SRA Advanced User Guide

Taken from SRA_AUG_Exp355

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°–850°C
- Heating rates: 0.10°–50°C/min
- Detector: 200°–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 cm^3)
- 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/CO_2 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 ft^3/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***).

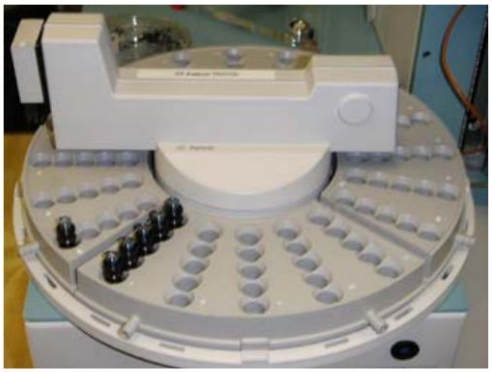

Figure 2. Autosampler Tray.

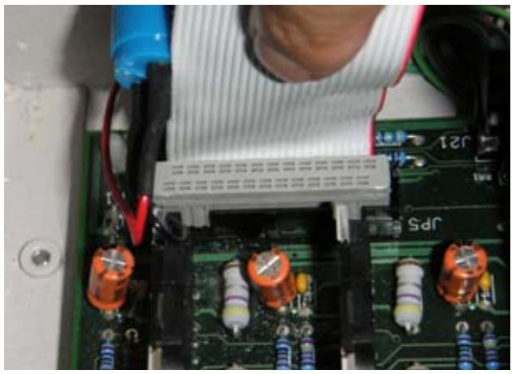

Figure 4. Serial Cable Connector to the Main Control Unit.

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

- H_2: Hydrogen gas flow controller
- AIR (FID): Air gas flow controller
- CC: Carrier gas flow controller
- CS (above CC): Carrier gas flow controller

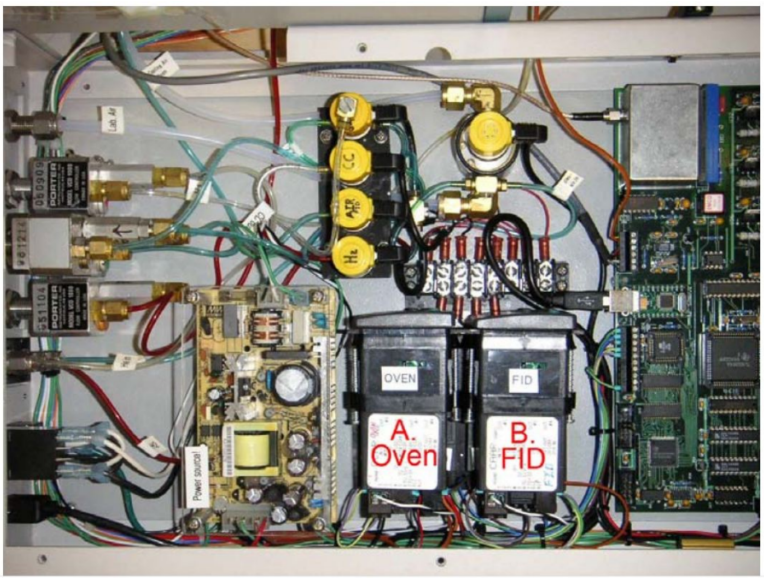

Figure 5. Internal View of the SRA Main Control Unit.

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.

Figure 6. Gas Flow Control Knobs on Rear Panel of Main Control Unit.

IR Section

The IR section (***Figure 7, Figure 8***) contains CO and CO_2 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 CO_2 detectors function as infrared detectors with maximum sensitivity set to detect the wavelengths of CO and CO_2, respectively. Results from these detectors are used to determine S3 and S4 parameters.

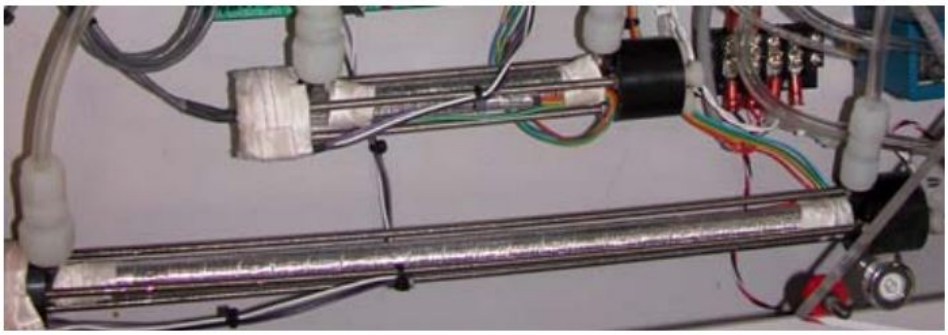

Figure 7. CO and CO_2 Detectors without Insulating Covers.

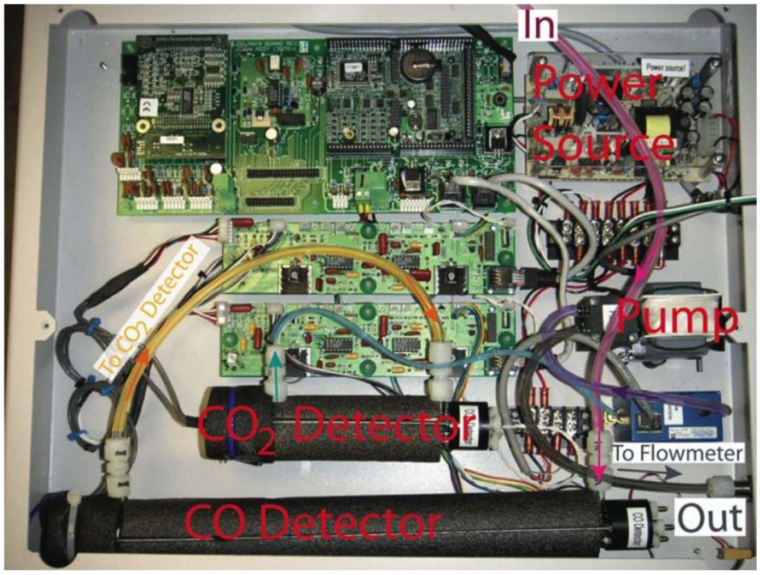

Figure 8. Internal View of the SRA IR Section.

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 S1, S2, and T_{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).

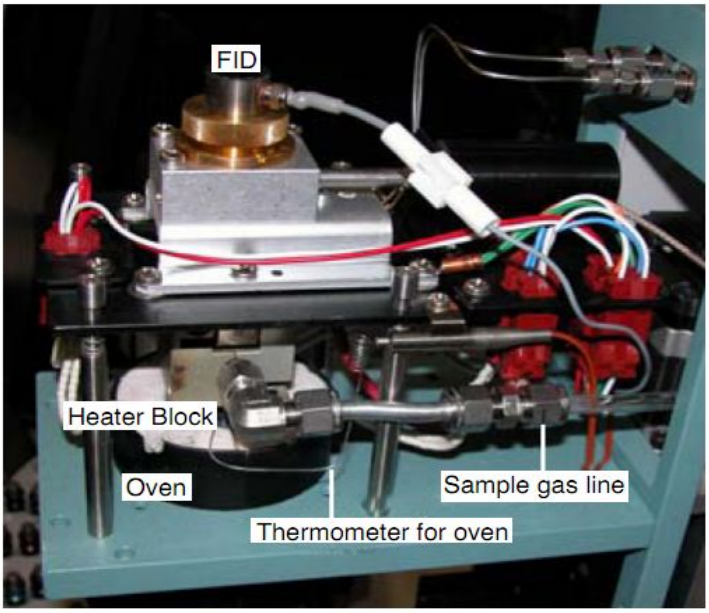

Figure 9. Internal View from Side of SRA FID Unit.

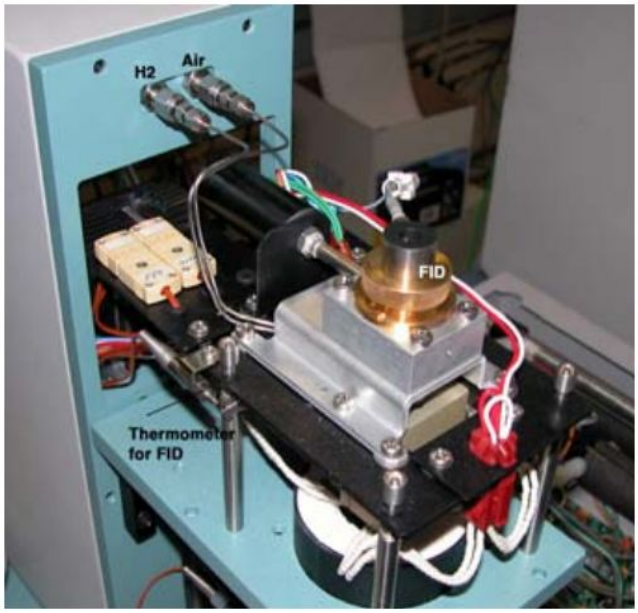

Figure 10. Internal View from Top of FID Unit.

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.

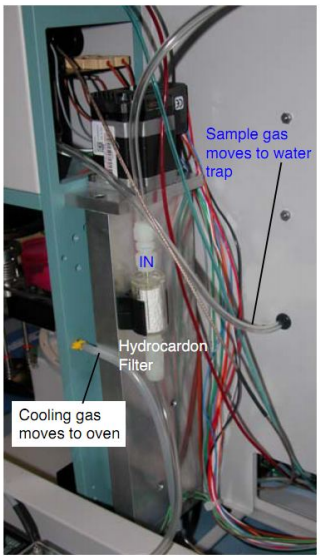

Figure 11. Back of SRA FID Unit.

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***).

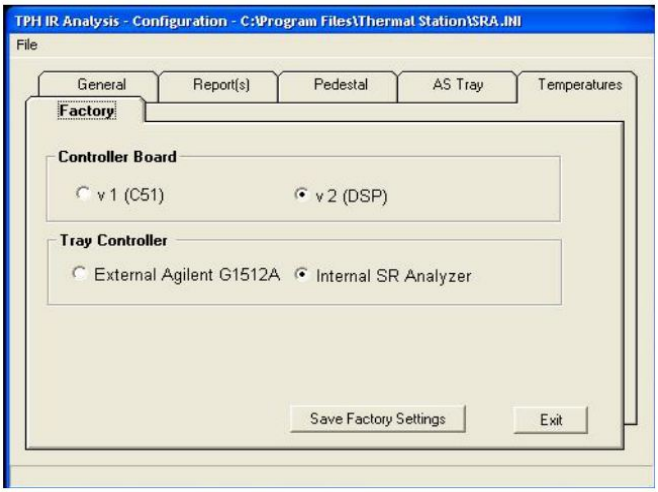

Figure 12. SRA > Configuration > Factory Tab.

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

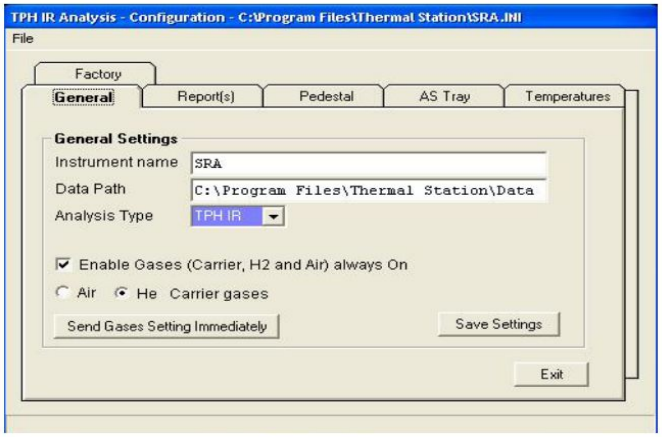

Figure 13. SRA > Configuration > General Tab.

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.

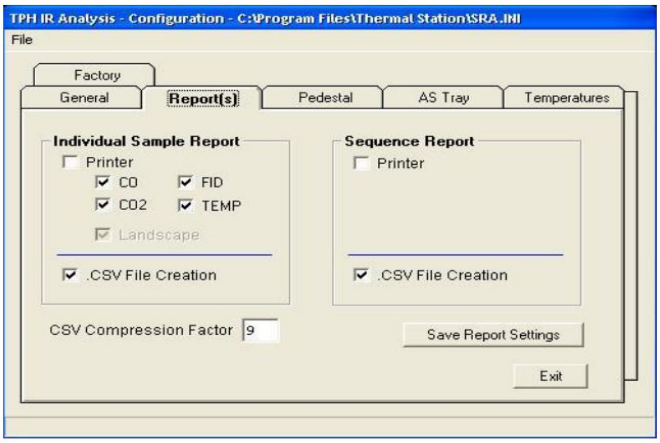

Figure 14. SRA > Configuration > Report(s) Tab.

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 #.**

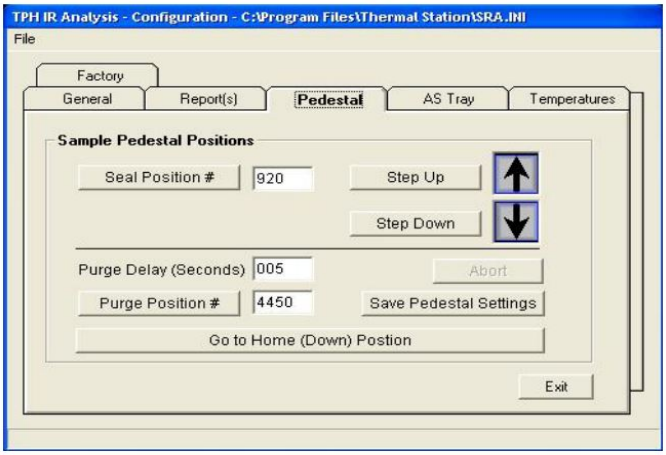

Figure 15. SRA > Configuration > Pedestal Tab.

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).

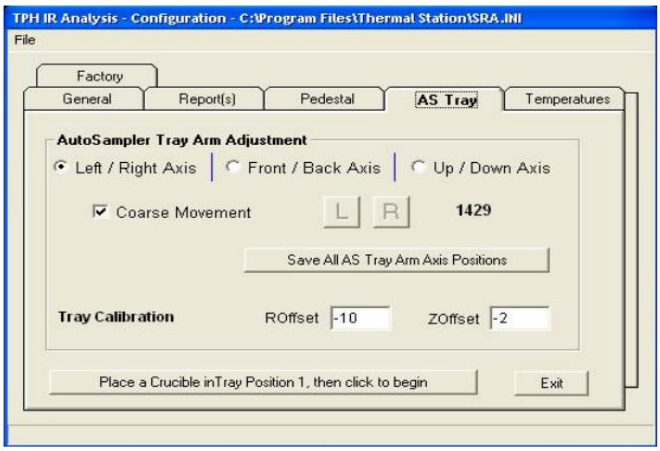

Figure 16. SRA > Configuration > AS Tray Tab.

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

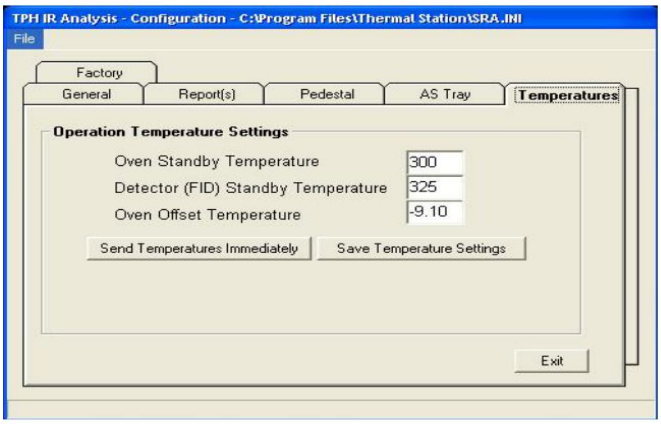

Figure 17. SRA > Configuration > Temperatures Tab.

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.

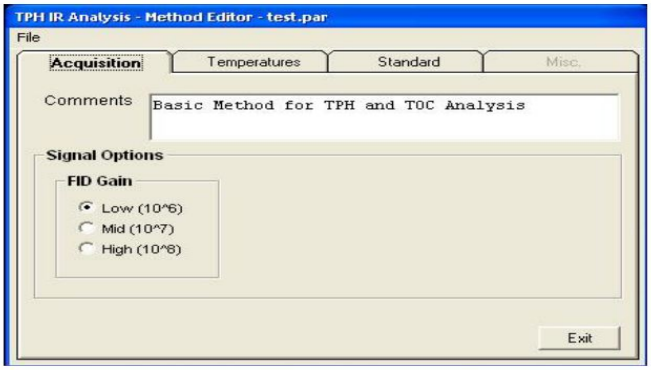

Figure 18. SRA > Method Editor > Acquisition Tab.

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.

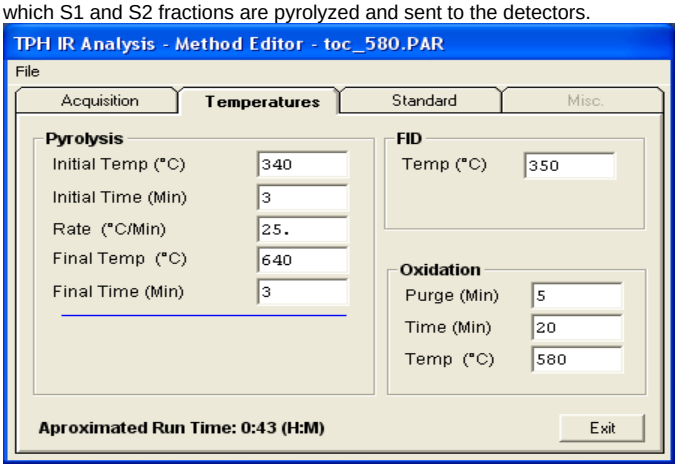

Figure 19. SRA > Method Editor > Temperatures Tab.

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***).

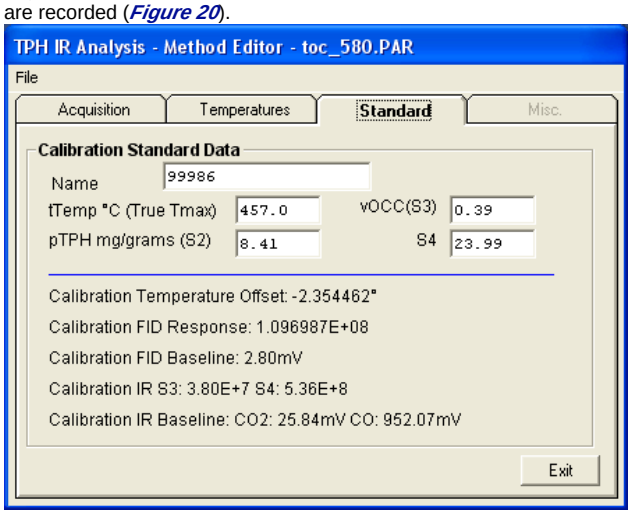

Figure 20. SRA > Method Editor > Standard Tab.

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_2)

Setting/Adjusting Oven Gas Flow

Carrier gas (He) and oxidation gas (O_2) 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.

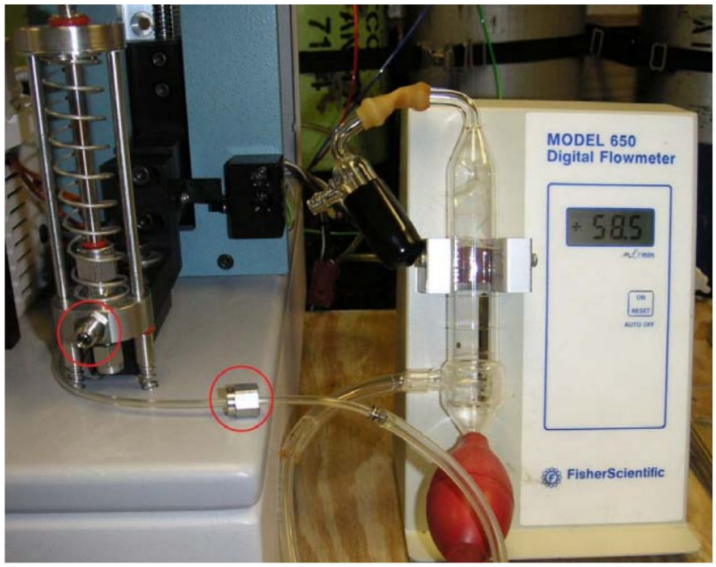

Figure 21. Gas Flow Measuring Equipment.

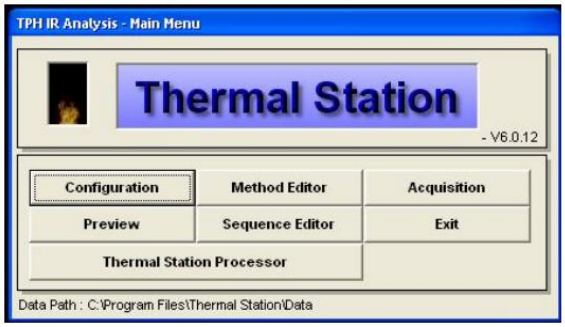

Figure 22. Main Menu Screen.

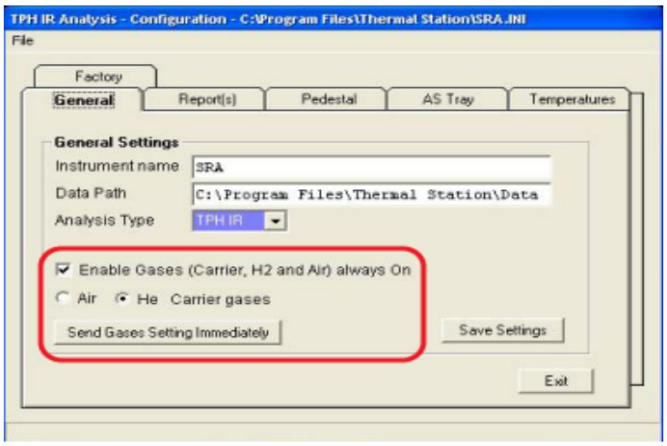

Figure 23. Gas Flow Settings on General Tab.

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 sudses, 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***).

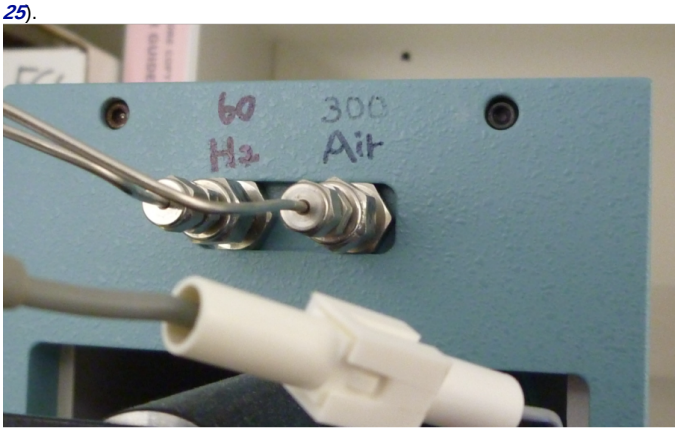

Figure 24. Gas Terminals Connected to the Front.

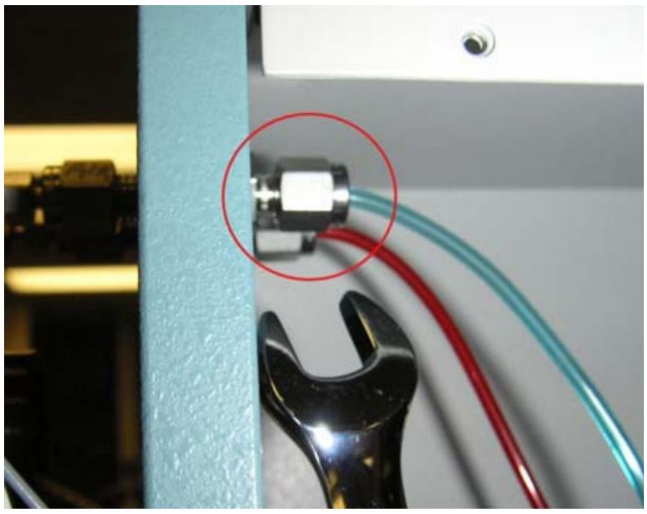

Figure 25. Gas Terminals Connected to the Back.

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.

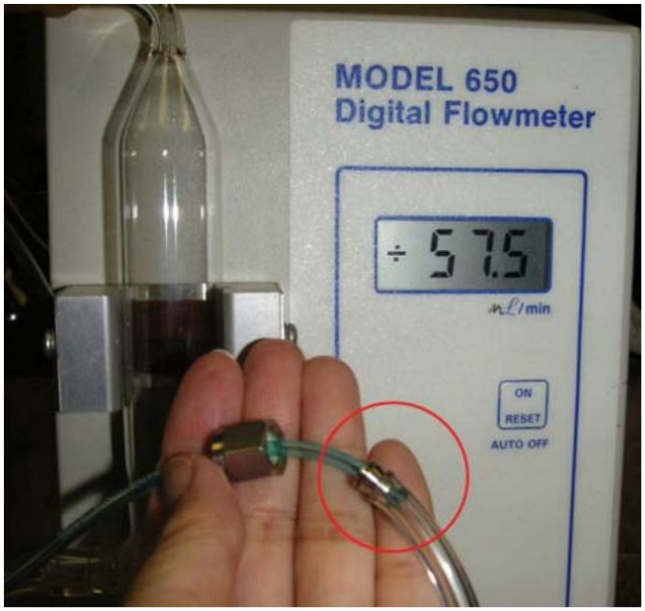

Figure 26. Gas Flow Measurement.

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***).

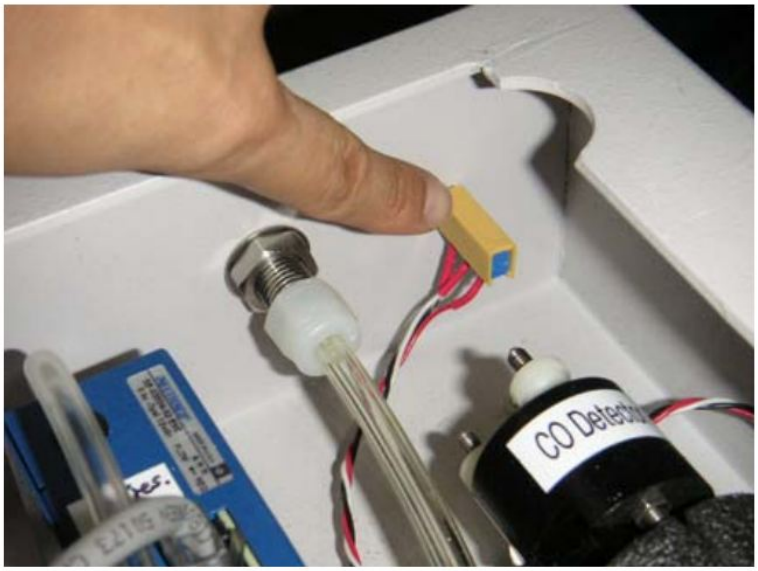

Figure 27. Pressure Controller (yellow).
This gas line does not need to be measured with the flowmeter; simply adjust the gas pressure to ensure that the gas pressure is ~40 psi on the pressure meter (***Figure 29***). To adjust gas pressure, turn the dial on the rear panel of the IR section of the SRA unit (counterclockwise for higher flow or clockwise for lower flow; ***Figure 28***).

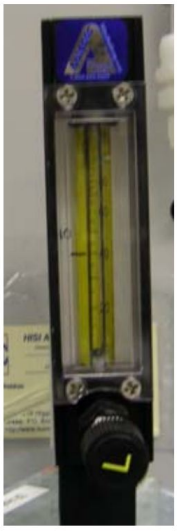

Figure 29. Gas Flow Meter.

Maintenance

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

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

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 CO_2 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 CO_2 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 CO_2 calibration gas to the inlet of the CO_2 detector and open the gas tank.
14. Wait until the CO_2 signal stabilizes and then click **Calib** under *CO2*.
15. Close the CO_2 tank and disconnect.
16. Select **STOP**, close the window, and reassemble the main instrument units.

Figure 30. Shortcut to IR Calibration.

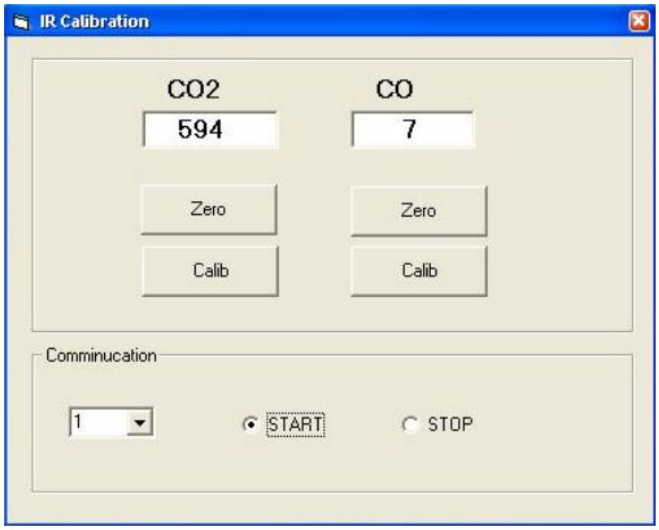

Figure 31. IR Calibration Screen.

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.

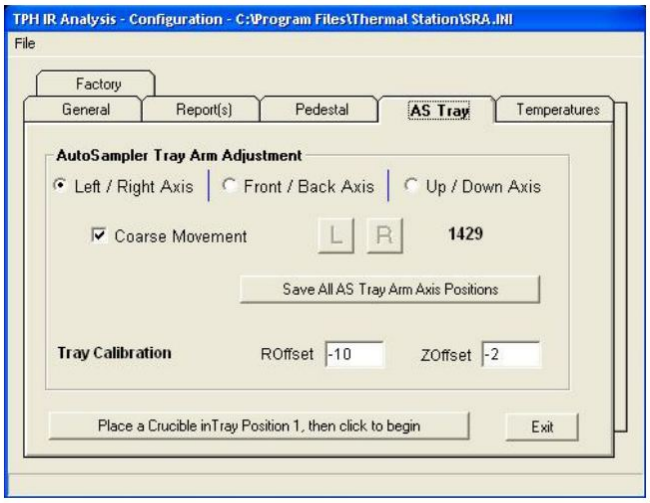

Figure 32. AS Tray Tab.

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