

Agilent Electrothermal Temperature Controller ETC 60

User's Guide



Notices

Manual Part Number

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Safety Notices

CAUTION

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

WARNING

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

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Manufactured

The Electrothermal Temperature Controller (ETC) is manufactured by:

Anatech BV
Nieuwstadterweg 3
6136 KN Sittard (the Netherlands)

Verifying Safe State

The following general safety precautions must be observed during all phases of operation, maintenance, and service of this instrument.

To ensure continued safety of the instrument after maintenance or service procedures, verify the instrument is returned to a safe state for the user. This includes running performance checks to verify the instruments safety systems are functioning correctly. Check the general condition of the instrument during operation for wear or signs of corrosion that are likely to inhibit function or safety.

Failure to comply with these precautions or with specific warnings elsewhere in this manual violates safety standards of design, manufacture, and intended use of the instrument. Agilent Technologies assumes no liability for the customer's failure to comply with these requirements.

General

Operation of the Agilent ETC 60 electrothermal temperature controller and an atomic absorption (AA) spectrometer involves the use of an electrically heated quartz cell, high temperatures, compressed gases, and hazardous materials, including corrosive fluids. Unskilled, improper, or careless use of the equipment can create fire hazards or other hazards which can cause death, serious injury to personnel, or severe damage to equipment and property.

Appropriate safety practices have been included in this operation manual and your spectrometer operation manual, to help you operate the equipment safely. Read all safety practices thoroughly before attempting to operate your system.

If the equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired.

Hazard Warnings

In addition to the hazard warnings specified in your spectrometer operation manual, specific hazard warnings have been included in this operation manual. These warnings state the hazard, describe how to avoid it and specify the possible consequences of not following the instructions. Read all warnings carefully and observe them at **all** times.

Electrical Hazards

The ETC 60 contains electrical circuits, devices and components operating at dangerous voltages. Contact with these circuits, devices and components can cause death, serious injury or painful electric shock. Covers which are retained by screws on the ETC 60 may be opened only by an Agilent field service engineer.

The ETC 60 **MUST** be switched off at the power switch, and the mains cable disconnected from the mains supply **BEFORE** any attempt is made to remove or replace the cell in the workhead, or the cables connecting the workhead to the Temperature Control Module.

Safety Practices and Hazards

Use of the wrong supply voltage, connection of the accessory to an incorrectly wired supply outlet, or lack of proper electrical grounding can create a fire hazard or a shock hazard which can cause death, serious injury, or serious damage to equipment.

Always use a three-wire outlet with ground connection which is adequately rated for the load.

The installation must comply with local, state, and national safety regulations.

Ensure that the voltage selector is correctly set for the specific mains power supply outlet that the ETC 60 will be connected to. You must do this before connecting the accessory.

A blown fuse should be replaced with one of the size and rating stipulated in the text adjacent to the fuse holder. Always ensure the accessory is turned off and disconnected from the mains supply before attempting any replacement.

Chemical Hazards

Operation of an Agilent AA spectrometer may involve the use of materials that are corrosive or otherwise hazardous. Careless, improper or unskilled use of these materials can cause serious personal injury or an environmental hazard.

Always ensure that laboratory safety practices governing the use, handling and disposal of hazardous materials are strictly observed. These safety practices should include wearing the appropriate safety clothing.

Always ensure that the sample introduction system is thoroughly flushed with distilled water and that the contents of the drain or waste container are known and disposed of if necessary before touching any part.

Notes and Tips

A Note or Tip message is used to give advice or additional information.

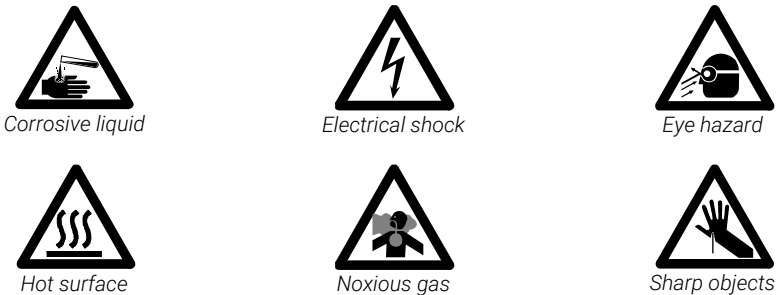
NOTE

This is a note.

Warning Symbols

The following is a list of symbols that appear in conjunction with warnings in this manual and on the ETC. The hazard they describe is also shown.

A triangular symbol indicates a warning. The meanings of the symbols that may appear alongside warnings in the documentation or on the instrument itself are as follows:



The following symbol may be used on warning labels attached to the instrument. When you see this symbol, refer to the relevant operation or service manual for the correct procedure referred to by that warning label.



Information Symbols

The following symbols appear on the instrument for your information.

I	Mains power on
0	Mains power off
	Fuse
	Single phase alternating current
	Caution, disconnect all supplies, risk of electric shock

Color Coding

The various indicator lights appearing on the instrument and any associated accessories have been color coded to represent the status of the instrument or accessory:

- A green light indicates the instrument is in normal standby condition.
- A blue light indicates that operator intervention is required.
- An orange light indicates a potential hazard.
- A red light indicates danger or an emergency.

Safety Practices and Hazards

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Introduction

The Agilent ETC 60 replaces the air-acetylene flame as a heat source to atomize the analyte hydrides in hydride generation methods of analysis.

The running costs of the ETC 60 workhead are about 50% lower than those incurred in flame heating of the tube, due to the elimination of the costs of flame gases.

The ETC 60 workhead may also be used on Zeeman graphite furnace systems as the heat source to atomize analyte hydrides.

The ETC 60 consists of a Temperature Control Module and a workhead containing a quartz absorption tube. It is designed to operate with an Agilent AA Series spectrometer and the Agilent VGA 77 vapor generation accessory.

If the instrument is used in any manner not specified by Agilent, the protection provided by the equipment may be impaired.



Figure 1. Installed ETC with VGA on a Flame (left) and Zeeman (right) instrument

Unpacking

Open the shipping case and remove the enclosed packages with care. Carefully open the packages and inspect all parts for damage in transit. Report damaged or incorrect items immediately.

In addition to this manual, you should have received one of each of the following items:



Figure 2. Workhead



Figure 3. Adapter for flame instruments

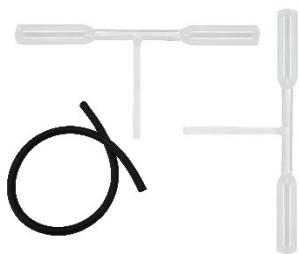


Figure 4. Pack of cells



Figure 4. Temperature control module

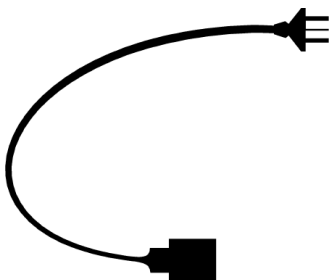


Figure 5. Power cable

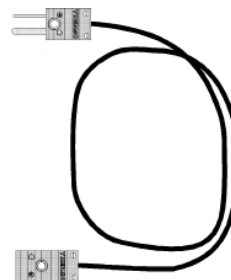


Figure 6. Temperature sensor cable



Figure 7. Zeeman models workhead bracket



Figure 8. Zeeman models workhead bracket adapter

Temperature Control Module

The Temperature Control Module monitors and regulates the temperature of the quartz tube. A safety system is incorporated to ensure that the tube temperature does not exceed 1025 °C. On start-up the system checks that the temperature sensor is working correctly.

The front panel consists of a two-line display and five keys. The display keys correspond to menu items on the display.

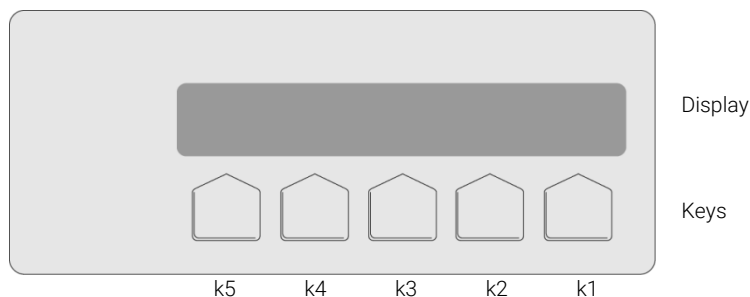


Figure 9 Front panel of the ETC 60

Mounted in the back panel are the following connectors:

- Mains power inlet with mains power switch and fuse holder, which also acts as the mains voltage selector
- Computer connection (serial interface, RS-232)
- Thermocouple input (temperature sensor)
- Power outlet to the workhead (cell heater)

Getting Started

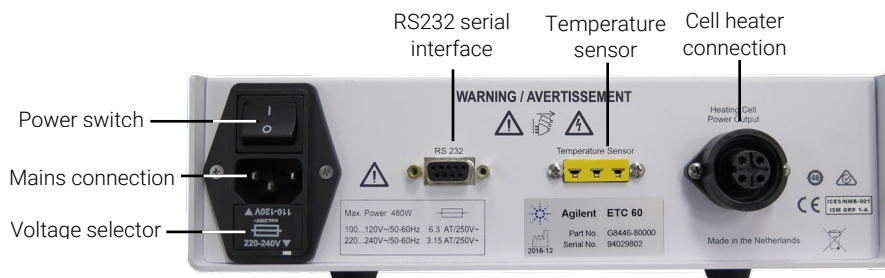


Figure 10 Rear panel of the ETC 60

Workhead

The workhead consists of a stainless-steel case containing:

- A quartz absorption tube
- The heating element
- The temperature sensor

WARNING



Electrical Shock

The workhead is connected to mains power. Contact with components operating at mains potentials can cause death, serious injury or harmful electric shock. Only open the workhead when the ETC Controller is disconnected from the mains power supply.

The heating element is a specially designed high-temperature filament. The temperature sensor is in direct contact with the quartz tube, to enable a rapid temperature response. The quartz cell, heating element, and temperature sensor are insulated by inert alumina and silica wool.

WARNING



Sharp Objects

Broken glass can cause severe cuts to hands. Handle the workhead with care, as the sidearm of the quartz tube is fragile and easily broken.

If the quartz cell is broken, it must be replaced. Replacement cells are available from Agilent (two per pack).

CAUTION

The supply cord to the workhead must only be replaced by a manufacturer approved part.

Pre-Installation

Before proceeding with the installation, you must be familiar with the 'Safety Practices and Hazards' section and the correct power requirements must be available (refer to Appendix A).

Position the instrument for easy access to the power switch or disconnecting device.

Allow at least 50 mm (2 in.) of space on the sides, and 150 mm (6 in.) at the rear of the system to permit free air circulation.

Voltage Selector

A combined voltage selector/fuse holder is located in the rear panel of the Temperature Control Module, immediately below the mains power supply socket, as shown in Figure 11. The ETC 60 operates on 100–120 VAC, 220–240 VAC (50/60 Hz). It is shipped with 220–240 V selected.

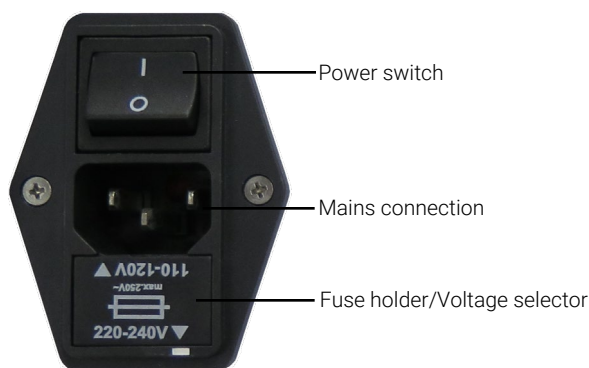


Figure 11. Power switch, mains connection, and fuse holder/voltage selector

Do not replace power cord with one of a lower rating than specified. Use only Agilent supplied power cord for your country.

Check that the correct voltage is selected for your laboratory's electricity supply, for example, in Figure 11 the current voltage range is 220–240 V. The selected voltage is indicated by the triangles that line up along the lower edge of the fuse holder. If necessary, change the voltage selector by removing the fuse holder and replacing it in its alternate orientation.

Getting Started

To change the voltage setting:

1. Ensure that the mains power is off and the power cable is removed.

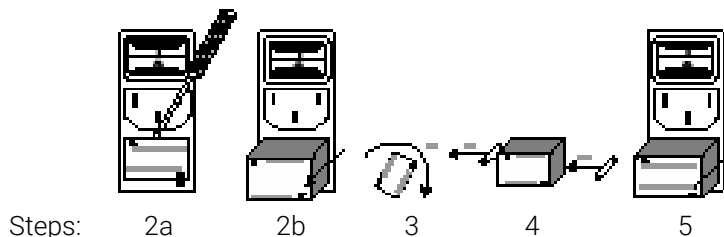


Figure 12. Changing the voltage selection

2. Remove the fuse holder/voltage selector from the rear panel:
 - a Place the tip of a small flat-blade screwdriver into the slot located just under the center earth pin on the mains power input connector. Gently lever the screwdriver to partly remove the holder/selector.
 - b Remove the holder/selector by sliding it out of the panel.
3. Rotate the holder/selector so that the required voltage range is at the bottom.
4. Remove the fuse from the upper left side of the fuse holder, and place it into the socket on the right side of the holder.
5. Replace the holder/selector into its socket, so that the triangles on the lower edge of the holder/selector line up and indicate the selected voltage range.

Installation Overview

While the individual steps differ during the installation of the ETC 60 for the Flame and Zeeman models, the general procedure is the same:

- Install the workhead bracket
- Install the workhead
- Install the absorption cell
- Install the VGA

Start on Page 19 for the ETC 60 Workhead Bracket installation on Flame instruments or on Page 22 for the ETC 60 Workhead Bracket installation on Zeeman instruments.

WARNING



Hot surface

Before starting the installation procedure, ensure the instrument has had ample time to cool down to avoid burning your hands.

Installing the Workhead Bracket (Flame Models)

To install the workhead bracket:

1. Remove the upper and lower flame shields from the spectrometer.



Figure 13. Removing upper (left) and lower (right) flame shields

2. Remove the chimney.



Figure 14. Removing the chimney

Getting Started

3. If it is fitted, remove the burner from the spraychamber.



Figure 15. Removal of burner (in flame models)

4. Remove the spraychamber and store it in the stand provided.

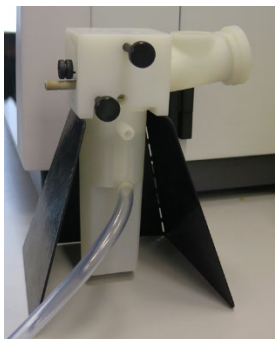


Figure 16. Spraychamber in stand

5. Loosen the two heat shield screws.



Figure 17. Heat shield screws

Getting Started

6. Remove the heat shield by lifting it up and out of the sample compartment.

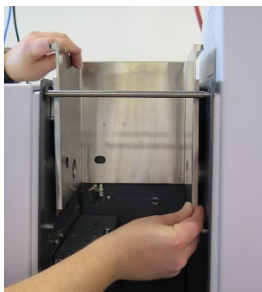
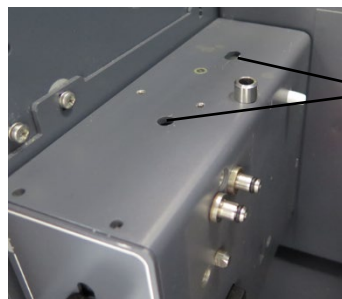


Figure 18. Heat shield removal (flame models)

7. Optimize the hollow cathode lamp signal in your instrument. Search for "Aligning lamps" in the SpectrAA Help. Open "Aligning Lamps (55B/240/280 AA)" for instructions.
8. Fit the two lugs on the workhead bracket into the two holes on top of the burner adjuster.



Lugs



Holes

Figure 19. Preparing to fit the workhead bracket



Bracket hole for workhead

Bracket

Bracket support

Burner adjuster

Thumb screw (not shown)

Figure 20. Workhead bracket fitted to the burner adjuster

Getting Started

9. Screw the thumbscrew through the bracket support and into the side of the burner adjuster to hold the bracket in place.

WARNING



Sharp Objects

Broken glass can cause severe cuts to hands. Handle the workhead with care, as the sidearm of the quartz tube is fragile and easily broken.

10. Install the heat shield and secure it with the two screws.
11. Go to 'Installing the Workhead' on Page 23.

ETC 60 Workhead Bracket Installation on Zeeman Instruments

Additional steps must be performed before installing the ETC workhead bracket on a Zeeman instrument.

1. Remove the PSD 120 autosampler (if present)
2. Remove the Zeeman workhead from the instrument.

Refer to the 240/280 instrument user manuals for detailed instructions.

Cell Holder and Adapter Removal

The standard cell holder must be removed to allow installation of the ETC 60 Workhead.

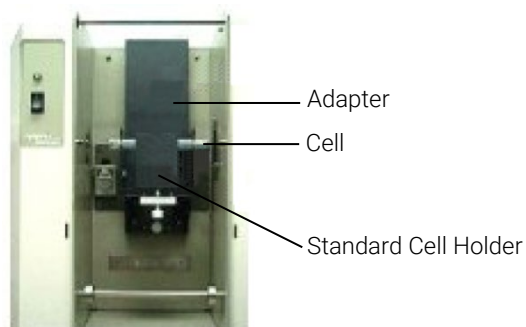


Figure 21. Adapter, standard cell holder and cell inside sample compartment

1. Lift the cell holder up to unhook cell holder from the adapter mounting.
2. Lift the adapter out of the sample compartment carefully avoiding the crossbar.

Getting Started

Installing the Workhead Bracket Adapter and Bracket

1. Lower the hooks of the ETC Bracket onto the pivot points of the bracket adapter.

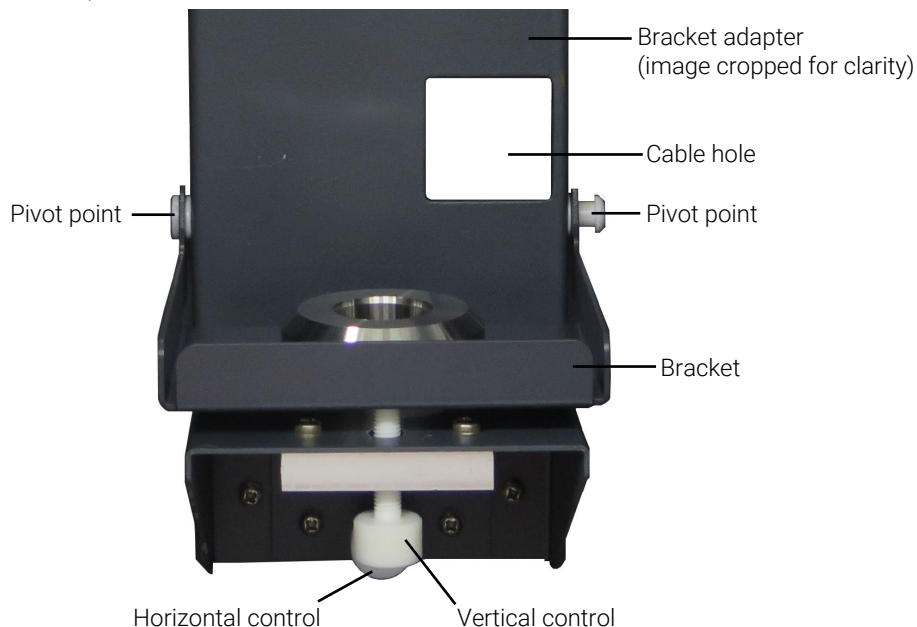


Figure 22. Workhead bracket adapter fitted with bracket

2. With the Zeeman Workhead removed, hook the adapter fitted with the ETC 60 bracket on the top rear edge of the sample compartment. See Figure 25 on Page 24.
3. Continue to 'Installing the Workhead' on Page 23.

Installing the Workhead

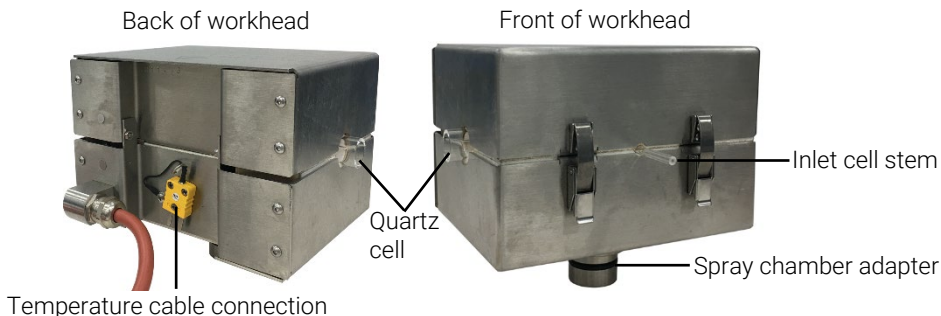


Figure 23 The ETC 60 workhead

Getting Started

To install the workhead:

1. Connect the temperature sensor cable to the workhead.



Figure 24. Temperature cable connected to the workhead. An older style cell heater cable is shown.

NOTE

The pins on the connector are sized differently and will only fit one way.

2. Fit the ETC 60 workhead into the hole in the bracket and push down firmly to seat it in the bracket. See Figure 20 on Page 21.

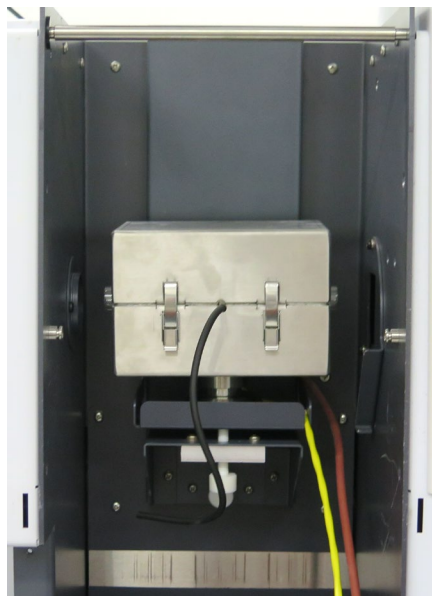


Figure 25. ETC 60 workhead installed (Flame instruments left and Zeeman instruments right)

Getting Started

3. Ensure the two cable curves down to the rear right hand side of the sample compartment and then out towards the front of the AA.

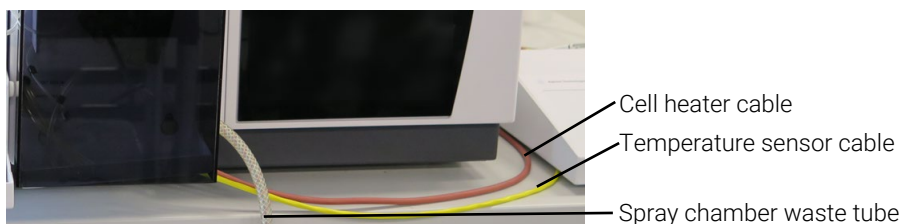


Figure 26. ETC 60 cable layout

CAUTION

The cables must be lying naturally with no strain on them; otherwise the workhead may be pulled out of alignment.

4. Plug the cell heater and temperature sensor cables into the appropriate sockets in the rear of the Temperature Control Module.

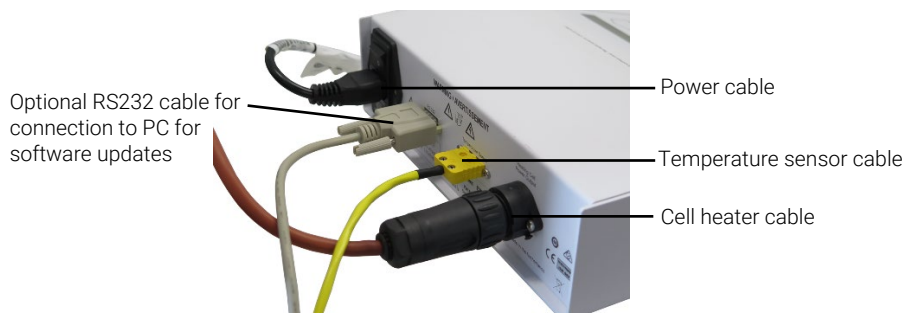


Figure 27. Cable connections on the back of the ETC 60

5. Ensure the ETC power switch is in the OFF position.
6. Plug in the power cable and optional RS232 cable (if required).

NOTE

The RS 232 connector on the back panel of the control module is used to download software upgrades to the ETC 60. Plug in the RS 232 cable (option) to this connector and connect the other end of the cable to your computer. This connection allows direct control of the temperature settings from within the SpectraAA software, version 5 or later.

7. Optimize the workhead position for maximum light transmission by using the burner adjustment controls on the burner adjuster for flame instruments or the adapter bracket for Zeeman instruments.
8. Check the rotational alignment of the workhead by rotating it to maximize the signal.

Getting Started

Installing an Absorption Cell

To install an absorption cell:

1. Carefully slide the black fluoroelastomer tubing onto the inlet cell stem.



Figure 28. Black fluoroelastomer tubing on the inlet cell stem

WARNING



Sharp Objects

Broken glass can cause severe cuts to hands. Handle the workhead with care, as the sidearm of the quartz tube is fragile and easily broken.

2. Undo the latches on the ETC 60 workhead cover and open the cover.
3. Carefully place the cell into the workhead. Ensure that the inlet stem is located in the groove at the front of the workhead.

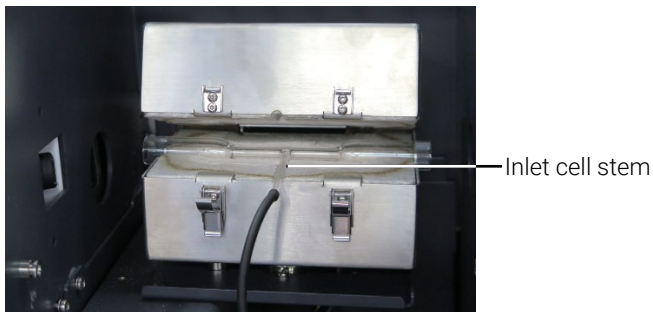


Figure 29. Cell placed in workhead

4. Carefully close the workhead cover and secure the latches.
5. If necessary, reoptimize the workhead position using the burner adjuster controls.

Getting Started

Installing the VGA

1. Mount the VGA on to the instrument as outlined in the manual that ships with the accessory.
2. Carefully connect the other end of the black fluoroelastomer tubing on the inlet cell stem to the outlet of the VGA gas-liquid separator.

The system is now ready for use.

Getting Started

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3 Operation

Power Up	30
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WARNING



Noxious Gas – Corrosive Liquid – Eye Hazard

Vapor generation methods of analysis involve the generation of toxic hydrides and require the use of materials which are toxic, highly corrosive or otherwise hazardous. Careless, improper, or unskilled use of such materials can cause serious personal injury.

Always ensure that laboratory safety practices governing the use, handling, and disposal of such materials are strictly observed. These safety practices should include wearing of appropriate safety clothing.

In the VGA 77 system, highly concentrated acids are being pumped under pressure. If a leak occurs, these acids could cause serious personal injury. Always wear approved safety glasses warranted to protect the eyes. Never pump concentrated sulfuric acid through the VGA.

The ETC replaces the air-acetylene flame as a heat source to atomize the analyte hydrides. When using the Agilent ETC 60 you will need to select the ETC-60 option in the **Vapor mode** parameter on the **Type/Mode** tab of the **Method Edit** page.

Turn all instrument gases off at the cylinders. Leave only the inert supply to the VGA 77 operating.

The instructions contained in this section provide general operating procedures when using the ETC 60. For temperature settings for specific elements, refer to the 'Analytical Notes' section. For details on caring for the workhead, refer to the 'Maintenance' section

When the ETC 60 is operating, the quartz tube glows red. Most of this glow is caused by reflected light from the heater, although the sidearm does become warm.

Operation

NOTE

The reflected light interferes with the operation of the flame sensor, preventing operation of the AA instrument. Tape a burner cleaning card over the sensor hole to prevent the reflected light interfering with the flame sensor.

TIP

The flame sensor is the smaller of the two holes, situated on the right side of the sample compartment of an Agilent 200 Series AA spectrometer.

CAUTION

Remember to remove the card when determinations with the ETC have been completed. Do not cover the flame sensor for conventional flame operation. If ignition is attempted with the card in place the flame will light but will be extinguished when the ignite button is released.

Power Up

Ensure that the ETC 60 is correctly installed as previously detailed, turn on the power switch to power up the module. The ETC 60 will perform a self-test that takes a few seconds. If all functions are operating correctly, the display will show the following:

```
M V1.30 ETC-60 temperature controller
```

After about half a second, the display will change:

```
MAIN MENU ETC-60
                run   setup
```

The ETC 60 is now ready for normal operation.

For normal operation with SpectrAA software, use the RS-232 cable to connect the ETC 60 temperature control module to the serial port of the controlling computer. Control of the module will be taken over by the SpectrAA software and the display will show:

```
REMOTE                                temp: +925 °C
```

Operation

NOTE

If you are using Version 4 (or earlier) of SpectrAA software, remove any connection between the ETC 60 and a computer. Earlier versions of the SpectrAA software do not support control of the ETC 60.

Error on Startup

If the ETC 60 encounters an error in the embedded software in the control module, the display first shows the following:

```
*** SELFTEST ***
```

Followed by the boot software version number:

```
ETC-60 B 1.3
```

And finally:

```
Standby
```

The above sequence of displays indicates that the ETC 60 is unable to be operated normally due to the error. New software can be downloaded to the ETC 60 from a PC by the serial interface. Contact Agilent for more information or to obtain assistance from an Agilent field service engineer.

NOTE

If the temperature control module is switched on without the thermocouple connected, the ETC 60 will go into a permanent error state when the self test is complete. To clear this error: switch off the module, connect the thermocouple cable, and power up the ETC 60 temperature control module again.

Making Selections

The ETC 60 is menu driven by the keys beneath the display. Available options are indicated on the lower line of the display. In the Main menu, there are two choices: run and setup, and these choices are selected by pressing keys k3 or k2 respectively.

Operation

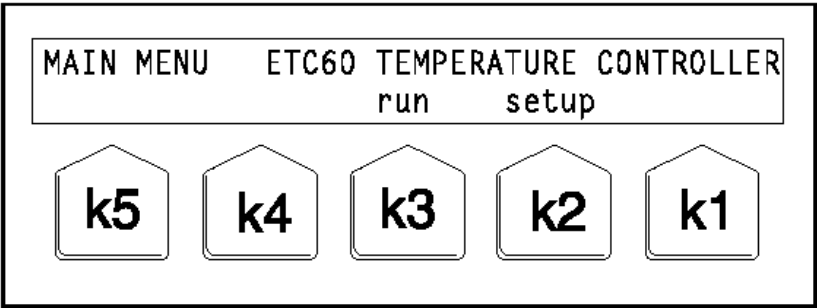


Figure 30 Main menu

Common Menu Options

Some items are common to more than one menu:

- back – return to previous menu
- OK – accept displayed values and return to previous menu
- start – start the method
- +/- – increase/decrease the selected parameter

Methods

With the ETC 60, you can run the following types of methods:

- Predefined isothermal
- Manual isothermal
- Manual step

Predefined Isothermal Methods

The following standard methods are predefined in the ETC 60 (all are for 15 minutes) for use with earlier versions of SpectrAA software:

Table 1 Predefined isothermal methods

	As	Sb	Bi	Se	Sn	Te
°C	925	850	850	850	900	900

Operation

NOTE If you are using Agilent SpectrAA software, version 5 or later, the ETC 60 downloads the appropriate temperature from the computer.

To select a predefined method from the main menu, select `run`, then `method`. Some of the elements will be shown. To see other methods, select `>>>`.
Select the key corresponding to the method in the display. The display will show the parameters of the method. For example, selecting the `As` method for arsenic would display:

METH	As	T_start	Time	23 °C
stop	925 °C	15:00	start	

The actual cell temperature is displayed in the upper right corner. If you want to change the start temperature or time, press the `k4` or `k3` key respectively, then change the parameters according to the 'Changing Settings' section, Page 34.

NOTE Any changes to a method are saved automatically, even if the temperature control module is switched off.

To start the method, select `start`. The display changes:

METH	As	T_start	Time	15:00	138 °C
stop		925 °C	15:00	hold	

When the start temperature is reached the time will start to count down. Key `k1` is now a toggle switch to `hold` or `restart` the countdown for a running method.

NOTE You cannot `hold` until the workhead is at the isothermal temperature.

You can now make hydride generation measurements in accordance with the instructions in the VGA operation manuals.

Manual Isothermal Method

To select this method, from the main menu select `run`, then `manual`, then `iso`.
Operation is the same as for a predefined isothermal method.

Manual Step Method

This consists of a start temperature, end temperature, a temperature step size, and a time during which each temperature step will be maintained.

Operation

To select this method, from the main menu select `run`, then `manual`, then `steps`. The following is displayed:

STEPS	T_start	T_end	time/step	23 °C
stop	800 °C	900 °C	10:00/10 °C	start

You can change the settings as described in the 'Changing Settings' section.

NOTE

Any changes to a method are saved automatically, even if the temperature control module is switched off.

The end temperature (T_end) may be higher or lower than the start temperature (T_start). If the program is running, the time for each next step begins to run directly after completion of the previous step, so the time is already running while the cell is heated to the set temperature of the next step. The last step in the step program is just large enough to reach the end temperature.

Changing Settings

To change a setting, select the key corresponding to the parameter you want to change. You will see the change menu:

OK	+	-
----	---	---

To change the setting, press and hold the appropriate key.

When changing temperatures, the change will be in units until a multiple of 10 is reached. The change will then be in tens until a multiple of 100 is reached. The change is then in hundreds. Release the key to restart the change in single units.

When changing times, the change will be in seconds until a multiple of a minute is reached, then the change will be in minutes.

Select OK to accept the settings.

Unattended Operation

As there is no flame present, you can use the AA spectrometer, VGA 77, ETC 60, and an autosampler for unattended operation.

CAUTION

When performing unattended operations ensure that all waste connections are securely mated and that waste containers are large enough to accommodate all of the waste that will be generated during the unattended run period.

When an AA system is operated unattended, the VGA should be powered through a timer socket. When the timer elapses, all power to the VGA is turned off, shutting down the peristaltic pump of the VGA and saving reagents.

NOTE

Even when the power switched off, approximately 50 mL/min of inert gas will flow through the VGA.

Timer sockets are available from most commercial electrical or department stores. You should carefully check that the equipment connected to the timer socket does not exceed the rated capacity of the socket.

When calculating the required setting for a timer switch, be sure to consider the time taken for on-going instrument calibration. The operational time can be calculated and a further time added to this (15–30 minutes). The switch off time can then be set on the timer.

Shutdown Procedure

To shut down your AA system:

- 2 Turn off the ETC 60.
- 3 Leave the VGA 77 on. Place the uptake capillary tubes in distilled water and leave the VGA running for five minutes to flush the system.
- 4 Remove the uptake tubes and let the VGA pump air for one minute. Turn off the VGA and release the pump beds. Refer to the VGA 77 manual for details.
- 5 Turn off the inert gas.

WARNING



Hot Surface – Electrical Shock

The workhead will be very hot, and contact with it could cause severe burns. Always ensure that the workhead is allowed to cool before attempting to remove it from the sample compartment.

The ETC 60 **MUST** be switched off at the power switch, and the mains cable disconnected from the mains supply **BEFORE** any attempt is made to remove or replace the workhead, or the cables connecting the workhead to the Temperature Control Module.

4 Analytical Notes

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The procedures for preparation of samples, standards, and reagents are the same as when using an air-acetylene flame. The spectrometer operating conditions will also be the same as when using an air-acetylene flame, except the **Vapor mode** parameter should be set to ETC-60, when using this accessory.

The ETC 60 permits atomization of analyte hydrides at the appropriate temperature for each analyte. The temperature of the electrically heated quartz tube is generally lower than that of the flame heated quartz tube, and so the residence time of atoms in the quartz tube is longer than when a flame is used. This results in greater residence times for the atoms in the light path, resulting in an increase in sensitivity. Typical improvements in sensitivity of around 20% or more can be expected, depending upon the analyte.

Table 2 Optimum temperatures for specific elements

Element, Symbol	Temperature (°C)
Arsenic, As	950 (refer to Page 38)
Antimony, Sb	900
Bismuth, Bi	850
Selenium, Se	850
Tellurium, Te	850
Tin, Sn	900

Arsenic

TIP

Use the 189.0 nm line and add 1% oxygen to the inert gas flow to improve sensitivity for arsenic determinations. Add the oxygen directly before the hydride vapor enters the tube.

Mercury

The flow-through mercury cell will not fit inside the workhead of the ETC 60. If mercury determinations are being carried out using the ETC 60, the standard flow through the cell must be used.

NOTE

When using the ETC 60 for mercury determinations there may be a slight loss of sensitivity in comparison to measurements made using the flow-through mercury cell.

WARNING



Noxious Gas

Mercury (Hg) vapor is highly toxic. Always ensure that laboratory safety practices governing the use, handling, and disposal of mercury-containing materials are strictly observed. These safety practices should include ensuring adequate ventilation to minimize exposure to the vapor and adherence to local environmental regulations regarding its subsequent release into the atmosphere.

If the ETC 60 is used for the determination of mercury, the Temperature Control Module should either be turned off or set to a temperature of 0 °C — unless condensation starts to occur.

NOTE

If condensation forms inside the cell, program the ETC 60 to operate at a constant temperature of approximately 100 °C. You can set the optimal isothermal temperature to suit your analytical requirements but never exceed 300 °C when analyzing for mercury.

Optimal Temperature Determination

To determine the optimum temperature for your analysis:

1. Set up the instrument and install the ETC 60 as described in the 'Getting Started' section.
2. Set the temperature to 300 °C (100 °C for mercury) and wait for the ETC 60 to reach the temperature.
3. Go to the **Instrument** parameters page, and set the **Measurement time** to read 30 s, four replicates, and **Delay time** to 0 s.
4. Set the signal graphics to display 0–250 sec and an Absorbance range of –0.20–1.00 A.
5. Pump a solution of the analyte (10–20 ppb) through the system.
6. Press **Read** and monitor the signal graphics. When a stable signal has been achieved, increase the temperature by 50 °C and then monitor the signal graphics.

NOTE

Do not exceed 300 °C when optimizing for mercury.

7. Repeat Step 6 until the signal strength does not increase with increase in temperature.

The lowest temperature at which the maximum stable signal is obtained is the optimum temperature setting for your analysis.

Analytical Notes

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Daily Maintenance

Due to the use of corrosive reagents in the vapor generation technique, the panels of the ETC 60 Temperature Control Module must be cleaned at the end of daily use with a soft cloth dampened with mild detergent solution. It is not necessary to clean the workhead.

Replacing the Fuse

WARNING



Electrical Shock

To prevent reduced safety protection or unwanted fusing, a blown fuse should be replaced with one of the size and rating stipulated in the text adjacent to the fuse holder.

Always ensure the accessory is turned off and disconnected from the mains supply before attempting any replacement.

To replace the fuse:

- 1 Remove the fuse holder from the rear panel of the module as described in the 'Voltage Selector' section, refer to Page 17.
- 2 Replace the blown fuse with a new one.
- 3 Replace the holder/selector into its socket, so that the triangles on the lower edge of the holder/selector line up and indicate the selected voltage range.

Quartz Absorption Cell

The quartz absorption cell of the ETC 60 has a finite lifetime, which is dependent upon the atomization temperature used and the effects of reagents and sample matrices. Since the quartz cell of the ETC 60 is heated under controlled conditions, an improvement in tube lifetime may result when compared to flame heating. Typically the quartz cell will last between 300–1000 hours, depending upon analytical conditions.

The lifetime of a quartz cell can be extended:

- Cut the connecting tube from the gas-liquid separator to the quartz cell, and insert a small plastic Y-connector. Close the tail of the 'Y' with a tube and laboratory clip. Any spray or foam carried over by the gas stream from the separator impinges on the walls of the 'Y' as the gas suddenly changes direction. The resulting liquid drains into the 'Y' tail. Typically, less than 0.5 mL will be collected during 8 hours of operation.

The best indication of whether a quartz cell has reached the end of its lifetime is sensitivity. You should keep a record of cell sensitivity for each of the elements you determine, and update it at the start of each analysis. An example of a table for tracking sensitivity is provided in Appendix C.

The quartz absorption cell inside the workhead is replaceable. Once the cell fails, or its sensitivity is affected, it should be replaced. Agilent recommends that you always have spare cells available in case of breakage

Replacing the Absorption Cell

WARNING

Hot Surface – Electrical Shock



The workhead will be very hot, and contact with it could cause severe burns. Always ensure that the workhead is allowed to cool before attempting to remove or replace the cell.

The ETC 60 **MUST** be switched off at the power switch, and the mains cable disconnected from the mains supply **BEFORE** any attempt is made to remove or replace the cell.

To replace the absorption cell:

- 1 Ensure that the mains power is off and the power cable is removed.

Maintenance

-
- 2 If the ETC 60 has been operating, allow the workhead to cool down to ambient room temperature.

WARNING



Sharp Objects

Broken glass can cause severe cuts to hands. Handle the workhead with care, as the sidearm of the quartz tube is fragile and easily broken.

-
- 3 Carefully disconnect the black fluoroelastomer tubing from the inlet of the absorption cell.
 - 4 Undo the latches on the ETC 60 workhead cover and open the cover.
 - 5 Remove the absorption cell from the workhead.
 - 6 Carefully place the replacement cell into the workhead. Ensure that the inlet stem is located in the groove at the front of the workhead.
 - 7 Carefully close the workhead cover and secure the latches.
 - 8 Reconnect the black fluoroelastomer tubing from the outlet of the VGA gas-liquid separator to the inlet cell stem taking care not to shift the workhead position. If necessary, reoptimize the workhead position using the burner adjuster controls.
 - 9 Reconnect the mains power cable to the ETC 60 temperature control module.

The system is now ready for use.

Removing the Workhead

WARNING



Hot Surface – Electrical Shock

The workhead will be very hot, and contact with it could cause severe burns. Always ensure that the workhead is allowed to cool before attempting to remove it from the sample compartment.

The ETC 60 **MUST** be switched off at the power switch, and the mains cable disconnected from the mains supply **BEFORE** any attempt is made to remove or replace the workhead, or the cables connecting the workhead to the Temperature Control Module.

Maintenance

Removal of the workhead is the reverse of the installation procedure outlined in 'Installing the Workhead' section, Page 19.

6

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Appendix A – Specifications

Dimensions (w × d × h):	25.5 × 26 × 8 cm
Weight:	5 kg
Sample temperature range:	Ambient–1025 °C
Temperature precision:	±1 °C
Power consumption:	550 VA max
Fuse rating:	100–120 VAC, T6.3AH250V; 220–240 VAC, T3.15AH250V
Thermocouple:	Type K (NiChrome/Nickel)
Operating voltage:	100–120 VAC, 220–240 VAC
Frequency:	50/60 Hz
Mains power connector:	Three-pin male (standard IEC type)

Environmental

Condition	Altitude	Temp (°C)	Humidity (%RH) non-condensing
Non-operating (transport)	0–2133 m (0–7000')	5–45	20–80
Operating within performance specifications	0–853 m (0–2800')	10–35	8–80
	853–2133 m (2800–7000')	10–25	8–80

Appendices

For optimum analytical performance, it is recommended that the ambient temperature of the laboratory is between 20–25 °C and held constant to within ± 2 °C throughout the entire working day.

Appendix B –Errors

There are two types of errors: warning and fatal:

- Warnings will be displayed on the screen for three seconds and the ETC 60 will continue to operate normally.
- Fatal errors messages remain on the display, the power to the cell is disconnected, and the ETC 60 will cease operating.

When either a warning or a fatal error occurs, the ETC 60 will beep.

To clear fatal errors, switch the ETC 60 off. If the error recurs, contact your Agilent field service engineer.

Warning Messages

`WAR_ALL_OTPROM`

Checksum error in the nonvolatile memory where the calibration data is stored. The default calibration data is reloaded and this might slightly decrease the temperature accuracy.

Please check with your Agilent field service engineer.

`WAR_ALL_DFLT_EEPROM`

Checksum error in the nonvolatile memory where methods, LCD contrast and LCD backlight are stored. The default settings are restored. Check settings and change if necessary, then switch the ETC 60 off then on again.

If the error persists, contact your Agilent field service engineer.

`WAR_ALL_I2C_RD_EEPROM` or `WAR_ALL_I2C_RD_EEPROM`

Error during reading or reprogramming nonvolatile memory. This will not affect normal operation, but settings may be lost when the ETC 60 is switched off.

`WAR_ALL_WATCHDOG`

A watchdog reset has occurred. After the warning the ETC 60 will be in the normal 'power on state. This should only occur after a software hang-up. For example; following a large mains power spike.

`WAR_ALL_AMB`

Ambient temperature measurement too high or too low. Ambient temperature is

Appendices

used for the thermocouple cold junction and for LCD contrast control. Check the operating environment of the ETC 60.

Fatal Errors

ERR_T_HIGH

Measured temperature of the cell assembly is too high. Possible cause is a defective power circuit.

ERR_HARDWARE

General error in the main printed circuit board.

ERR_AMB

Ambient temperature is out of range for correct operation, or the ambient temperature measurement is faulty. Check the operating environment.

ERR_T_HIGH_OR_TH_CPL or ERR_THERMO_CPI

Probably a disconnected or faulty thermocouple cable. Switch off the ETC 60, check the thermocouple cable, and then switch the ETC 60 on. If the error recurs, the thermocouple may be faulty.

Appendix C – Sensitivity Test Record Sheet

Solution (concentration):

Matrix:

[illegible]

Appendix C – Sensitivity Test Record Sheet (cont.)

Solution (concentration):

Matrix:

[illegible]

Appendices

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In This Book

The manual describes the following (list all chapter headings):

- Safety Practices and Hazards
- Getting Started
- Operation
- Analytical Notes
- Maintenance
- Appendices

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