

# **Agilent Electrothermal Temperature Controller ETC 60**

## **User's Guide**



**Agilent Technologies**

## Notices

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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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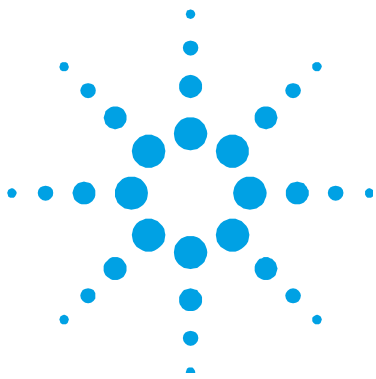
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### Manufacture

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.

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.

## Safety Practices and Hazards

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 and Caution Messages

A Warning message is used in the text when failure to observe instructions or precautions could result in death or injury.

---

#### WARNING



#### Name of Warning

Detail of hazard. Information on how to avoid the hazard.

---

A Caution message is used when failure to observe instructions could result in damage to equipment (Agilent supplied and/or other associated equipment).

#### CAUTION

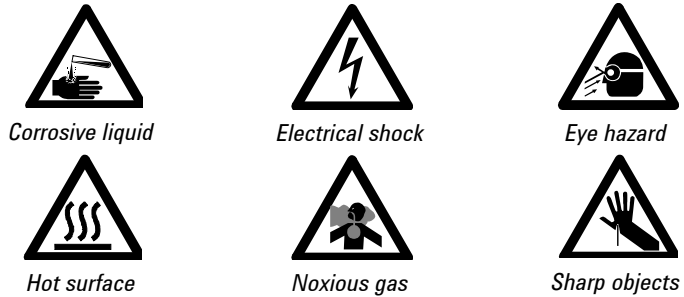
This is a caution statement.

---

## 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.

<b>I</b>	Mains power on
<b>0</b>	Mains power off
	Fuse
	Single phase alternating current

### 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.

### CE Compliance

Your ETC 60 has been designed to comply with the requirements of the Electro-magnetic Compatibility (EMC) Directive and the Low Voltage (electrical safety) Directive (commonly referred to as the LVD) of the European Union. Agilent has confirmed that each product complies with the relevant Directives by testing a prototype against the prescribed EN (European Norm) standards.

Proof that a product complies with the Directives is indicated by:

- The CE Marking appearing on the rear of the product.
- The documentation package that accompanies the product, containing a copy of the Declaration of Conformity. This Declaration is the legal declaration by Agilent that the product complies with the Directives, and also shows the EN standards to which the product was tested, to demonstrate compliance.

### Electromagnetic compatibility

#### EN55011/CISPR11

**Group 1 ISM equipment:** group 1 contains all ISM equipment in which there is intentionally generated and/or used conductively coupled radio- frequency energy which is necessary for the internal functioning of the equipment itself.

**Class A equipment** is equipment suitable for use in all establishments other than domestic and those directly connected to a low voltage power supply network which supplies buildings used for domestic purposes.

This device complies with the requirements of CISPR11, Group 1, Class A as radiation professional equipment. Therefore, there may be potential difficulties in ensuring electromagnetic compatibility in other environments, due to conducted as well as radiated disturbances.

Operation is subject to the following two conditions:

- 1 This device may not cause harmful interference.
- 2 This device must accept any interference received, including interference that may cause undesired operation.

If this equipment does cause harmful interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try one or more of the following measures:

- 1 Relocate the radio or antenna.
- 2 Move the device away from the radio or television.
- 3 Plug the device into a different electrical outlet, so that the device and the radio or television are on separate electrical circuits.
- 4 Make sure that all peripheral devices are also certified.
- 5 Make sure that appropriate cables are used to connect the device to peripheral equipment.
- 6 Consult your equipment dealer, Agilent Technologies, or an experienced technician for assistance.

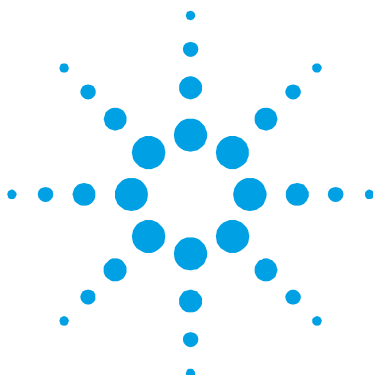
Changes or modifications not expressly approved by Agilent Technologies could void the user's authority to operate the equipment.

### **ICES/NMB-001**

This ISM device complies with Canadian ICES- 001.

Cet appareil ISM est conforme à la norme NMB-001 du Canada.

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## 2. Getting Started

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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 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.

### 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.

## Getting Started

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



**Figure 1:** Workhead



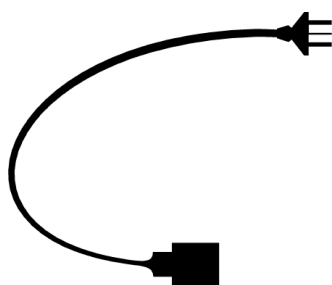
**Figure 2:** Adapter for flame instruments



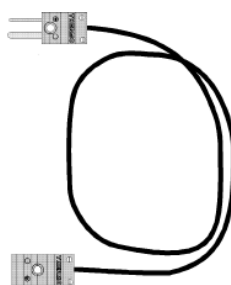
**Figure 3** Pack of cells



**Figure 4** Temperature control module



**Figure 5** Power cable



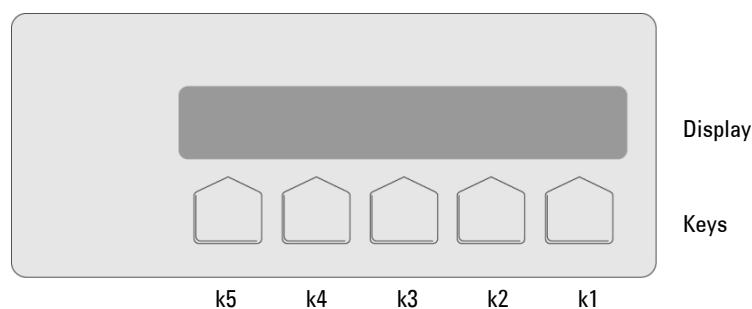
**Figure 6** Temperature sensor cable



## 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.

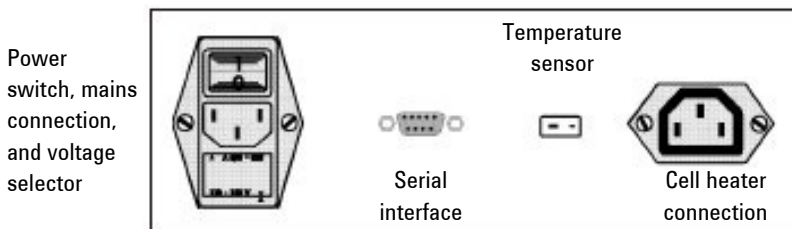
The front panel consists of a two-line display and five keys. The display keys correspond to menu items on the display.



**Figure 7** 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)



**Figure 8** 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 unit 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.

---

## Installation

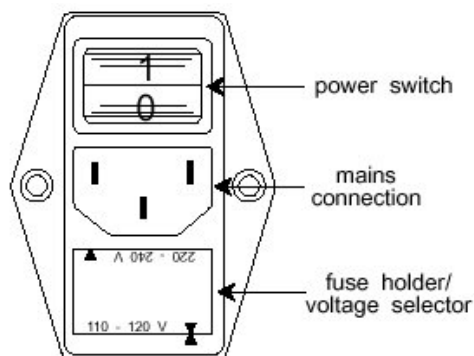
Before proceeding with the installation, you must be familiar with the ‘Safety Practices and Hazards’ section and the correct power requirements must be available (refer to Appendix A).

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 9. The ETC 60 operates on 100–120 VAC, 220–240 VAC (50/60 Hz). It is shipped with 220–240 V selected.



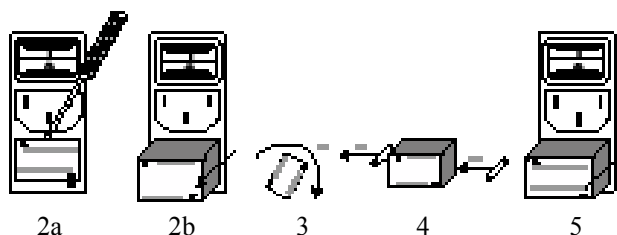
**Figure 9** Power switch, mains connection, and fuse holder/voltage selector

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 9 the current voltage range is 110–120 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.

### To change the voltage setting:

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



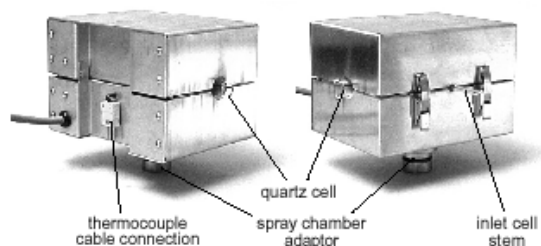
**Figure 10** Changing the voltage selection

- 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.

## Installing an Absorption Cell

### To install an absorption cell:

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



**Figure 11** The ETC 60 workhead

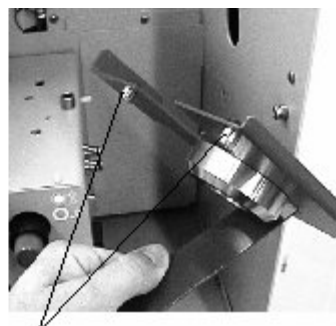
## Installing the Workhead

### To install an absorption cell:

- 1 Place the Temperature Control Module on top of the spectrometer.
- 2 Remove the upper and lower flame shields from the spectrometer.
- 3 If it is fitted, remove the burner from the spraychamber.
- 4 Remove the spraychamber and store it in the stand provided.
- 5 Align the lamps in your instrument.
- 6 Fit the two lugs on the workhead bracket into the two holes on top of the burner adjuster.

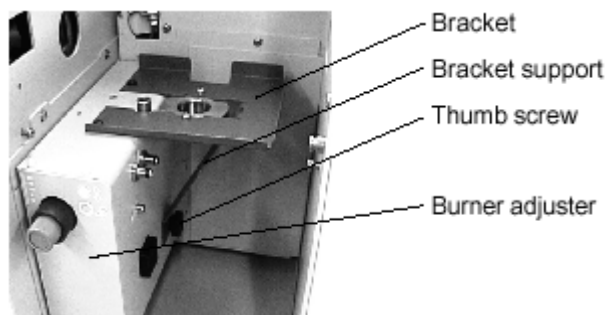


Holes on burner adjuster



Lugs on workhead bracket

**Figure 12** Preparing to fit the workhead bracket



**Figure 13** Workhead bracket fitted to the burner adjuster

- 7 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.**

- 8 Fit the ETC 60 workhead into the hole in the bracket and push down firmly to seat this in the bracket.

- 9 Connect the temperature sensor cable to the workhead.

**NOTE**

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

---

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

**NOTE**

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

---

- 11 Plug the cell heater and temperature sensor cables into the appropriate sockets in the rear of the Temperature Control Module.
  - 12 Optimize the workhead position for maximum light transmission by using the burner adjustment controls.
  - 13 Check the rotational alignment of the workhead by rotating it to maximize the signal.
  - 14 Carefully attach the black fluoroelastomer tubing to the inlet cell stem taking care not to shift the workhead position. If necessary, reoptimize the workhead position using the burner adjuster controls.
- 

**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.**

---

- 15 Mount the VGA on to the instrument as outlined in the manual that ships with the accessory.
- 16 Carefully connect the black fluoroelastomer tubing to the outlet of the VGA gas-liquid separator.

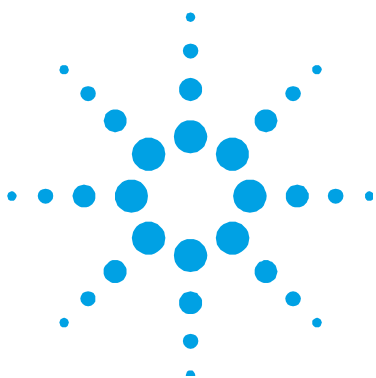
## Getting Started

- 17 Connect the mains power cable to the Temperature Control Module.

The system is now ready for use.

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 SpectrAA software, version 5 or later.





### 3. Operation

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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.

## Operation

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.

---

### 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
```

### 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 ***
```

## Operation

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.

### TIP

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.

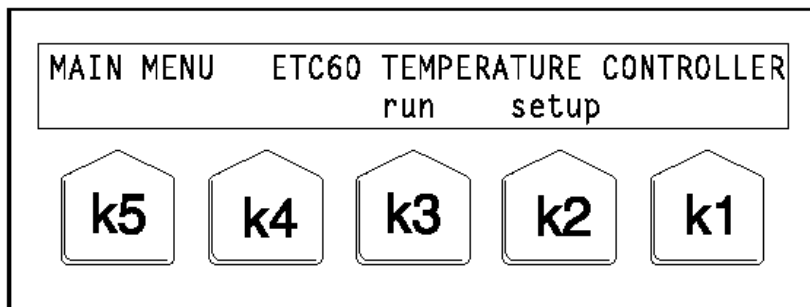


Figure 14 Main menu

## 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

#### 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 `>>>`.

## Operation

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 31.

### 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.

### TIP

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.

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.

## Operation

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:**

- 1 Turn off the ETC 60.
  - 2 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.
  - 3 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.
  - 4 Turn off the inert gas.
- 

### **WARNING**



#### **Hot Surface – Electrical Shock**

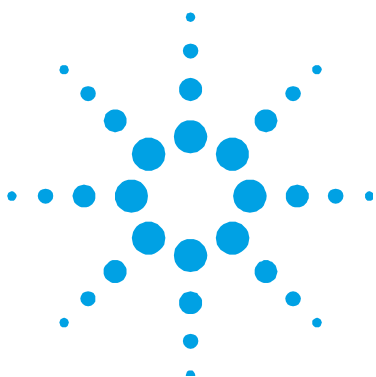
The workhead will be very hot, and contact with it could cause severe burns. Always ensure that the workhead is allowed to cool before attempting to remove it from the sample compartment.



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.

---

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## 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 lists the suggested optimum temperatures for production of atomic vapor from the hydride.

**Table 2** Optimum temperatures for specific elements

Element, Symbol	Temperature (°C)
Arsenic, As	950 (refer to Page 36)
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**

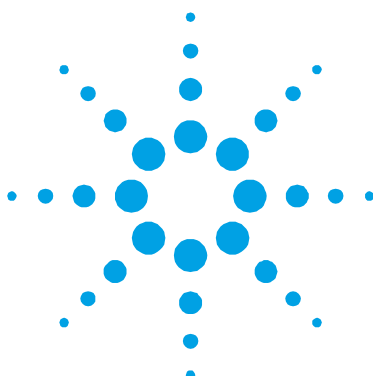
Do not exceed 300 °C when optimizing for mercury.

---

- 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.

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## 5. Maintenance

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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 20.
- 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 in one of two ways:

- 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.
- Instead of using a Y-connector, fit a desiccant tube to capture and retain any spray. Pack the tube so as not to create excessive back pressure. Contact your Agilent field service engineer for advice on how to do this.

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.
  - 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.

## Maintenance

- 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**

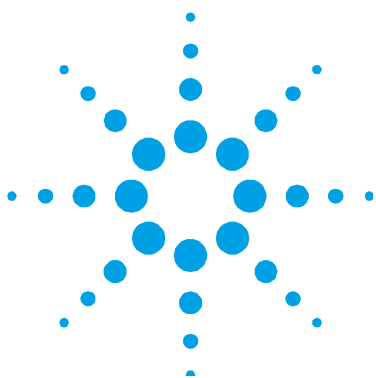
**The workhead will be very hot, and contact with it could cause severe burns. Always ensure that the workhead is allowed to cool before attempting to remove it from the sample compartment.**



**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.**

---

Removal of the workhead is the reverse of the installation procedure outlined in ‘Installing the Workhead’ section, Page 21.



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Appendix C — Sensitivity Test Record Sheet (cont.)	47

### 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

For optimum analytical performance, it is recommended that the ambient temperature of the laboratory is between 20–25 °C and held constant to within  $\pm 2$  °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 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:

Date	Absorbance	Measured Concentration
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### Appendix C — Sensitivity Test Record Sheet (cont.)

Solution (concentration):

Matrix:

Date	Absorbance	Measured Concentration
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## **In This Book**

The manual describes the following:

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

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