

## Agilent Atom Concentrator Tube ACT 80

## **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.

#### **Contents**

## Contents

1.	Safety Practices and Hazards	5
	General	5
	Hazard Warnings	5
	Notes and Tips	6
	Warning and Caution Messages	6
	Warning Symbols	7
2.	Introduction	9
3.	Installation	11
	Installation on the Mark 7 Burner	12
	Fitting an ACT 80 Tube to the Holder	15
	Installation on the Mark VI Burner	16
	Fitting the Brackets to the Burner	16
	Fitting an ACT 80 Tube to the Holder	17
	Fitting the Tube Holder to the Burner	18
	Installation on the Mark V Burner	19
	Fitting the Adaptor to the Burner	19
	Fitting an ACT 80 Tube to the Holder	20
	Fitting the Tube Holder to the Burner	21
4.	Alignment	23
	Aligning the ACT 80 Tube on the Burner	23
	Optical Alignment of the ACT 80 Tube	24

#### Contents

5.	Operation	25
	Operating Principle	25
	Igniting the Flame	26
	Reigniting the Flame	27
	Practical Notes	28
	Formation of Acetylides	28
	Metal Deposits	29
6.	Maintenance and Spare Parts	31
	Cleaning the ACT 80 Tube	31
	Tube Life	31
	Spare Parts	32



## **1. Safety Practices and Hazards**

General	5
Hazard Warnings	5
Notes and Tips	6
Warning and Caution Messages	6
Warning Symbols	7

## General

This section should be read in conjunction with the Safety Practices and Hazards section in your AA spectrometer manual.

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.

#### **Safety Practices and Hazards**

## **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.



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. 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:





Eye hazard



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.



**Safety Practices and Hazards** 

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There are analytical situations in flame atomic absorption where the elemental concentration gives absorbance values that are too low to enable determination using the flame AA technique, and too high to enable determination using the graphite furnace AA technique. Dilution can of course be used, but this requires additional sample preparation and introduces a potential source of error or contamination.

The Atom Concentrator Tube (ACT) provides a potential solution for these types of applications. When installed, the Agilent ACT 80 atom concentrator tube enables the sensitivity of elements determined in an air-acetylene flame to be increased by a factor of 2–3. A slight improvement in the detection limit usually occurs as well.

The ACT 80 is suitable for use with the air-acetylene burner and is compatible with any AA instrument in which the tube can be mounted on the burner and positioned in the optical path. The tube may be placed on the air-acetylene burner in a few seconds and quickly aligned in the optical path. Measurement with the ACT 80 is as fast as normal flame determinations, and can be used with manual or automated sampling. The kit comprised by the ACT 80 consists of this manual, a sample quantity of quartz tubes, and a tube holder to enable installation and use with the air-acetylene burner. Additionally, assemblies ordered for use with the Mark V burner include a pair of mounting brackets and those for the Mark VI burner include an adaptor that allows the tube holder to be mounted over the air-acetylene flame.

#### NOTE

For the Mark 7 burner, the tube holder fits directly over the burner. For the Mark VI burner, the two brackets are secured to the burner and used to support the tube holder in position. For the Mark V burner, the adaptor consists of an assembly which is clamped to the burner and the tube holder is then mounted on top.



## 3. Installation

Installation on the Mark 7 Burner	12
Installation on the Mark VI Burner	16
Installation on the Mark V Burner	19

The atom concentrator tube assembly can be easily fitted to an airacetylene burner. The atom concentrator tube itself is mounted in a tube holder fitted directly over the burner.

NOTE On some model instruments fitted with the Mark V or Mark VI burners, an additional mounting bracket/adaptor is required to be installed on the burner to locate the tube holder on top of the burner

#### CAUTION

Do **not** fit the tube holder on to a nitrous oxide-acetylene burner. The ACT 80 tube will be permanently damaged if exposed to a nitrous oxide-acetylene flame.

## Installation on the Mark 7 Burner

The ACT 80 for the Mark 7 burner is supplied with a compatible tube holder which fits directly to the burner. A tube holder for a Mark 7 burner does not require any additional mounting brackets.



#### **Hot Surface**

If the burner has been operating, the burner and its surrounds will be very hot. Allow the burner to cool, and wear protective gloves when installing equipment into the burner compartment. Otherwise, a serious burn may result.



Figure 1 Mark 7 burner without the tube holder installed

#### To mount the tube holder on a Mark 7 burner:

**1** Position the seat cut-outs of the tube holder over the back edge of the burner.





**2** Pull the tube holder forward so that the cut-outs hook around the burner edge.





#### Installation

**3** Lower the tube holder into position.



Figure 4 Lowering the tube holder into position on the Mark 7 burner



Figure 5 Mark 7 burner with the tube holder correctly installed

#### Fitting an ACT 80 Tube to the Holder

NOTE Before its first use, an ACT 80 tube should be washed in a warm, dilute solution of laboratory detergent (such as Decon 90 or Triton X-100), rinsed well with distilled water and dried.

## CAUTION To prevent contamination buildup, avoid handling the slotted section of the ACT 80 tube. Handle the tube by its ends, or wear gloves.

#### To fit an ACT 80 tube into the holder:

- 1 Lift the left spring and pass the left side of the ACT 80 tube through the cut out in the tube holder.
- **2** Gently release the left spring.
- **3** Lift the right spring, gently push the ACT 80 tube fully home and gently release the right spring.
- **4** Ensure that the ACT 80 tube is installed in the holder so that the second shorter slot in the tube is facing towards the rear of the sample compartment.

## **NOTE** If this second slot is facing towards the operator, the ACT 80 tube must be removed from its mounting and reversed.

### Installation on the Mark VI Burner

The ACT 80 for the Mark VI burner is supplied with a compatible tube holder and two brackets that mount directly to the burner.

#### Fitting the Brackets to the Burner



#### Hot Surface

If the burner has been operating, the burner and its surrounds will be very hot. Allow the burner to cool, and wear protective gloves when installing equipment into the burner compartment. Otherwise, a serious burn may result.

#### To install the mounting brackets onto a Mark VI burner:

- 1 Position a mounting bracket at one end of the burner, with the mounting peg pointing inward and the hole in the end of the bracket aligned over the hole in the end of the burner.
- 2 Use one of the screws provided to fix the bracket into position.
- **3** Repeat the above for the other mounting bracket.



Figure 6 Mark VI burner with the mounting brackets installed

Once the brackets are attached to the burner, they can be left permanently installed since they will not interfere with normal flame analyses.

**NOTE** The Mark VI A burner looks slightly different to the Mark VI burner; however the procedure for installing the brackets is the same.

#### Fitting an ACT 80 Tube to the Holder

**NOTE** Before its first use, an ACT 80 tube should be washed in a warm, dilute solution of laboratory detergent (such as Decon 90 or Triton X-100), rinsed well with distilled water and dried.

## **CAUTION** To prevent contamination buildup, avoid handling the slotted section of the ACT 80 tube. Handle the tube by its ends, or wear gloves.

#### To fit an ACT 80 tube into the holder:

- **1** Lift the left spring clip.
- **2** Pass the tube through the left notch in the tube holder until the other end of the tube rests against the right notch of the holder.
- **3** Gently release the left spring clip.
- **4** Open the right spring clip. Gently push the tube until it is fully home in the right notch. Ensure that the tube is positioned in the center of the holder.
- 5 Gently release the right spring clip. The tube should be held firmly in the notches provided in the tube holder, with the tube being secured in position by the two spring clips.
- **6** Hold the tube at one end and rotate the tube until the long slot is at the bottom, and the shorter slot faces away from you.

With an ACT 80 tube fitted, the tube holder is now ready to be placed onto the burner.

#### Fitting the Tube Holder to the Burner

Hot Surface

## WARNING



If the burner has been operating, the burner and its surrounds will be very hot. Allow the burner to cool, and wear protective gloves when installing equipment into the burner compartment. Otherwise, a serious burn may result.

#### To fit the tube holder onto a Mark VI burner:

- 1 Holding the handle, position the tube holder so that the two slots at the bottom of the tube holder are over the pegs on the mounting brackets.
- **2** Gently lower the holder onto the pegs and rest it on top of the burner (refer to Figure 7).



Figure 7 Mark VI burner with the tube holder and ACT 80 tube installed

## Installation on the Mark V Burner

The ACT 80 for the Mark V burner is supplied with a compatible tube holder and an adaptor that fits directly on to the burner.

Fitting the Adaptor to the Burner



#### Hot Surface

If the burner has been operating, the burner and its surrounds will be very hot. Allow the burner to cool, and wear protective gloves when installing equipment into the burner compartment. Otherwise, a serious burn may result.

#### To install the adaptor onto a Mark V burner:

- 1 Loosen the two clamping screws on the underside of the adaptor.
- 2 Orient the clamp so that the two mounting pegs are at the front of the burner. This will ensure that the burner identification label is still visible through the 'open' portion of the clamp.

NOTE

The front of an air-acetylene burner carries the identification label.

- **3** Fit the adaptor over the burner, so that the two positioning lugs at each end of the clamp rest on top of the burner and lie on either side of the burner slot.
- **4** Secure the adaptor into position by retightening the clamping screws.

Once the adaptor is attached to the burner, it can be left permanently installed since they will not interfere with normal flame analyses.

Installation



Figure 8 Mark V burner with the adaptor installed

#### Fitting an ACT 80 Tube to the Holder

NOTE Before its first use, an ACT 80 tube should be washed in a warm, dilute solution of laboratory detergent (such as Decon 90 or Triton X-100), rinsed well with distilled water and dried.

CAUTION

To prevent contamination buildup, avoid handling the slotted section of the ACT 80 tube. Handle the tube by its ends, or wear gloves.

#### To fit an ACT 80 tube into the holder:

- **1** Lift the left spring clip.
- **2** Pass the tube through the left notch in the tube holder until the other end of the tube rests against the right notch of the holder.
- **3** Gently release the left spring clip.
- **4** Open the right spring clip. Gently push the tube until it is fully home in the right notch. Ensure that the tube is positioned in the center of the holder.

- 5 Gently release the right spring clip. The tube should be held firmly in the notches provided in the tube holder, with the tube being secured in position by the two spring clips.
- **6** Hold the tube at one end and rotate the tube until the long slot is at the bottom, and the shorter slot faces away from you.

With an ACT 80 tube fitted, the tube holder is now ready to be placed onto the burner.

#### Fitting the Tube Holder to the Burner



#### Hot Surface

If the burner has been operating, the burner and its surrounds will be very hot. Allow the burner to cool, and wear protective gloves when installing equipment into the burner compartment. Otherwise, a serious burn may result.

#### To fit the tube holder onto a Mark V burner:

- 1 Holding the handle, position the tube holder so that the two slots at the bottom of the tube holder are over the mounting pegs on the burner adaptor.
- **2** Gently lower the holder onto the pegs and rest it on top of the burner (refer to Figure 9).



Figure 9 Mark V burner with the tube holder and ACT 80 tube installed

**Agilent Atom Concentrator Tube ACT 80 User's Guide** 

Installation

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23

24



The alignment of an ACT 80 tube requires two separate procedures. First the ACT 80 tube must be visually aligned correctly over the burner slot. Secondly, it must be aligned optically.

## Aligning the ACT 80 Tube on the Burner

Visually align the quartz tube so that the longest slot is directly over the burner slot, refer to Figure 10. You may have to rotate the tube and/or move the tube sideways in the support.

Ensure that the ACT 80 tube is installed in the holder so that the second shorter slot in the tube is facing towards the rear of the sample compartment, refer to Figure 10. If this second slot is facing towards the operator, the ACT 80 tube must be removed from its mounting and reversed.



Figure 10 Diagram of an ACT 80 tube correctly aligned on the burner

## **Optical Alignment of the ACT 80 Tube**

#### To align the ACT 80 tube with the optical path:

- 1 Lower the burner using the burner height controls so that it is clear of the optical path.
- 2 Fit the required hollow cathode lamp and set up the spectrometer as described in your instrument operation manual.
- **3** Select **Optimize**, then **Optimize Lamp**.
- **4** Align the position of the ACT 80 tube with the optical path as follows:
  - **a** Hold a piece of white card between the end of the ACT 80 tube that is furthest from the lamp and the sample compartment window.
  - **b** Using the burner positioning controls, adjust the position of the ACT 80 tube until the light from the hollow cathode lamp passes through the tube and onto the card. Remove the card.
  - **c** Use the burner positioning controls to adjust the position of the cell for maximum transmission.

Once the ACT 80 tube has been aligned in the optical path, it can be used for determinations with any method that uses an air-acetylene flame.



Operating Principle	25
Igniting the Flame	26
Practical Notes	28

## **Operating Principle**

The ACT 80 tube is constructed from quartz and has two longitudinal slots cut into it, which are positioned at an angle of  $120^{\circ}$  with respect to each other relative to the axis of the tube (refer to Figure 10, on Page 23). The tube is mounted above the air-acetylene burner such that the longer slot (10 cm) is positioned immediately above the flame. The shorter slot faces to the rear of the instrument. The flame enters the tube through the long slot and exits through the short slot and at both ends of the tube.

When a solution is aspirated, the atoms pass into the tube. The quartz tube wall prevents the atoms from diffusing out of the optical path, thereby increasing the residence time of the atoms in the optical path. This provides an enhancement in sensitivity. The tube wall also stops air diffusing into the flame, which prevents the formation of oxides that would otherwise result in poorer sensitivity.

#### NOTE Background correction may be required for some samples.

#### Operation

### **Igniting the Flame**

With an ACT 80 tube installed, the burner will be positioned lower than usual, so a standard mixture or a fuel-lean mixture may not ignite readily.

## WARNING



To avoid an explosive accumulation of fuel and oxidant in the sample compartment, ALWAYS ensure that the gas mixture is fuel-rich before operating the Ignite button.

Before igniting the flame, carry out any safety checks described in your spectrometer operation manual. Follow all instructions and observe all warnings.

#### To ensure flame ignition with an ACT 80 tube in position:

1 Set the acetylene flow higher than normal in order to obtain a fuel-rich air-acetylene flame.

# **NOTE** This is required for igniting the flame with an ACT 80 tube in position. Some instrument models may automatically set a fuel-rich mixture. You should refer to your spectrometer operation manual for details.

- 2 If a Mark V or Mark VI burner is fitted, tilt tube cell holder up to clear the ACT 80 tube from it position above the burner. On older instrument models this ensures that the igniter arm does not strike the ACT 80 tube during ignition.
- **3** Press the **Ignite** button to ignite the flame and then adjust the fuel flow to give a lean flame.

**CAUTION** If you leave the flame burning with a fuel-rich mix, carbon particles may become deposited on the outside wall of the ACT 80 tube. Under these conditions, the tube can be permanently damaged by localized overheating.

- **4** If necessary, lower the cell holder back into position directly above the burner.
- 5 Allow the ACT 80 tube approximately 30 s to equilibrate.

The thermal shock of turning off the flame, or repositioning a tube in the flame is the cause of most ACT 80 tube failures. Longer tube lifetimes may be obtained by reducing the number of times the flame is turned off to the absolute minimum. Leave the flame running at **all** times whilst the instrument is being attended. Aspirate distilled water during idle periods and only turn the flame off at the end of the working day or at the end of an analysis.

#### **Reigniting the Flame**



TIP

**Hot Surface** 

If the burner has been operating, the burner and its surrounds will be very hot. To avoid serious burns, wear suitable heat-resistant gloves when manipulating a tube holder that may still be too hot to handle with unprotected hands.

If the flame goes out and needs reigniting, refer to the 'Igniting the Flame' section on Page 26.

## **Practical Notes**

#### **Formation of Acetylides**

Explosion Hazard – Eye Hazard





Analysis of solutions containing high concentrations ( $\geq 1\% \text{ w/v}$ ) of copper or silver using the ACT 80 may lead to the formation of explosive acetylide compounds. These unstable organometallic compounds may spontaneously detonate and could cause an explosion, a major flashback of the air-acetylene flame, or a minor explosion within the quartz tube itself. Agilent recommends that you do not use the ACT 80 when performing analysis of solutions that contain high concentrations ( $\geq 1\% \text{ w/v}$ ) of copper, silver, or both. Always ensure that all safety covers, doors, and the chimney are in position on the instrument.

If you have no choice but to analyze solutions containing high concentrations of copper or silver with the ACT 80, it may be possible to reduce the risk of explosion or flashback by taking the following measures:

- Reduce the concentration of metal in all analytical solutions to the lowest practicable level. A diluted solution will extend the safe operating time, allowing a longer aspiration time, before any explosion or flashback occurs.
- Aspirate all solutions for the shortest practicable period.
- Always aspirate distilled water between samples.
- Clean or replace the ACT-80 tube frequently.

#### **Metal Deposits**

Deposits of metal inside an ACT 80 tube form a bright reflective surface on the inside of the (normally transparent) tube.



#### Fire Hazard – Explosion Hazard

Heavy deposits of metal inside an ACT 80 tube may increase the heat reflected back from the flame. This may cause melting of the burner base or softening of the plastic spray chamber around the burner port that can lead to damage the seal between the O-ring on the burner and the spraychamber. Any such gas leak poses a fire or explosion hazard. To reduce the possibility of the spraychamber being damaged by an overheated burner, always keep the ACT 80 tube free of heavy deposits of metal.

## NOTE

For most low concentration aqueous solutions, the ACT 80 may be operated for extended periods (at least eight hours) with the flame on continuously, without softening or melting the spraychamber.

**Operation** 

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## **Cleaning the ACT 80 Tube**

Clean an ACT 80 tube by washing it regularly in a warm, dilute solution of laboratory detergent. Rinse the tube well with distilled water and allow it to dry thoroughly before reusing it.

### **Tube Life**

The working life of the ACT 80 tube will depend on the type of analytical programs performed. At some stage, the quartz will start to turn white indicating the onset of devitrification. Initially, devitrification will not degrade the analytical performance, but eventually the devitrified area will extend over most of the tube at which point it may be necessary to discard the tube. If devitrification is excessive, the tube will eventually crack spontaneously or fail through thermal shock.

In some applications, where quantities of alkali elements are present in the samples, the devitrification process is accelerated. The alkali atoms migrate into the quartz tube and form silicates. These silicates then crystallize when the tube is cooled, causing devitrification.

For these types of samples, the devitrification process has been slowed by applying a coating of lanthanum to the ACT 80 tube before testing any samples. This can be done by aspirating a 1% w/v solution of lanthanum chloride for 10-15 minutes. The coating is thought to inhibit migration of the alkali elements.

Whether a tube becomes devitrified or not, and even if it is kept meticulously clean, the inside surface of the tube may still deteriorate depending on the matrix of the samples used. You should regularly monitor the performance of the tube using absorbance measurements obtained from standard solutions. Discard the tube as soon as the results of this monitoring become unacceptable.

Finally, under some circumstances, the center section of the tube may gradually soften in the flame and sag. In this case, you must discard the tube immediately and replace it with a new one.

### **Spare Parts**

The following spare parts are available from Agilent for the ACT 80:

Table 1	Spare quartz tubes
---------	--------------------

1100	crintion	
003	onpuon	

Replacement ACT 80 tubes, quartz

For the most up-to-date information on spare parts and consumables ordering information, refer to the Agilent Technologies website:

www.agilent.com

#### www.agilent.com

#### In This Book

The manual describes the following:

- Safety Practices and Hazards
- Introduction
- Installation
- Alignment
- Operation
- Maintenance and Spare Parts

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