade, 99.9995% purity
  - Pressure: regulated 60–80 psi (4–5.5 bars) stable
  - Connection: 1/8 in. Swagelok male compression fitting
  - Consumption: ~100 mL/min
- Hydrogen
  - Purpose: FID fuel gas
  - Quality: HC analysis grade, 99.9995% purity
  - Pressure: regulated 60–80 psi (4–5.5 bars) stable
  - Connection: 1/8 in. Swagelok male compression fitting
  - Consumption: ~60 mL/min
- Air
  - Purpose: FID fuel gas and oxidation gas
  - Quality: HC analysis grade, zero-grade/CO₂ free or better purity
  - Pressure: regulated 60–80 psi (4–5.5 bars) stable
  - Connection: 1/8 in. Swagelok male compression fitting
  - Consumption: ~300 (400) mL/min
- Air or nitrogen
Purpose: oven cooling gas
Quality: dry and oil free
Pressure: regulated 80–100 psi (4–6.8 bars) stable
Connection: 1/4 in. Swagelok male compression fitting
Consumption: ~240 mL/min. Max. = 2 standard ft³/min) intermittently used

Instrument Components

The SRA system consists of the following main components (Figure 1):

- Autosampler
- Main control unit: oven and detector temperature control unit and gas flow controllers for the FID combustion gas, carrier gas, and oxidation gas
- Infrared (IR) section
- Combustion with gas separation and FID (pedestal, oven, and conversion FID)

Figure 1. Main Components of the SRA System.

Autosampler Unit
The Agilent autosampler can handle up to 100 samples (Figure 2). The tray is divided into quarter trays that hold 25 samples each. Each sample hole is numbered; these numbers must match the weighing container numbers. Confirm the relationship between the autosampler and pedestal (see Autosampler Tray Arm Adjustment) and ensure the autosampler is connected to the main control unit with the serial cable connector (Figure 4).

![Figure 2. Autosampler Tray.](image)

![Figure 4. Serial Cable Connector to the Main Control Unit.](image)

**Main Control Unit**

Gas flow controllers and temperature control units are located inside the main control unit (Figure 5). Gas flow procedures and a flow circuit diagram are available in the Flow Control & Calibration section.

**Temperature Control Units**

1. Oven temperature control unit: WATLOW 96 with cables
2. FID temperature control unit: WATLOW 96 with cables

**Gas Flow Controllers**

- H₂: Hydrogen gas flow controller
- AIR (FID): Air gas flow controller
- CC: Carrier gas flow controller
- CS (above CC): Carrier gas flow controller
Rear Panel

Four gas connections are available on the rear panel (Figure 6). Each tube is a different color and/or size to differentiate the gases:

- Green: Helium
- Red: Hydrogen
- Blue (narrow): Air
- Blue (thick): Cooling Air

White numbers on the connections indicate the appropriate flow rate for the different gases. The four gas tubes connect to the gas ports on this panel. To adjust gas flows, review the Flow Control & Calibration section.

IR Section
The IR section (Figure 7, Figure 8) contains CO and CO₂ detectors in series, pump, tubing, and power supply. The pump pulls a split of sample gases from the oven to the IR absorbance detectors. A pressure controller in the IR unit maintains a constant gas pressure between the oven and IR detectors. The CO and CO₂ detectors function as infrared detectors with maximum sensitivity set to detect the wavelengths of CO and CO₂, respectively. Results from these detectors are used to determine S3 and S4 parameters.

Figure 7. CO and CO₂ Detectors without Insulating Covers.

Figure 8. Internal View of the SRA IR Section.

FID Unit
The FID detector combusts pyrolysis products in a hydrogen/air flame (Figure 9, Figure 10). Both the energy released upon combustion and the temperature at which the pyrolysis products are generated reveal hydrocarbon characteristics. Results from this detector are used to determine $S_1$, $S_2$, and $T_{\text{max}}$ parameters.

**WARNING!** During operation keep the FID unit covered to avoid injuries (the FID temperature is 325°C; be careful when handling to prevent serious burns—even when the unit is covered).

![FID Back Panel](image)

Figure 9. Internal View from Side of SRA FID Unit.

![FID Back Panel](image)

Figure 10. Internal View from Top of FID Unit.

**FID Back Panel**
Combustion gases for the FID, electric cables for oven and FID control, cooling air tube for oven, and sample gas transport pipes with hydrocarbon filter are located at the back of the FID (Figure 11). The filter needs to be replaced periodically when it turns brown (see Maintenance). The FID cover panel must be in place during operation.

![Figure 11. Back of SRA FID Unit.](image)

**Software Configuration**

Configuration options for the SRA software are organized in 6 tabs within the Configuration screen:

- Factory
- General
- Report(s)
- Pedestal
- AS Tray
- Temperatures

**Factory Tab**
The **Factory** tab contains settings for hardware controls (*Figure 12*).

![Figure 12. SRA > Configuration > Factory Tab.](image)

### General Tab

The **General** tab specifies the instrument name, data path, analysis type, and gas settings (*Figure 13*).

- **Data Path** = C:\Program Files\Thermal Station\Data
- **Analysis Type** = TPH IR
- **Enable Gases always On** must be selected at all times to protect the detectors

![Figure 13. SRA > Configuration > General Tab.](image)

### Report(s) Tab
In the Report(s) tab (Figure 14), sample reports (CO, CO2, FID, TEMP) are selected and .CSV File Creation is selected for Individual Sample Report and Sequence Report. Make sure Printer is not selected.

![Figure 14. SRA > Configuration > Report(s) Tab.](image)

**Pedestal Tab**

The Pedestal tab configures the seal and purge position numbers and the purge delay time (Figure 15). Generally, these positions do not need to be adjusted. Default Purge Delay (Seconds) is set to 150. To light the FID flame, on the Pedestal tab reseal the oven by completing the following:

1. Click Go to Home (Down Position).
2. Click Seal Position #.

![Figure 15. SRA > Configuration > Pedestal Tab.](image)

**AS Tray Tab**
If the autosampler is disconnected, reconnect and click on the AS Tray tab to adjust the pedestal and calibrate the autosampler (Figure 16; see instructions in Autosampler Tray Arm Adjustment).

**Temperatures Tab**

The **Temperatures** tab controls oven and FID detector temperatures (Figure 17). To protect the detector, FID gases must be flowing when the detector temperature is >150°C. If gases are not on and FID temperature is >150°C, type a lower temperature in the Detector (FID) Standby Temperature field and select **Send Temperatures Immediately** to reset detector temperature. Standard Temperatures tab settings are as follows:

- Oven Standby Temperature: 300
- Detector (FID) Standby Temperature: 325
- Oven Offset Temperature: –9.10

**Method Editor Screen**

The **Method Editor** screen has three active tabs:

- Acquisition
- Temperatures
- Standard
Acquisition Tab

In the Acquisition tab (Figure 18), an SRA acquisition method can be defined, comments about the method entered, and FID gain (attenuation) set.

Temperatures Tab

In the Temperatures tab (Figure 19), the oven temperature program and FID temperature setting are specified. The oven program determines the rate at which S1 and S2 fractions are pyrolyzed and sent to the detectors.

Standard Tab
Any time a new standard material is used to calibrate the SRA, it must be documented in the **Standard** tab, where the standard name and known values are recorded. (Figure 20.

![Figure 20. SRA > Method Editor > Standard Tab.](image)

**High TOC Samples**

To measure a high TOC sample, the following method settings are recommended:

- FID temperature 350°C
- Pyrolysis
- Initial temperature 340°C
- Hold 3 min
- Ramp 25°C/min
- Final temp 640°C
- Hold 1 min
- Oxidation
- Purge 5 min
- Time 20 min
- Temperature 580°C

**Flow Control & Calibration**

Gas flow and gas pressure are set by hand on the SRA system. These gas flow systems must be checked:

- Carrier gas (He)
- Oxidation gas (O<sub>2</sub>)

**Setting/Adjusting Oven Gas Flow**

Carrier gas (He) and oxidation gas (O<sub>2</sub>) flow into the oven from the bottom of the pedestal. Adjust these gas flows as follows.

1. Unscrew the carrier/oxidation gas connector and connect gas line to a gas flowmeter (Figure 21). See *Using the Flowmeter to Measure Gas Flow* for instructions on using the flowmeter.
2. Click Configuration on the Main Menu (Figure 22) and then select the **General** tab.
3. Select He (Figure 23).
4. Measure the He flow using the flowmeter more than once.
5. If gas flow is higher or lower than recommended (50–55 mL/min), rotate the Helium (Carrier) knob on the rear panel counterclockwise for higher flow or clockwise for lower flow (Figure 6).
6. On the General tab, switch gas selection from He to Air and then click Send Gases Setting Immediately to activate the change.
7. Measure the air flow using the flowmeter more than once.
8. If air flow is not ~250–260 mL/min, adjust using the Air (Oxidation) knob on the rear panel: counterclockwise for higher flow or clockwise for lower flow.
9. Reattach the carrier/oxidation gas line to the SRA.
10. On the General tab, switch gas selection from Air to He, and then click Send Gases Setting Immediately to activate a change.
11. Check for gas leaks and click Exit to close the General tab on the software.
Using the Flowmeter to Measure Gas Flow

1. Fill the red bulb on the flowmeter half full or less with soap solution.
2. Squeeze the bulb several times to coat the inside of the glass with solution.
3. Press the ON/RESET button on the flowmeter so the readout is 0.0.
4. Unscrew the carrier/oxidation gas connector, and connect the gas line to the gas flowmeter at the T-junction just above the red bulb (Figure 21).
5. Gently squeeze the red bulb to release a single soap bubble, which travels up the glass tube and passes the sensors to get an accurate reading. If the soap suds, try squeezing the bulb more gently or lower the gas flow until the flowmeter stabilizes, then raise the flow to the required level.
6. Press ON/RESET with no bubble flowing and start a new soap bubble as many times as it takes to get 3–5 stable readings in mL/min output.
7. Adjust the gas flow on the instrument as needed according to the flowmeter reading.

Setting/Adjusting FID Gas Flow

FID gas flow can be measured either from the front or back of the SRA. To measure gas flow in the front (Figure 24), the FID cover must be removed. However, the stainless steel gas tube from the joint to the FID has little flexibility, so FID gas should be measured from the back of the instrument (Figure 25).

![Figure 24. Gas Terminals Connected to the Front.](image1)

![Figure 25. Gas Terminals Connected to the Back.](image2)

To measure and adjust gas flow from the rear of the instrument, follow these steps.

1. Unscrew hydrogen gas connector and connect the gas line to a gas flowmeter. See Using the Flowmeter to Measure Gas Flow for instructions on using the flowmeter.
2. Measure hydrogen flow more than once.
3. If hydrogen flow is outside the target range of ~50–55 mL/min, rotate the Hydrogen (FID) knob on the rear panel counterclockwise for higher flow or clockwise for lower flow (see Figure 6).
4. Measure the air flow more than once.
5. If air flow is outside of the target range of ~300–305 mL/min, rotate the AIR (FID) knob on the rear panel of the main control unit counterclockwise for higher flow or clockwise for lower flow.
6. Reconnect air and hydrogen lines to FID, light the FID, and check to make sure the flame is lit.

![Figure 26. Gas Flow Measurement.](image)

**Figure 26. Gas Flow Measurement.**

**IR Gas Pressure Control**

At the end of the sample gas line, a small vacuum pump brings sample gases from the oven to the IR detectors. The pressure controller keeps constant pressure between the oven and IR detectors. The pressure controller is in the IR section of the SRA (Figure 27).

![Figure 27. Pressure Controller (yellow).](image)

**Figure 27. Pressure Controller (yellow).**

This gas line does not need to be measured with the flowmeter; simply adjust the gas pressure to ensure that the gas pressure is ~40 psi on the pressure meter (Figure 28). To adjust gas pressure, turn the dial on the rear panel of the IR section of the SRA unit (counterclockwise for higher flow or clockwise for lower flow; Figure 28).
Maintenance

Routine maintenance of the SRA includes the following. Additional troubleshooting information (i.e., encountered problems) is available in the binder and log books.

<table>
<thead>
<tr>
<th>Maintenance</th>
<th>How Often</th>
<th>See</th>
</tr>
</thead>
<tbody>
<tr>
<td>Calibrate IR detectors</td>
<td>Once per year</td>
<td>IR Calibration</td>
</tr>
<tr>
<td>Adjust autosampler tray arm</td>
<td>After autosampler tray is disconnected</td>
<td>Autosampler Tray Arm Adjustment</td>
</tr>
<tr>
<td>Change filter behind FID back panel</td>
<td>When it turns brown</td>
<td>FID Unit Filter</td>
</tr>
<tr>
<td>Warm up 6 hr</td>
<td>After power shutdown</td>
<td>SRA Instrument Preparation section in the SR Analyzer User Guide</td>
</tr>
<tr>
<td>Calibrate with Calibration Standard</td>
<td>After power shutdown</td>
<td>Quality Assurance/QA Control section in the SR Analyzer User Guide</td>
</tr>
<tr>
<td></td>
<td>With each batch or 1 per day</td>
<td></td>
</tr>
</tbody>
</table>

IR Calibration

The IR must be calibrated at least once a year. Turn the instrument on at least 2 hr before calibrating. Calibration history records are located on the side panel of the instrument. Use the following standardized gases to calibrate the SRA (method error is 5% [-500]).

- 1% carbon monoxide, balance is nitrogen
- 1% carbon dioxide, balance is nitrogen

Calibrating the CO and CO₂ Infrared Detectors
1. Turn off SRA instrument.
2. Open the IR Flow and Pressure Control Unit.
3. If you disconnect the sample gas tube and electric cable that run between the Main Control Unit and the IR Flow and Pressure Control Unit to open IR Flow and Pressure Control Unit, connect both tube and cable.
4. Turn on the SRA.
5. Click the IRCal.exe shortcut icon (Figure 30) to open the IR Calibration screen (Figure 31).
6. On the screen, select START.
7. On the screen, click Zero under CO.
8. After the value starts to change, click Zero under CO2 Error of measurement is 5% (~500).
9. Connect the CO calibration gas to the inlet of the CO detector and disconnect the vent from the CO2 detector.
10. Open the calibration gas tank. On the screen, CO will increase until to ~10,000 (8000 ~ 9000).
11. Wait until the CO signal stabilizes and then click Calib under CO.
12. Close the CO tank and disconnect.
13. Connect the CO2 calibration gas to the inlet of the CO2 detector and open the gas tank.
14. Wait until the CO2 signal stabilizes and then click Calib under CO2.
15. Close the CO2 tank and disconnect.
16. Select STOP, close the window, and reassemble the main instrument units.

Figure 30. Shortcut to IR Calibration.

Figure 31. IR Calibration Screen.

Autosampler Tray Arm Adjustment

The position of the autosampler can seriously affect measurement results. After movement or installation of the autosampler unit, use the following procedure to confirm the positional relationship between the autosampler and the pedestal.

1. Click Configuration on the Main Menu and select the AS Tray tab (Figure 32).
2. Place an empty crucible in autosampler tray Position 1.
3. Click Place a Crucible in Tray Position 1, then click to begin.
4. Select an axis direction on the window: left/right, front/back, or up/down.
5. Unlock Coarse Movement.
6. Adjust arm position using L or R buttons on the screen.
7. When finished, select Save All AS Tray Axis Positions to save changes.
FID Unit Filter

The hydrocarbon filter on the back of the FID unit is filled with a white powdery substance (Figure 11). When the material in the hydrocarbon filter turns brown, replace the filter by disconnecting tubing from top and bottom.

**Warning!** Burn hazard—replace FID protective cover before operating the FID.

Vendor Information

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Related Documentation and Links

The following vendor support documents can be downloaded from Cumulus:

- SRA Pedestal Seal Adjustment.pdf
- SRA Pedestal Seals.pdf
- SRA-IR Gas Control.pdf
- SRA Instrument Gas flows.pdf
- SRA Install Requirements.pdf
FID lab air -> CO2 trap -> moisture trap -> unit
CO2: proprietary formulation

- Indicating CO2 adsorbent
- Sodium hydroxide
- Non-fibrous silicate

Moisture trap

- 13X-Z8, 4A-Z8 molecular sieve, synthetic sodium potassium or calcium aluminosilicate