Q5000 SA Sorption Analyzer



Q SeriesTM Getting Started Guide



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Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions.

A NOTE highlights important information about equipment or procedures.



A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.



A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.

Regulatory Compliance

Safety Standards

For Canada:

CAN/CSA-22.2 No. 1010.1-92 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.

CAN/CSA-22.2 No. 1010.2.010-94 Particular requirements for laboratory equipment for the heating of materials + Amendments.

<u>For the European Economic Area</u>: (In accordance with Council Directive 73/23/EEC of 19 February 1973 on the harmonization of the laws of Member States relating to electrical equipment designed for use within certain voltage limits.)

EN61010-1: 2001 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.

EN61010-2-010: 1994 Particular requirements for laboratory equipment for the heating of materials + Amendments.

For the United States:

UL 61010A-1: Electrical Equipment for Laboratory Use; Part 1: General Requirements. UL 61010A-2-010 Part 2: Particular requirements for laboratory equipment for the heating of materials.

Electromagnetic Compatibility Standards

For Australia and New Zealand:

AS/NZS CISPR 11:2004 Limits and methods of measurement of electronic disturbance characteristics of industrial, scientific and medical (ISM) radiofrequency equipment.

For Canada:

ICES-001 Issue 3 March 7, 1998 Interference-Causing Equipment Standard: Industrial, Scientific, and Medical Radio Frequency Generators.

For the European Economic Area: (In accordance with Council Directive 89/336/EEC of 3 May 1989 on the approximation of the laws of the Member States relating to electromagnetic compatibility.)

EN61326-1: 1997 Electrical equipment for measurement, control, and laboratory use-EMC requirements-Part 1: General Requirements + Amendments. Emissions: Meets Class A requirements (Table 3). Immunity: Meets performance criteria A for non-continuous operation (Table B.1).

For the United States:

CFR Title 47 Telecommunication Chapter I Federal Communications Commission, Part 15 Radio frequency devices (FCC regulation pertaining to radiofrequency emissions).

Safety

Instrument Symbols

The following label is displayed on the instrument for your protection:

Symbol

Explanation



This symbol on the rear access panel indicates that you must unplug the instrument *before* doing any maintenance or repair work; AC mains power voltage is present in this system.

High voltages are present in this instrument. If you are not trained in electrical procedures, do not remove the cabinet covers unless specifically instructed to do so in the manual. Maintenance and repair of internal parts must be performed only by TA Instruments or other qualified service personnel.

Please heed the warning labels and take the necessary precautions when dealing with these areas. The *Q5000 SA Getting Started Guide* contains cautions and warnings that must be followed for your own safety.

Electrical Safety

You must unplug the instrument *before* doing any maintenance or repair work; voltages exceeding 120 Vac are present in this system.



WARNING: High voltages are present in this instrument. If you are not trained in electrical procedures, do not remove the cabinet covers unless specifically instructed to do so in the manual. Maintenance and repair of internal parts must be performed only by TA Instruments or other qualified service personnel.

Chemical Safety

Use only the purge gases listed in Chapter 1. Use of other gases could cause damage to the instrument or injury to the operator.



WARNING: If you are using samples that may emit harmful gases, vent the gases by placing the instrument near an exhaust.

Thermal Safety

After running an experiment, allow the sample pans to cool down before you touch them.

Mechanical Safety



WARNING: Keep your fingers and all other objects out of the path of the humidity chamber when it is moving. The seal is very tight.

Lifting the Instrument

The Q5000 SA is a fairly heavy instrument. In order to avoid injury, particularly to the back, please follow this advice:



WARNING: Use two people to lift and/or carry the instrument. The instrument is too heavy for one person to handle safely.

Chapter 1 Introducing the Q5000 SA

Overview

Your Sorption Analyzer, the TA Instruments Q5000 SA, is used to determine the amount of moisture a material can adsorb or desorb as a function of its mass, temperature, time, and relative humidity. This instrument is primarily concerned with applications where changing levels of humidity can influence or significantly alter important properties or uses of a material (*e.g.*, physical properties (Tg), stability, shelf life, bioavailability).

The Q5000 SA is a symmetrical system where the sample and reference (tare) are exposed to the same environmental conditions (temperature and humidity). The main components of the system are the temperature-controlled vertical microbalance, a humidity chamber, and an autosampler. The humidity chamber maintains identical temperature and humidity conditions for the sample and reference areas, which enable precise sorption/desorption analyses.



Q5000 SA

The Q5000 SA is used in conjunction with a controller (computer) that performs the following functions:

- Provides an interface between you and the Q5000 SA
- Enables you to set up experiments and enter constants
- Stores experimental data
- Runs data analysis programs.

Q5000 SA Components

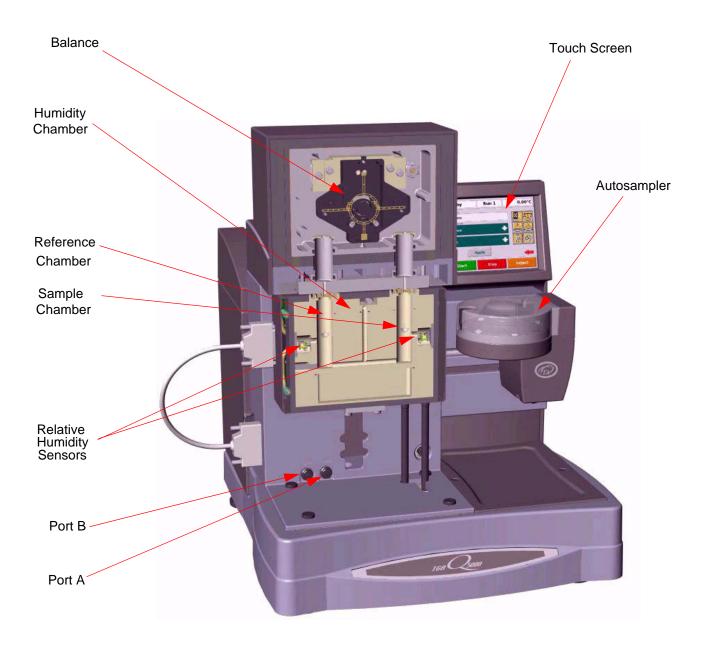
The Q5000 SA has the following major components:

- The *balance*, which provides precise measurement of sample weight. Balance sensitivity is a major component of the Sorption Analysis system performance.
- A humidity chamber, which incorporates individual sample and reference chambers; Mass Flow Controllers (MFCs), transmission and mixing purge gas lines, and a humidity reservoir used to generate a purge gas of known %RH; and two separate relative humidity sensors, which provide an indication of the humidity in the two chambers.
- The *Autosampler*, which loads and unloads the sample to and from the balance.
- The Mass Flow Controllers (or MFCs), which control the purge gas to the balance and to the humidity chamber. Proper regulation of the humidity chamber MFCs is used to generate the desired %RH. There are three MFCs used in the Q5000 SA.
- A touch screen, which is used to communicate commands to the instrument and provides real-time data display.
- The *cabinet*, where the system electronics and mechanics are housed.

• The *heat exchanger*, which works with the Peltier elements on the walls of the humidity chamber to maintain the temperature of the chamber.

See the schematic below for the location of the various components. Consult the online documentation associated with the instrument control software for detailed information on their operation.

NOTE: For technical reference information, theory of operation, and other information not found in this manual, see the online help associated with the instrument control software.



Balance Assembly

The Q5000 SA balance assembly consists of the balance meter movement, the balance arm, the balance arm sensor, the hang-down wire assemblies, the sample pan, and the tare pan.

The balance meter movement is a taut-band meter movement to which the balance arm is attached.

The *balance arm* is an assembly constructed of beryllium copper alloy attached to the meter movement. It is in a null balance system. A hang-down loop is attached to each end to hold the hang-down wires.

The *balance position sensor* is comprised of an LED source and a printed circuit board assembly that detects the null position of the meter movement. The balance beam sensor is mounted above the balance arm. It is used in conjunction with the analog circuitry to maintain a null position.

The instrument has two *hang-down wire assemblies*: one for the reference (tare) pan and one for the sample pan. Each assembly consists of a hang-down wire and loop. The hang-down wire has hooks at each end and connects the pan to the loop. The loop has eyelets at each end; it is used to connect the hang-down wire to the balance arm. The reference hang-down wire and reference pan mechanically balances the weight of the sample pan and sample hang-down wire.

Humidity Chamber

The humidity chamber consists of the sample and reference chambers, humidifier, gas transmission and mixing lines, and two independent relative humidity sensors. All of these components are maintained at the same temperature. The temperature range is 5 to 85°C.

The relative humidity (RH) is controlled by a pair of very precise mass flow controllers (MFCs). Sensors located near the sample and reference provide ongoing verification that the relative humidity achieved is what was requested.

The relative humidity range of the chamber can be controlled from 0 to 98 %RH.

The humidity chamber housing is motorized and can be moved up and down for automatic loading and unloading of samples onto the hang-down wire within the sample chamber. Samples are loaded via the Autosampler, which is capable of holding a total of ten hemispherical quartz pans.

Q5000 SA Autosampler

The Q5000 SA Autosampler (see the figure here) allows you to place multiple samples on the platform for automatic loading and run sequencing. Using the quartz pan tray for the humidity chamber, up to ten 180 μL hemispherical quartz pans can be accommodated at one time. Experiments are performed as they normally would be using the instrument—but samples can be run on a continual basis.

To calibrate the sample tray, refer to Chapter 3 of this manual and the online help found in the instrument control software.



The Q5000 SA Touch Screen

The Q5000 SA instrument has a built-in integrated display and keypad in the form of a touch screen for local operator control. The functions on the screen change depending upon the menu you are using. This section briefly describes the basic layout of these functions.

The *status line* along the top of the display shows the current instrument status, run selection, and temperature.

At the bottom of the screen is a set of keys that are used for the primary instrument functions. See the table below for a description of each key.

The functions in the middle of the touch screen will vary depending on the screen displayed.



Primary Function Keys

Use the following keys for the main functions of the instrument.

Key Name	Description
Start	Begins the experiment. This is the same function as Start on the instrument control software. Start automatically loads the sample pan and closes the humidity chamber, if necessary, before beginning the experiment.
Stop	If an experiment is running, this key ends the method normally, as though it had run to completion; i.e., the method-end conditions go into effect and the data that has been generated is saved. This is the same function as Stop on the instrument control software. If an experiment is not running (the instrument is in a standby or method-end state), the Stop key will halt any activity (all mechanical motion, etc.). If an Autosampler sequence is in progress, Stop will halt the sequence.
Control (table continued)	Displays a list of the control command functions. These are used to control the instrument actions such as humidity chamber movement, sample loading/unloading, taring, etc. Items can be selected from the icons or from the drop-down menu. Select Apply to initiate the command. See the next page for more details on this screen.

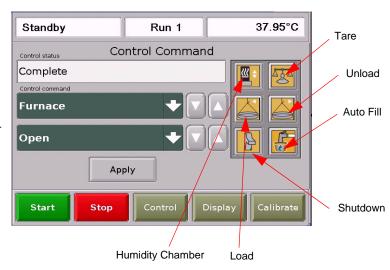
Display	Accesses the display screen, which displays the signals from the instrument such as signal display, real-time plot, instrument information, etc.
Calibrate	Displays the calibration functions available for this instrument. Functions such as autosampler and touch screen calibration can be accessed using this key.

Control Menu

The Control Menu (see the figure to the right) is accessed by touching the **Control** key at the bottom of the touch screen. A brief description of each control command is provided in the table below.

NOTE: Most of the commands shown are not available during an active experiment.

Select the desired function either from the drop-down list of Control Commands or by pressing the icon. Then press **Apply** to initiate the action.



Control Command	Description
LOAD/UNLOAD	Loads or unloads a sample pan from the sample platform onto the balance. This function will automatically close and open the humidity chamber, if necessary.
TARE	Zeros the displayed weight of an empty sample pan—automatically loads the pan from the sample platform, raises the humidity chamber to protect the pan from air currents, weighs the pan, stores the weight as an offset, and then unloads the pan.
TARE ALL	Electronically zeros the displayed weight of an entire tray of empty pans.
FURNACE (Humidity Chamber)	Toggles between the humidity chamber closed (up) and humidity chamber open (down) functions, depending on where the humidity chamber is when you press the key. This key can be pressed while the humidity chamber is moving, to reverse the direction of movement.
(table continued)	

AUTO FILL	Use this button to enable the Humidity Fill Mode to allow filling of the humidity chamber with distilled water. This is the same function as selecting "Humidity Fill Mode" using the Control Command pull-down menu on the Control Screen.
HEAT EXCHANGER	Toggles the heat exchanger on or off.
RESET AUTO	Resets the autosampler.
PAN TO FRONT	Use the pull-down menu to select the desired pan number position on the autosampler sample tray. The selected pan number shown on this window will be brought to the front.
RESET SAVED PARAMETERS	Resets the saved instrument parameters and resets the instrument.
SHUTDOWN	Shuts down and resets the instrument.

Display Touch Screen Options

The Display Options are accessed by touching the **Display** key at the bottom of the touch screen. The keys shown in the figure to the right are displayed.

A brief description of the function of each key is provided in the table below.



Key Name	Description
SEGMENTS 2 3	Accesses the experimental method that is currently being used for this experiment.
INFORMATION	Displays instrument information such as the software version, options, and the IP address.
(table continued)	

STATUS	0	Displays the three main signals indicating the current status of the experiment.
SIGNALS	0.0	Displays the real-time signal data that comes directly from the instrument. The signals displayed here are customized through the instrument control software by accessing Tools/Instrument Preferences.
PLOT	\mathcal{I}	Displays a time-based plot of data as it is received from the instrument during experiments.
SCREEN SAVER		Allows you to choose a screen saver for the touch screen.
номе	î	Returns to the opening window.

Calibration Options

The Calibration Options are accessed by touching the **Calibrate** key at the bottom of the touch screen. The keys shown in the figure below are displayed. A brief description of the function of each key is provided in the table below.



Key Name		Description
TOUCH SCREEN	RL F	Allows you to calibrate the touch screen display.
AUTOSAMPLER		Accesses the Autosampler Calibration functions.

Instrument Specifications

The tables found on the following pages contain the technical specifications for the Q5000 SA.

Q5000 SA Instrument Characteristics

Dimensions	Depth 55.9 cm (22 in.) Width 47 cm (18.5 in.) Height 61 cm (24 in.)
Weight of Instrument Weight of Transformer (for 230 Vac operation)	39.5 kg (87 lbs) 8.18 kg (18 lbs)
Power	120 Vac, 50/60 Hz, standard 230 Vac, 50/60 Hz, if configured with a step-down transformer
Energy Consumption	1.44 kVA maximum, includes accessory power outlets
Insulation Rating	All electrical insulation between hazardous and low voltage components have been designed to meet the requirements of reinforced insulation. Low voltage circuits are grounded.
Room Operating Temperature	15°C to 30°C (non-condensing)
Temperature Control Range	5 to 85°C
Humidity Range	0 to 98 %RH
Humidity Reservoir Liquid	Distilled water

SA Sampling System

The following tables contain the specifications associated with the SA sample pans, balance mechanism and humidity chamber.

Sample Pan Options for Ten-Pan Tray

Types	Hemispherical quartz
Pan Volume Capacity	180 μL
Number of Pans per Tray	10 quartz pans

NOTE: Other pan types are available (such as platinum and sealed aluminum). A 25-pan tray is required for these pans. Consult online help for a complete listing.

Balance Mechanism

Weighing capacity (sample) ¹	100 mg (nominal)
Balance measurement	Tare weight required
Resolution	0.01 μg
Accuracy	≤±0.1% of value or 10 µg, whichever is greater



¹ CAUTION: The total mechanical capacity of the balance suspension is 5 g. In order to avoid damaging the balance assembly, never allow the total weight of the sample, tare weight, balance beam, hang-down wires, and pans to exceed 5 g. The total mass of the balance system, excluding the sample pan, reference pan, sample and tare mass is 3.23 g. If the Autosampler is used, the maximum sample and pan mass will be 600 mg with a 500 mg reference pan and tare mass. If manual loading is performed, the maximum sample and pan mass is 930 mg with 830 mg on the reference side.

Atmosphere Control/Humidity

Purge Gases	Nitrogen (dry, 99.999 %RH)
MFC Purge Rate:	Recommended 200 mL/min for humidity chamber Recommended 10 mL/min for balance

Operating Environment

Ambient temperature range	15 – 30°C
Altitude	Less than 2 km above sea level

Chapter 2 Installing the Q5000 SA

Unpacking/Repacking the Q5000 SA

The instructions needed to unpack and repack the instrument are found as separate unpacking instructions in the shipping box and in the online documentation associated with the instrument control software. You may wish to retain all of the shipping hardware, the plastic pallet, and boxes from the instrument in the event you wish to repack and ship your instrument.



WARNING: Have an assistant help you unpack this unit. Do not attempt to do this alone.

Preparing the System

Before shipment, the instrument is inspected both electrically and mechanically so that it is ready for operation upon proper installation. Only limited instructions are given in this manual, consult the online documentation for additional information. Installation involves the following procedures:

- Inspecting the system for shipping damage and missing parts
- Filling the heat exchanger
- Connecting the Q5000 SA to the TA Instruments controller
- Installing the voltage configuration unit, if 230 Vac will be used rather than 120 Vac.
- Connecting the heat exchanger cable and water lines, purge gas lines, accessories, and power cable
- Unpacking the balance, including removing the shipping bracket as directed.
- Installing the hang-down wires
- Leveling the instrument and aligning the hang-down wires
- Adjusting the sample platform (see online documentation)
- Filling the humidity chamber

It is recommended that you have your instrument unpacked and installed by a TA Instruments Service Representative. Call for an installation appointment when you receive your instrument.



CAUTION: To avoid mistakes, read this entire chapter before you begin installation.

Inspecting the System

When you receive your instrument, look over the instrument and shipping container carefully for signs of shipping damage, and check the parts received against the enclosed shipping list.

- If the instrument is damaged, notify the carrier and TA Instruments immediately.
- If the instrument is intact but parts are missing, contact TA Instruments.

Choosing a Location

Because of the sub-microgram sensitivity of sorption analysis experiments with the Q5000 SA, it is important to choose a location for the instrument using the following guidelines. The instrument should be:

- *In* ... a temperature-controlled area. Temperatures should be in range 20-30°C.
 - ... a clean, vibration-free environment, preferably on the ground floor in the building. It should be located away from pumps, motors, or other devices which produce vibrations.
 - ... an area with ample working and ventilation space.
- **On** ... a stable work surface. A marble table is recommended.
- *Near* ... a power outlet (120 Vac, 50 or 60 Hz, 15 amps or 230 Vac, 50 or 60 Hz, 10 amps, if configured with a step down transformer).
 - ...your TA Instruments thermal analysis controller.
 - ...purge gas supplies with suitable regulators, flowmeters and driers, if required.

Away

from ... dusty environments.

- ... exposure to direct sunlight.
- ... direct air drafts (fans, room air ducts).
- ... poorly ventilated areas.
- ... noisy or mechanical vibrations.
- ... high traffic areas, where constant movements from passing personnel could create air currents or mechanical disturbances.

Filling the Heat Exchanger

The heat exchanger contains a liquid reservoir that supplies the instrument with coolant to dissipate heat from the humidity chamber. The coolant exits the heat exchanger through the supply line, circulates to the humidity chamber, and comes back to the reservoir via the return line as seen in the figure here (for instructions on how to connect the water lines, turn to page 27). To fill the heat exchanger, follow the directions given below.

1. Unscrew the water reservoir cap on the heat exchanger (see the figure below).





2. Add TA Instruments TGA Conditioner (PN 952377.901) into the water reservoir. Refer to the instructions on the conditioner bottle for the amount of conditioner to add to the reservoir. Then fill the reservoir to the inner rim with distilled water.

NOTE: After the system has been started, recheck the level of water in the reservoir and refill to the inner rim, if necessary.



CAUTION: Do not put any liquid other than distilled water and TA Instruments' TGA Conditioner in the heat exchanger reservoir.

3. Replace and tighten the water reservoir cap.

Connecting Cables and Lines

To connect the cables and gas lines, you will need access to the instrument's rear panel. All directional descriptions are written on the assumption that you are facing the back of the instrument.



CAUTION: Connect all cables BEFORE connecting the power cords to outlets. Tighten the thumbscrews on all computer cables. There should be no power to the unit at this time.



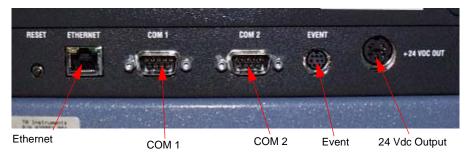
CAUTION: Whenever plugging or unplugging power cords, handle them by the plugs, not by the cords.



WARNING: Protect power and communications cable paths. Do not create tripping hazards by laying the cables across accessways.

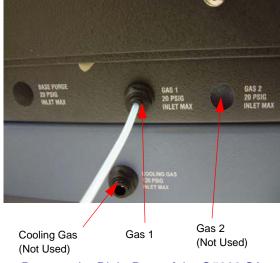
Ports

The instrument has ports that are located on the back of the instrument. The following table provides a description of function of each port. Refer to this list when connecting cables and lines.



Five Ports on Left Rear of Q5000 SA

Port	Function
Ethernet	Provides network communication capabilities
Com 1	Not used for the Q5000 SA.
Com 2	Not used for the Q5000 SA.
Event	Capable of providing a general purpose relay contact closure.
24 VDC output	Provides Heat Exchanger detection signals and voltage.
Base Purge	Not used for the Q5000 SA.
Gas 1	Inlet port to Mass Flow Controller 1 (balance purge). 140 kPa gauge (20 psig) maximum pressure.
Gas 2	Inlet port to Mass Flow Controllers 2 and 3 (humidity chamber purge). 140 kPa gauge (20 psig) maximum pressure.
Cooling Gas	Not used for the Q5000 SA.



Ports on the Right Rear of the Q5000 SA

Heat Exchanger Cable and Water Lines

Follow these instructions to connect the heat exchanger cable and water lines:

- 1. Locate the 24 Vdc output connector on the left rear of the instrument cabinet (see figure on page 26).
- 2. Connect the heat exchanger cable to the connector. The heat exchanger cable is the only cable that fits into this connector.
- 3. Remove the water lines from the packaging.
- 4. Connect one end of the water line marked "SUPPLY" to the connector labeled "SUPPLY" on the right side of the instrument cabinet (shown here).
- 5. Connect the other end of the water line marked "SUPPLY" to the connector labeled "SUPPLY" on the heat exchanger.
- 6. Connect one end of the unmarked water line to the connector labeled "RETURN" on the right side of the instrument cabinet (shown above).



Supply & Return Lines on Q5000 SA

7. Connect the other end of the unmarked water line to the connector labeled "RETURN" on the heat exchanger.

NOTE: Air trapped in the heat exchanger system must be purged before starting the first run. After installation of the Q5000 SA is complete, turn on the instrument. Then start the heat exchanger pump by selecting **Control/Prime Exchanger** from the instrument control program or scroll through the **Control command** functions on the touch screen until you come to "Heat Exchanger" and press **Apply**. Refill the coolant reservoir as needed. Repeat this process until all the air has been purged from the system and the instrument stops reporting an error.

Ethernet Switch Setup

In order to connect the instrument to a network, you will need to make the necessary cable connections as described below. The instrument and controller will be connected to an Ethernet switch. In addition, there are instructions for connecting the controller to a LAN.

Connecting the Instrument to the Switch

- 1. Locate the Ethernet port on the left rear of the instrument (shown in the figure to the right).
- 2. Connect one end of the Ethernet cable into the instrument's Ethernet port.
- 3. Connect the other end of the Ethernet cable to one of the network ports on the Ethernet switch (shown in the figure below).



Ethernet Switch

- 4. Check the configuration switches, located on the back panel. They must be set to off, or the up position, for the controller to communicate to the instruments.
- 5. Check the Ethernet port on the rear of the instrument. If communication between the instrument and the switch has been properly established, a solid green light and flashing yellow light will appear at the port.



Green Light

Yellow Light

Configuration Switches

6. Follow the directions in the next section to connect the controller to the Ethernet switch.

Connecting the Controller to the Switch

- 1. Locate the Ethernet port on the back of the computer.
- 2. Plug one end of the Ethernet cable into the computer's Ethernet port (shown in the figure to the right).
- 3. Connect the other end of the cable to one of the network ports on the switch.
- 4. Check the Ethernet port on the rear of the computer. If communication between the computer and the switch has been properly established, a solid green light and flashing yellow light will appear at the port.



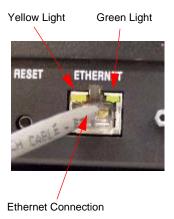
Computer Ethernet Port

5. Follow the directions in the next section to connect the controller to a LAN for networking capabilities.

Connecting the Controller to a LAN

Before you can connect the controller to a LAN, you will need to have already installed a network interface card into the computer.

- 1. Locate the second Ethernet port on the back of the computer.
- 2. Plug one end of the Ethernet cable into the computer's Ethernet port.
- 3. Plug the other end into the LAN.
- 4. Check the Ethernet port on the rear of the computer. If communication between the computer and the LAN has been properly established, a solid green light and flashing yellow light will appear at the port.



Purge Lines

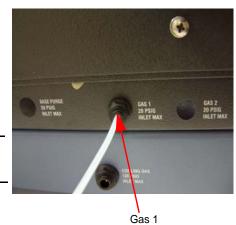
The Q5000 SA uses only nitrogen as the purge gas for the balance housing and for creating the humidified gas that flows to the sample and reference chambers. Hence, the Q5000 SA only has a Gas 1 port on the back of the

instrument for connection to the nitrogen source. In addition, the purge gas cannot be switched during the experiment. The three mass flow controllers (MFC) in the Q5000 SA are used to control the relative humidity atmosphere inside the humidity chamber and provide a dry gas purge to the balance housing. Your TA Instruments service representative may connect the line for you. Instructions are provided here in the event they are needed. Refer to the figure on the right to locate the purge line.



CAUTION: Do not use any liquid in the purge lines. Make sure that you use a dry gas.

Follow these instructions to connect the purge line. Teflon TFE tubing is recommended and is supplied in the instrument shipping accessory kit.

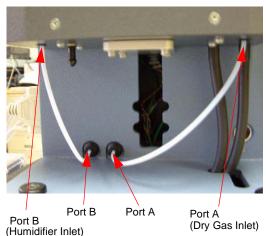


1. Locate the Gas 1 port on the back of the instrument. The Gas 1 port is used for both the balance and humidity

chamber purges. Connect the nitrogen gas line to the Gas 1 port using 1/8-inch o.d. (outside diameter) tubing.

The actual flow rate to the balance and humidity chamber are controlled through settings chosen using the instrument control software.

- 2. Locate Port A on the front of the instrument. Connect the gas line to Port A using 1/8-inch o.d. tubing. Connect the other end of the tubing to Port A on the humidity chamber. See the figure to the right.
- 3. Locate Port B on the front of the instrument. Connect the gas line to Port B using 1/8-inch o.d. tubing. Connect the other end of the tubing to Port B on the humidity chamber. See the figure to the right.



- 4. Make sure that the pressure of your purge gas source is regulated between 70 to 140 kPa gauge (10 to 20 psig) maximum.
- 5. View the connected gas on the **Instrument Preferences/Humidity Page** using the instrument control software. It is set by default to "Nitrogen."
- 6. Set the purge rate to the value needed for your experiments on the **Notes Page** of the **Experiment View**. 200 mL/min is the default setting and is recommended for most experiments. Click **Apply** to save the changes.

NOTE: If you are using laboratory purge, rather than bottled nitrogen purge, it is highly recommended that you install an external drier and a five-micron filter.



CAUTION: Corrosive gases cannot be used with this instrument.



WARNING: Explosive gases should never be used as purge gases.

NOTE: DO NOT connect any gases to the Cooling Gas or Gas 2 ports on the back of the

instrument. They are not used with the Q5000 SA.

Humidity Chamber Serial Cable

To install the serial cable follow these steps:

- 1. Make sure the power cable is unplugged so there is NO POWER to the instrument.
- 2. Connect the serial cable on the side of the humidity chamber to the port on the side of the instrument cabinet as shown in the figure to the right.



Serial Cable

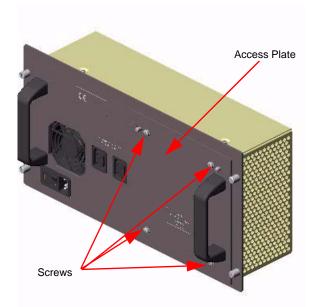
Voltage Configuration Unit

A voltage configuration unit is required if you use 230 Vac, rather than 120 Vac. Follow these steps to install the unit on the Power Control Unit (PCU):



WARNING: High voltages are present in this instrument. Be sure to unplug the instrument before performing these instructions. See the WARNING on page 9.

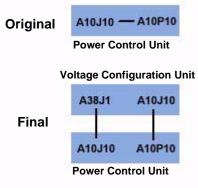
- 1. Remove the contents from the shipping box and verify that all of the components are present.
- 2. Remove the access plate located on the rear of the instrument by removing the four (4) screws that secure it in place. See the figure below.

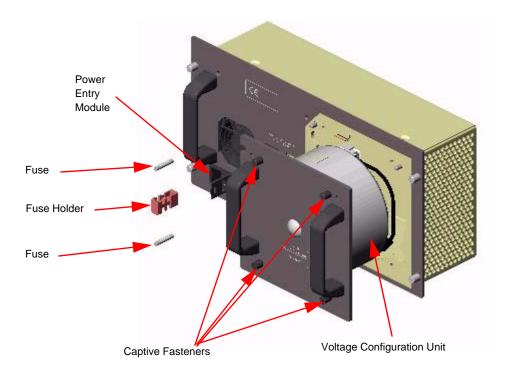


- 3. Disconnect the A10J10 connector from A10P10 located inside the PCU. Now connect the A10J10 connector on the voltage configuration unit to A10P10 located inside the PCU. Then connect A10J10 located inside the PCU to A38J1 on the anti-surge subassembly. See the diagram to the right for clarification.

 Original

 A10J10 A10P10
- 4. Install the subassembly into the PCU and tighten the four (4) captive fasteners to secure it.
- 5. Remove the fuse holder from the power entry module and replace the 10 amp fuses with 6.3 amp fuses, which are supplied in the kit. Discard the 10 amp fuses. See the figure on the next page.



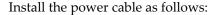


Power Switch

The power switch is located at the rear of the instrument. It is part of the assembly called the *power entry module*, which also contains the power cable connection. The power switch is used to turn the instrument on and off. If a transformer is required, it must be installed before turning on the power.



NOTE: A < HAR>-marked (harmonized) power cable meeting the standards of the country of installation is required for the European Economic Area.

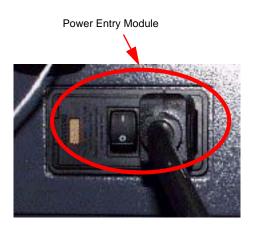


- 1. Make sure the instrument POWER switch is in the Off (0) position.
- 2. Plug the power cable into the instrument power entry module.



CAUTION: Before plugging the instrument power cable into the wall outlet, make sure the instrument is compatible with the line voltage. Check the label on the back of the unit to verify the voltage.

3. Plug the power cable into the wall outlet.



Installing the Q5000 SA

TA Instruments recommends that you complete the installation instructions stated previously in this chapter before you unpack the instrument's balance mechanism. After the instrument has been removed from the box and placed on the marble table, following the instruction sheet found in the packing box, use these steps to install your instrument:

- Remove the shipping bracket
- Unpack the balance
- Start the instrument
- Install the reference hang-down wire
- Install the sample hang-down wire
- Install the humidity close-out disks
- Align the balance
- Close the balance chamber
- Condition the instrument
- Fill the humidity chamber

Removing the Shipping Bracket

This step is also covered in the unpacking instruction sheet shipped with the instrument. It is repeated here for your convenience in the event the step was not completed previously.

Loosen the foot of the shipping bracket shown in the figure below. Raise the foot of the bracket and remove the entire bracket. You will need to pull the bracket and plate off the four posts at the top. Retain this bracket in case the instrument needs to be shipped in the future.



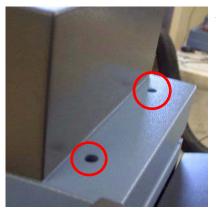


CAUTION: When unpacking the balance in the next section, be careful not to damage the balance arm or hang-down loops.

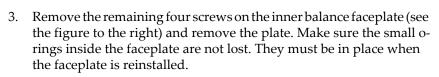
Unpacking the Balance

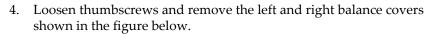
After removing the bracket, you can proceed to unpack the balance. This is a very important procedure to be done before you can use the instrument.

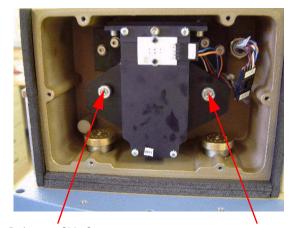
1. Remove the balance housing cover by removing the two screws in the back with a screwdriver. (See the figure below.) Retain the screws. Lift the cover off.



- 2. Remove the four balance shipping L-brackets from the housing using the Phillips screwdriver. Remove the horizontal screws first, then remove the vertical screws. See the figures shows and helesy
 - ures above and below. Retain the brackets and screws in case you need to repack the instrument for shipping in the future.



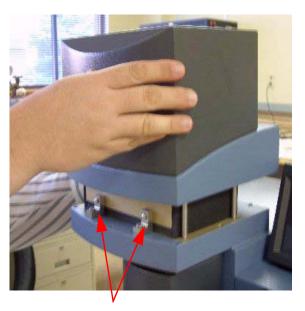




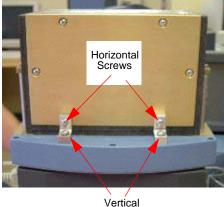
Reference Side Cover

Sample Side Cover

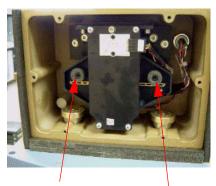
5. Using tweezers, compress the foam and rotate it 90 degrees to eliminate contact with the beam. Gently remove the foam inserts from the sample and reference sides (see figure directly to the right for the location), being careful not to touch the balance.



L-Brackets



Screws



Foam in Reference Side Foam in Sample Side

6. Replace the reference and sample side covers on the left and right side of the balance. Tighten the thumbscrews until they are finger tight.

- 7. Locate the four screws that are taped on the outside of the faceplate, which was removed in step 1.
- 8. Place two of those screws in the back of the balance housing.

NOTE: If the hang-down wires need to be installed, skip steps 9, 10 and 11. The inner balance faceplate and balance housing cover will need to remain off until after the hang-down wires are installed.

- 9. Replace the inner balance faceplate using two of the screws from step 7 and the four screws removed in step 3. Make sure the small o-rings are in position on the inside of the faceplate so that it seats correctly.
- 10. Install the foam insulation from the accessory kit inside the balance assembly cover leaving the bottom open. Press it firmly against the top surface of the balance assembly cover.
- 11. Lower the cover with the foam carefully over the balance housing.

Follow the steps in the next section to power up and start the instrument. If you need to install the hang-down wires, continue with the instructions on the next several pages.

Starting the Instrument

- 1. Check all connections between the instrument and the controller. Make sure each component is plugged into the correct connector.
- 2. Set the instrument power switch to the ON (1) position.

After the proper power up sequence, the TA Instruments logo will be displayed on the touch screen. This indicates that the instrument is ready for use.

NOTE: Allow the instrument to warm up for at least 30 minutes and condition the balance before performing an experiment. See the instrument control online help for further details.

Installing the Hang-Down Wires

After you have finished unpacking the instrument and removing the foam you will need to install the hangdown wires. The same type of hang-down wire is used for both the reference and sample sides of the balance. The procedure is described in this section.

NOTE: If you are performing this procedure after unpacking the balance, the balance faceplate is already off and you will not have to perform steps 1 to 3 below. Begin with the next section.

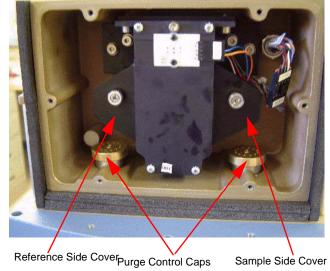


CAUTION: During installation, take care not to bend the hang-down wires or damage the hang-down loops.

- 1. Turn on the instrument as directed. The humidity chamber will automatically lower.
- 2. Remove the screws securing the inner balance chamber faceplate to the instrument and remove the faceplate. Make sure the small o-rings inside the faceplate are not lost. They must be in place when the faceplate is reinstalled.
- 3. Loosen the thumbscrew holding the balance covers on both sides of the balance mechanism and take off the covers. See the figure to the right. Continue with the next section.

Installing the Reference or Sample Hang-Down Wires

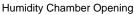
Install the hang-down wires as follows:

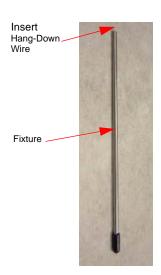


- Locate the hang-down wire and fixture in your accessory Kit.
- 2. Position the hang-down wire so that the double bend hook is at the top. Grasp the hang-down wire with brass tweezers, being careful not to bend it.
- 3. Lower the hang-down wire into the opening in the fixture and turn the double-loop gently to the left.

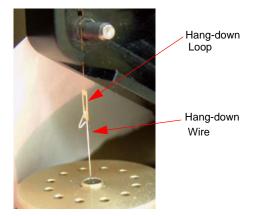
4. Holding the fixture only, angle the fixture and wire so that the bottom goes into the chamber slightly to allow enough space for the top of the wire to clear the humidity chamber without hitting it. See the figure below.







5. Straighten the fixture so that it is vertical and slowly insert the wire up through the hole in the cooling plate being careful not to bend or bow the hang-down wire. Continue to raise the fixture until the hook protrudes slightly from the purge control cap. See the figure below.



- 6. *Optional:* At this point you may wish to place a small piece of white paper carefully behind the hang-down loop in order to see it better.
- 7. Using tweezers, if needed, gently feed the double-bend hook (pointing to the left) through the hole in the hang-down loop. Maneuver the hook until it is fully inserted through the loop.
- 8. Allow the fixture to drop straight down into the furnace, do not try to angle the fixture as you remove it or the wire may bend.
- 9. Carefully remove the fixture and the paper (if used).
- 10. Repeat this procedure for the other hang-down wire located on the reference side. (The same length hang-down wire is used for both sides.)
- 11. Install the Autosampler sample platform (found in the accessory kit). There is a guide pin that will help you orient the platform correctly.
- 12. Select Autosampler Reset from the touch screen or from the instrument control program.

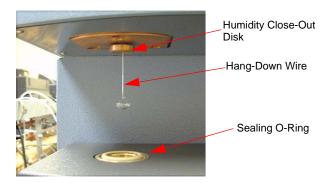
Installing the Humidity Close-Out Disks

The Q5000 SA humidity close-out disks prevent condensation from forming on the top surface of the sample chamber when an experiment is in progress. When you first receive your instrument those disks are not yet installed. Follow these instructions to install the disks:

- 1. Locate the humidity close-out disks (shown in the figure to the right) in your accessory kit.
- 2. Lower the humidity chamber as far as it will go and make sure no pans have been loaded yet on either hang-down wire.
- 3. Position the close-out disk with the stem up as shown in the figure, then slide the disk up over the sample side hang-down wire. Push it up so that the stem seats enough to hold the close-out disk in place, but do not fully seat the disk.



- 4. Repeat this procedure to install the reference side humidity close-out disk.
- 5. Raise the humidity chamber. As the chamber rises to its fully closed position, it will push up against the close-out disks and complete the seating process. When the chamber is opened the disks will hang loosely from the cooling plate as seen in the figure below.



After installation of the humidity close-out disks, you are now ready to align the balance, if desired.

The balance has been aligned at TA Instrument. If needed, you can align the balance as directed in the next section.

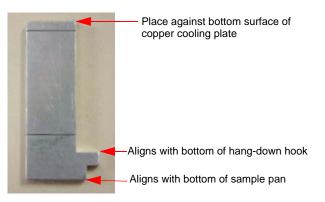
Aligning the Q5000 SA Balance

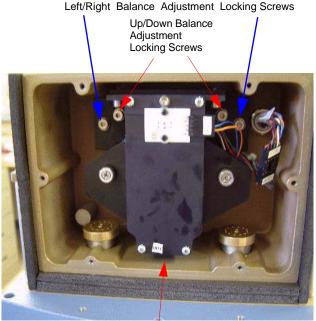
To avoid weight signal noise, the Q5000 SA must be level so that the sample pan and hang-down wire hang inside the humidity chamber without touching the sides. The angle at which the pan hangs is very sensitive to slight irregularities in benchtop surfaces, so it is important that you select a sturdy table or bench for your instrument.

Once you have the instrument in a satisfactory location, you will need to adjust the top and bottom of the sample hang-down wire and level the instrument using the following procedures. These procedures are performed with pans loaded on the hang-down wires.

Aligning the Sample Hang-Down Wire

- 1. Adjust the position of the hang-down wire and pan until the bottom of the pan is 2.2 in. (5.6 cm) from the cooling plate as follows:
 - Loosen the two inner up/down balance adjustment locking screws located inside the balance as shown in the figure to the right.
 - b. Turn the center balance assembly hex head screw until the pan height is at the correct distance. See the figure to the right for the location of the screw. The figure below shows the correct distance for each item. Make your adjustments using the alignment gauge for guidance.





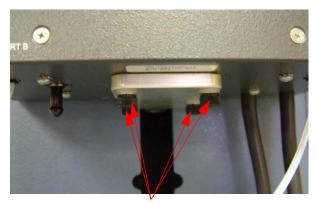
Center Balance Assembly Screw

- c. Tighten the up/down screws to lock them in position.
- d. Turn the hex head screw in step 1b all the way up until it is tight against the balance assembly.
- 2. Adjust the side-to-side position of the sample hang-down wire as follows:
 - a. Loosen the two outer left/right balance adjustment locking screws shown in the figure above.
 - b. Manually position the balance assembly left or right until the hang-down wire is centered in the purge cap at the top and in the sample tube at the bottom.
 - c. Tighten the screws to lock them in position.

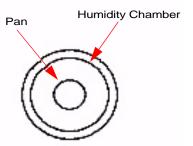
Aligning the Bottom of the Sample Hang-Down Wire

The purpose of this procedure is to center the sample pan within the humidity chamber so that its movement is not hindered as the humidity chamber is opened and closed. See the figure to the right.

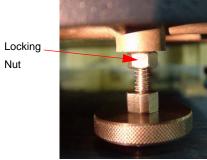
- 1. Use the touch screen Calibrate/Autosampler/Motor Test/Furnace Up/ Down functions to slowly raise the humidity chamber just to the bottom of the sample pan, and touch STOP.
- 2. Check the alignment of the sample pan within the humidity chamber. It should hang freely, roughly centered, and should not be touching the sides of the humidity chamber (shown in the figure here).
- 3. If the sample pan is not centered and hanging freely within the humidity chamber, level the instrument by adjusting the two front feet on the bottom. Turn the feet clockwise to lengthen or counterclockwise to shorten the legs making sure the feet and the stabilizer bar contact the table securely. Continue adjusting until the pan hangs correctly.
- 4. Use a 7/16-inch wrench to tighten the locking nuts up against the bottom of the cabinet when the instrument is level to fix the position of the mounting feet in place. See the figure to the right.
- 5. In some cases, minor adjustments of the humidity chamber itself must be made. This is accomplished by loosening the four screws that connect the chamber to the lift arm (shown in the figure below), moving the chamber until the pan is centered and then tightening the screws.



Humidity Chamber Adjustment Screws



Pan should be centered within the humidity chamber when viewed from above.



5. Lower the humidity chamber then manually remove the pan the place it back on the tray.

Closing the Balance Assembly

After you have finished the procedures on the previous pages, follow these instructions to complete the installation of the Q5000 SA:

NOTE: If the balance faceplate is already on, you will not have to perform step 1. Begin with step 2.

- 1. Replace the inner balance faceplate and secure with six screws. (Four screws were removed during step 3 on page 34 and two were shipped separately in a plastic bag that was taped inside the cover.) Make sure the small o-rings are in position on the inside of the faceplate so that it seats correctly.
- 2. Obtain the balance housing foam insert from the accessory kit.
- Install the foam insulation from the accessory kit inside the balance assembly cover leaving the bottom open. Press it firmly on the inside top surface of the balance assembly cover. See the figure to the right.
- 4. Lower the cover with the foam carefully over the balance assembly. Replace the two screws on the rear of the cover.
- 5. Install the Autosampler cover. The installation procedure is now complete.



Conditioning the Balance & Humidity Chamber

Balance Conditioning

Balance conditioning is required for the Q5000 SA in order to achieve optimum performance from your instrument and keep your balance housing dry. Balance conditioning is needed when the instrument is first installed and whenever the balance housing is opened.

Temperature Stabilization

The recommended balance housing temperature is 35°C. Allow the temperature of the meter movement to stabilize for one hour during installation or after the balance housing was opened. During this time the weight measurement will show some drift as the magnet strength in the balance changes with temperature.

Drying the Balance

After the temperature of the meter movement has stabilized, you will need to dry the balance with nitrogen at 100 mL/min for 12 hours. Moisture in the balance housing must be eliminated to maximize long term stability of the weight signal. It takes a considerable amount of time to fully remove the moisture that has been adsorbed by the small amount of polymeric material in the suspended balance components.

Maintaining a Dry Balance

To maintain a dry balance after conditioning, use a balance purge rate of 10 mL/min.

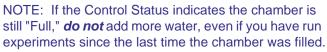
NOTE: If the balance has been opened to the atmosphere for more than a few minutes (e.g., during hang-down wire replacement) and very long experiments are to be run requiring the ultimate stability of the weight measurement, the instrument should be left purging at 100 mL/min overnight.

Filling the Humidity Chamber with Water



CAUTION: Distilled water is required for the Q5000 SA humidity chamber to prevent mineral buildup. <u>Do not add any conditioner to the distilled water</u>.

- 1. Using the touch screen on the instrument, select the Control Screen.
- 2. Using the Control Command pull-down menu on the Control Screen, select: "Humidity Fill Mode."
- 3. Select **Apply** to enable Humidity Fill Mode. Wait until the message at the top of the screen says "Ready" before proceeding.





4. Remove the rubber cap from the water fill fitting on the left side of the humidity chamber shown in the figure to the right.



- 5. Using the syringe (see the figure below), **slowly** add distilled water to the humidity chamber until a beep is heard from the instrument. When the chamber is full, the Control Status on the display will change to "Full."
 - NOTE: There is a time delay associated with the FULL sensor. It will take several seconds for the sensor to register that the chamber is full and to activate the display and beep. Therefore, fill the chamber slowly to avoid overfilling.
- 6. Press **Apply** to disable Humidity Fill Mode when complete. NOTE: You will <u>not</u> be able to

run subsequent humidity experiments if the instrument is still in the Fill Mode.

7. Replace the rubber cap on the water fill fitting. NOTE: You will <u>not</u> be able to obtain reasonable humidity results if this cap is not replaced.

NOTE: You will need to allow sufficient time for the water to equilibrate to the temperature of interest before proceeding with an experiment. Because the water in the humidity chamber should be at temperature equilibrium, it is generally not desirable to add water *during* an experiment.

Shutting Down the Instrument

Before you decide to power down your instrument, consider the following:

- All of the components of your thermal analysis system are designed to be powered on for long periods.
- The electronics of the Q5000 SA and the controller perform more reliably if power fluctuations caused by turning units on and off are minimized.

For these reasons, turning the system and its components on and off frequently is discouraged. Therefore, when you finish running an experiment on your instrument and wish to use the thermal analysis system for some other task, it is recommended that you leave the instrument on with a 10 ml/min balance purge/

To ensure proper shutdown of the instrument, it is recommended that you select **Control/Shutdown Instrument** from the Instrument Control menu or select **Shutdown** from the touch screen Control options. A confirmation message will be displayed. Select OK (touchscreen) or Shutdown (Instrument Control) to proceed. All communication to the instrument will be halted while the instrument saves data to the flash memory. Once this procedure is complete, the instrument will post a message indicating that it is safe to turn off the power to the instrument or reset the instrument.

To power down your instrument set the power switch on the rear of the instrument to the OFF (0) position.

Chapter 3

Use, Maintenance, & Diagnostics

Using the Q5000 SA

All of your Q5000 SA sorption analysis experiments will have the following general outline. In some cases, not all of these steps will be performed. The majority of these steps are performed using the instrument control software. The instructions needed to perform these actions can be found in the online help in the instrument control program; therefore, they will not all be covered in detail here.

- Calibrating the instrument
- Creating or choosing the test procedure and entering experiment information through the TA instrument control software
- Selecting and taring the sample pan
- Loading the sample
- Starting the experiment
- Unloading the sample at the end of the experiment.

To obtain accurate results, follow the procedures carefully and check calibration or validate performance at least once a month.

Before You Begin

Before you set up an experiment, ensure that the Q5000 SA and the controller have been installed properly. Make sure you have:

- Made all necessary cable connections between the Q5000 SA and the controller (ethernet connections)
- Connected heat exchanger power and water lines
- Connected all gas lines
- Powered on each unit
- Become familiar with controller operations
- Conditioned and calibrated the Q5000 SA, if necessary.

Calibrating the Q5000 SA

To obtain accurate experimental results you should calibrate the instrument when you first install it. For the best results, however, you should validate performance periodically and recalibrate, if necessary.

The following calibrations are performed on the Q5000 SA:

- Weight calibration
- Temperature calibration
- Humidity (flow) calibration
- Autosampler calibration
- Touch screen calibration

NOTE: You can perform all of these calibrations except temperature and humidity calibrations. See the following sections for information. Calibration of temperature requires a high accuracy external meter and must be performed by TA Instruments Service personnel. Humidity (flow) calibration also requires specialized external devices and must be performed at the factory. Therefore, if humidity calibration issues are detected, the Mass Flow Controller (MFC) assembly must be replaced.

Weight Calibration

Weight calibration is required when you first install the system. This calibration can be performed through the instrument control program (Calibrate/Weight) using the certified 50 mg weight found in the Q5000 SA accessory kit. The calibration can also be performed through the Platinum functions using the certified 100 mg matched pan set, also found in the accessory kit. See online help for more information.

Periodic validation of weight calibration can be performed using either the instrument control program or Platinum functions and the same 50 mg or 100 mg matched pan certified standards.

Temperature Calibration

Temperature calibration on the Q5000 SA is performed at the factory before the instrument is shipped. At installation your TA Instruments Service representative will verify this calibration at 35°C using a highly accurate, certified external meter.

Humidity (Flow) Calibration

The Q5000 SA uses an "open loop" arrangement for generating and measuring the humidity level in the chamber around the same and reference. The humidity is generated by controlling the ratio of gas passing through two calibrated Mass Flow Controllers (MFCs). These Mass Flow Controllers are calibrated at the factory prior to shipment using a certified external flow measurement device. Proper calibration is verified at installation (and can be periodically verified by the operator) by running deliquescent salts. TA Service uses sodium bromide, which deliquesces at 57.6 ± 2 %RH (absolute) at 25° C for verification.

The humidity sensors located near the sample and reference pans provide an ongoing indication of humidity and provide further confirmation of proper humidity calibration.

Autosampler Calibration

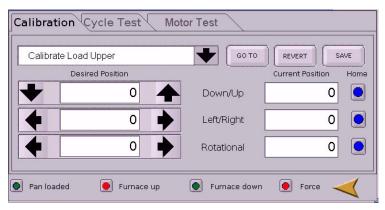
If the Autosampler fails to pick up a sample pan correctly during an automatic loading procedure, the Autosampler may need to be calibrated. The calibration procedure is performed at installation and should be done periodically thereafter, as needed.

Other possible causes for improper pan pickup include:

- The instrument is not level. See "Aligning the Balance" for instructions to solve this problem.
- The hang-down wire is not straight. Replace the hang-down wire. See "Installing the Hang-Down Wires" for instructions.
- The bails are bent, be sure to use straight pan bails.

To perform Autosampler calibration follow the instructions below.

- Touch the Calibrate button at the bottom of the touch screen. The screen shown in the figure to the right will be displayed.
- 2. Press the **Autosampler** button.
- Touch the Calibration tab, if it is not already selected. The screen shown in the figure below is displayed.





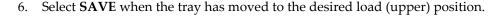
 Follow the instructions below to adjust the two Autosampler pan positions "Calibrate Load Upper" or "CalibrateLoad Lower."

In addition to calibration, two other functions are available: *TGA Autosampler Cycle Test*, which allows you to test load/unload and punch functions, and *TGA Autosampler Motor Test*, which allows you to test the individual motors. See the online help for information.

Calibrate Load Upper Position

This calibration is used to adjust the position of the sample tray as it slides across to load a sample pan.

- 1. Place a pan in the number 1 position of the sample tray.
- 2. Select "Calibrate Load Upper" from the **Calibration** page on the touch screen.
- 3. Select the **GO TO** button. The pan will be moved to the load position.
- 4. Observe the position of the bail and the hang-down wire hook when the pan stops. The hook should be in the center of the bail's angled handle and should not touch the bail so that the hook will not pull off the pan when the tray moves.
- 5. Touch the directional arrows, followed by the **GO TO** button, to move the desired position for the tray up/down, left/right, or clockwise/counterclockwise until the tray is correctly positioned.





Calibrate Load Lower Position

This calibration is used to adjust the position of the sample tray so that the tray can move out of the way without interfering with the pan when the pan hangs from the hang-down wire hook.

- 1. Load the pan from in the number 1 position of the sample tray.
- 2. Select "Calibrate Load Lower" from the **Calibration** page on the touch screen.
- 3. Select the **GO TO** button.
- 4. Observe the motion of the tray. The tray should move down and clear the bottom of the pan without touching it.
- 5. Touch the directional arrows to move the tray up/down, left/right, or clockwise/counterclockwise until the tray is correctly positioned.
- 6. Select SAVE when the tray has been moved to the desired unload (lower load) position.

NOTE: The left/right and rotational positions will be saved to both the **Calibrate Load Lower** and **Calibrate Load Upper** windows. Therefore, you will automatically save the values to both places.

Running a Q5000 SA Experiment

Experimental Procedure

All of your Q5000 SA experiments will have the following general outline. In some cases, not all of these steps will be performed. See the instrument control software online help for anything not covered in this manual.

- Filling water reservoir in humidity chamber
- Taring the empty sample pan.
- Loading the sample into the pans. Elimination of static electricity effects.
- Entering experiment information through the TA controller, this includes both sample and instrument information.
- Creating or selecting the experimental procedure using the instrument control software.
- Starting the experiment.

Filling the Water Reservoir

Before initiating a sorption experiment (or a sequence of experiments using the Autosampler), it is important to check the water level in the humidity chamber reservoir. If the Control Status display indicates "OK" or "LOW," fill the chamber using the procedure on page 43. In addition, review the topic called "About the Humidity Chamber" in online help (select **Help/Help Topics** from the menu and use the Contents to locate the topic). This topic provides additional information about verification of sufficient water for the experiments being run, as well as information on software safeguards that prevent Autosampler experiments from starting if the water reservoir is low.

Typically it is not a good idea to add water to the reservoir in the middle of an Autosampler sequence. However, if the experiments are being run at 25°C, water can be added, particularly during the early portion of an equilibration step, without affecting results.

Taring the Sample Pan

Taring must be done before the sample is loaded to ensure that the balance gives you an accurate reading of sample weight.

Place an empty sample pan on the platform and select **TARE** from the Q5000 SA Control Menu touch screen or select **Control/Tare** from the instrument control software. Select the desired pan on the tray. The pan will automatically be loaded and the humidity chamber raised to make the measurement. When the tare procedure is complete, the humidity chamber will automatically lower and unload the pan.

NOTE: A sample pan of the same size and type is required on the tare side of the balance for proper operation.

Loading the Sample

See online help for details concerning sample preparation. After taring the sample pan, load the sample into the Q5000 SA humidity chamber as follows:

1. Place the sample in the sample pan, and position the pan on the sample tray at the same position used for the tare. (This may be done with the tray on or off the instrument.) The bail of the pan should align with the line etched on the tray so that the sample pan can be picked up by the sample hang-down wire.

NOTE: Always use brass tweezers to handle the sample pans.

NOTE: Some materials readily build up static electricity charges that can create problems during loading. This phenomenon is most likely to occur in experiments where the ambient humidity is low. The metal-coated quartz pans supplied with the Q5000 SA should eliminate this problem by completing a grounding path to the balance housing.

Replace the sample tray on the Autosampler, if needed, and place the plastic cover over the samples, if desired.

You can preweigh the samples prior to the start of the experiment, if desired, in order to obtain a record of the weight. A preweight is valuable for highly volatile materials. This weight is stored within the data file record.

NOTE: Samples that might gain or lose weight, (based on adsorbing moisture or losing volatiles respectively) while sitting in the Autosampler tray awaiting analysis, are best analyzed using sealed aluminum pans, which are punched just prior to analysis.

Sample Size

The Q5000 SA balance is very sensitive to small weight changes. Therefore, it is not necessary to use large samples, particularly if the sample gains more than 10% of its original weight during exposure to humidity. A sample size of 5-10 mg is sufficient. A slightly larger sample size (25-30 mg) is recommended if the total weight gain is less than 0.5%. Generally smaller samples will allow more rapid equilibration with the humidity being used. Smaller samples are also recommended when performing ramped humidity or ramped temperature experiments.

Creating an Experimental Procedure

The Q5000 SA instrument control software allows a variety of sorption analysis experiments to be run, including: stepped humidity at constant temperature, ramped humidity at constant temperature, stepped temperature while holding humidity constant, and ramped temperature while holding humidity constant. The software contains templates that simplify setting up these various types of experiments. See "Available Q5000 Test Templates" in the online help for further details.

Starting an Experiment

Before you start the experiment, ensure that the Q5000 SA is online with the controller and you have entered all necessary information through the instrument control software.

Start the experiment by touching the **START** key on the instrument touch screen or by selecting **Start** on the instrument control software. When you start the instrument, the system automatically loads the sample pan and closes the humidity chamber if necessary, and then runs the experiment to completion.

Stopping an Experiment

If for some reason you need to discontinue the experiment, you can stop it at any point by touching the **STOP** key on the Control menu touch screen or by selecting Stop through the instrument control software. Another function that stops the experiment is **Reject**. However, the **Reject** function discards all of the data from the experiment; the **Stop** function saves any data collected up to the point at which the experiment was stopped.

NOTE: See Chapter 2 for information on shutting down the instrument.

Plotting Results

When plotting Q5000 SA results, either Time or Relative Humidity (%) are almost always used as the X-axis signals. The Sample Sensor RH (%) or Reference Sensor RH (%) provide an indication of the humidity in the area of the sample and reference pans during an experiment, but are generally not used as the X-axis because those values are not as accurate at the Relative Humidity (%) signal.

In addition to direct plots of the raw experimental signals, the Universal Analysis program provides many other useful ways to treat/view the results. For example, the "initial sample weight" can be used for weight change measurements or the "weight after initial drying" can be used. Analyses such as BET and GAB are also available.

Maintaining the Instrument

The primary maintenance procedures described in this section are the customer's responsibility. Any further maintenance should be performed by a representative of TA Instruments or other qualified service personnel. Consult the online documentation installed with the instrument control software for further information.



WARNING: Because of the high voltages in this instrument, untrained personnel must not attempt to test or repair any electrical circuits.

Cleaning the Instrument

You can clean the Q5000 SA touch screen as often as you like. The touch screen should be cleaned with a household liquid glass cleaner and soft cloth. Wet the cloth, not the touch screen with the glass cleaner, and then wipe off the touch screen and surrounding surfaces.



CAUTION: Do not use harsh chemicals, abrasive cleansers, steel wool, or any rough materials to clean the touch screen as you may scratch the surface and degrade its properties.

Cleaning/Drying the Quartz Pans

The Q5000 SA quartz sample pans are designed to be reusable. However, test materials that remain from previous sorption experiments must be removed prior to reuse of the sample container for subsequent experiments.



CAUTION: The quartz pans can not be cleaned with a propane torch since quartz may soften and become misshapen with exposure to the high temperatures of a flame.

Since many of the materials evaluated by sorption analysis are water soluble, first attempt to clean the quartz sample pans by flushing the pans with HPLC grade or distilled/deionized water. Rinse the quartz pans after flushing using methanol, ethanol, or isopropyl alcohol and air dry them for 15 to 20 minutes. You can use a Q-tip swab to remove excess alcohol before air drying. **Do not use detergents** for cleaning the quartz pans.

If water does not remove the residue, you can use mild organic solvents such as acetone. The solvent method may need to be combined with short duration ultrasonic cleaning. However, this is a last resort since mechanical agitation could break or deform the quartz sample pans.

Once clean, the sample pans should be stored in a desiccator until ready for use.

Maintaining the Heat Exchanger

The heat exchanger does not require any maintenance other than to maintain the level and quality of the liquid coolant. If the level drops too low, or the coolant becomes contaminated, this could result in problems with your instrument.



CAUTION: Do not put any liquid other than distilled water and TA Conditioner in the heat exchanger reservoir.

You should check the level and condition of the heat exchanger coolant periodically. We recommend routine checks every three to six months, depending on use of the instrument.

Add distilled water to the reservoir, if necessary, to keep the reservoir at least 2/3 full. If algae growth is visible, drain the reservoir bottle, refill it with distilled water, and add TA Instruments TGA Q5000 Conditioner. See page 25 for instructions on filling the heat exchanger.

Monitoring the Humidity Chamber

The water reservoir within the humidity chamber will require periodic refills with distilled water. When the chamber is low, a message will appear in the status line. Follow the instructions on page 42 to fill the chamber again. The SA has several water level sensors built into the humidity reservoir that indicate "full," "OK" (broad range between "full" and "low") and "low."

NOTE: The "low" indicator initially triggers when approximately 40 mL are left in the reservoir.

The water reservoir in the Q5000 SA holds approximately 150 mL of water when full. The rate of consumption of that water during experiments is, of course, dependent on temperature, %RH requested, and time. The dominant factor, however, is temperature. At 25°C, the rate of consumption is low, even if high humidities are used. At 25°C it takes more than 30 days for the reservoir to be depleted. This is more than enough time to accommodate the analysis of a full Q5000 SA autosampler tray (10 samples). At extreme conditions (*e.g.*, isothermal at 85°C and 85 %RH), on the other hand, the rate of water consumption is much higher. Hence, it takes only about 15 hours for depletion of the reservoir. This should be enough time to perform at least one stability experiment.

NOTE: It is good practice to always fill the water reservoir prior to starting an Autosampler sequence of experiments. Water can be added in the middle of an experiment or Autosampler sequence provided it is added at a point in the experiment/sequence where no critical data is being taken. Depending on the temperature of the humidity chamber at the time of water addition, it might also be beneficial to add warm water rather than water at ambient conditions.

Replacing Fuses

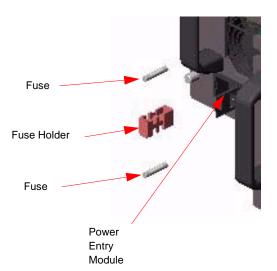


WARNING: Always unplug the instrument before you examine or replace the fuses.

The Q5000 SA contains internal fuses that are not user serviceable. If any of the internal fuses blow, a hazard may exist. Call your TA Instruments service representative.

The only fuses that you can replace yourself are the fuses located in the power entry module located at the rear of the instrument. To check or change these fuses:

- 1. Turn the instrument off and remove the power cord.
- 2. Insert a small screwdriver at the edge of the power entry module door and pry it open.
- 3. Insert the screwdriver on the edge of the fuse holder to pull it out of the instrument.
- 4. Remove old fuses and replace the fuses only with the type and rating indicated on the instrument's rear panel.
- 5. Place fuse holder back into opening and push the door shut.



Replacement Parts

Fuses, Cords, and Cables

Part Number	Description
205221.001 205221.002 251470.025 253827.000 920223.901	Fuse (6.3 amp, 250 V) for 230 Vac operation with the Voltage Configuration unit Fuse (10 amp, 250 V) for 120 Vac operation Ethernet cable (25 foot, unshielded) Power cord Event cable

Q5000 SA Accessories

Part Number	Description
920163.901	Power Control Unit
259508.000	Brass tweezers
259509.000	Spatula, curved, 165 mm long
271621.001	O-ring, furnace housing to balance chamber
269920.004	Balldriver, 5/64-inch
269920.026	Balldriver, 7/64-inch
269920.005	Balldriver, 3/32-inch
952162.901	Heat Exchanger tubing
952377.901	Heat Exchanger conditioner kit
953160.901	TGA Heat Exchanger assembly
957331.901	Hang-down loop
957082.901	Sample hang-down wire
200391.001	Wrench, double angle, 1/4-inch
200392.001	Mirror, adjustable, 7/8-inch diameter
957357.001	Gauge, position, furnace/hook/pan
957156.901	Humidity Sensor (package of 2)
957323.901	Humidity Close-out disks

Q5000 SA Sample Pans and Accessory Kits

Part Number	Description
957216.901	10-pan Tray, Autosampler
957210.903	$180~\mu L$ metalized quartz hemispherical pans (package of 3)
957099.901	25-pan Tray, Autosampler
957207.904	$100~\mu L$ platinum sample pans (package of 3) 1
957387.902	$100 \mu L$ platinum-HT sample pans (package of 3) ¹
957363.901	$80~\mu\text{L}$ aluminum sample pans (package of $100)^{1,2,3}$
957362.901	Aluminum sample lids (package of 100)
957364.901	Stainless steel bails (package of 15)
957352.901	Sealed aluminum pan kit, which contains:
	957362.901 Aluminum sample lids (package of 100)
	957363.901 80 μ L aluminum sample pans (package of 100) ^{1,2,3}
	957364.901 Stainless steel bails (package of 15)
	957358.001 Sealed pan punch alignment tool
	957201.001 Sealed pan crimping tool

¹ Requires 25-pan tray, PN 957099.901.

Q5000 SA Calibration/Reference Materials and Kits

Part Number	Description
200413.001	Calibration weight 50 mg - Class 1
957450.901	Humidity reference material, sodium bromide
957400.901	Autocal weight kit for Q5000 SA

Requires several additional parts found in Sealed Aluminum Pan Kit, PN 957352.901. Refer to online help for full details.

³ Requires TA Instruments blue sample press, PN 900878.902.

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