

# Agilent 7650A **Automatic Liquid** Sampler

# Installation, Operation, and **Maintenance**



# Notices

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## 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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# Part 1:

# **Quick Start**

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Agilent 7650A Automatic Liquid Sampler Installation, Operation, and Maintenance

# **Quick Start**

About Your Agilent 7650A ALS System 12 System Overview 12 Features 12 Where to Find Information 13 Agilent GC and GC/MS Hardware User Information & Utilities DVDs 13 Online Help 14

This chapter provides an overview of the Agilent 7650A Automatic Liquid Sampler (ALS) and the user information available to you.



# About Your Agilent 7650A ALS System

## System Overview



Figure 1 The 7650A ALS

#### **Features**

The key features of the 7650A ALS include:

- A sample turret to accommodate up to 50 sample vials, four solvent bottles, and four waste bottles
- 8 mL of accessible solvent, 16 mL of total solvent
- 16 mL of waste vial capacity
- A standard syringe carriage for syringes up to 100 µL
- An optional enhanced sample handling syringe carriage, optimized with a high-powered motor for syringes over 100  $\mu L$
- Ability to run sandwich injections with up to three sample layers separated with air gaps
- Fast, slow, and variable plunger speeds under Agilent data system control (see "Fast, Slow, and Variable Plunger Speeds" on page 58)

# Where to Find Information

This manual provides safety and regulatory information, installation procedures, operation information, and a maintenance and troubleshooting section. This manual is available on the Agilent GC and GC/MS Hardware User Information & Utilities DVDs as an installed HTML-based manual and as a printable PDF.

## Agilent GC and GC/MS Hardware User Information & Utilities DVDs



In addition to this manual, Agilent provides several learning products that document how to install, operate, maintain, and troubleshoot Agilent's gas chromatograph (GC), gas chromatograph/mass spectrometer (GC/MS), and low thermal mass-gas chromatograph/mass spectrometer (LTM-GC/MS) product lines. Included on the DVDs are localized versions of the information you need most, such as:

- Getting Started documentation
- Site Preparation information
- Safety and Regulatory information
- Installation, Operation, Maintenance, and Troubleshooting information

There are two formats of documentation available on the Agilent GC and GC/MS Hardware User Information & Utilities DVDs.

#### **Comprehensive HTML-based documentation**

The comprehensive HTML-based documentation format provides full-text search capabilities across the entire suite of documentation for an instrument. This documentation is integrated seamlessly with the accompanying Agilent Instrument Utilities software, and can also be accessed as a stand-alone documentation set from the Microsoft<sup>®</sup> Windows<sup>®</sup> **Start** menu.

#### **Print-optimized and localized PDFs**

The print-optimized and localized PDF format provides easy printing and text searching within a single document at a time.

### **Online Help**

Agilent data systems are designed to maximize the use of your Agilent 7650A ALS and accompanying instruments. Use the data system's online help to access user interface descriptions, key concepts, common tasks, and tutorials for the software.

The online help topics are written as detailed step-by-step instructions with links to other relevant material. Reading these topics will help you complete your daily activities (Figure 2).

😵 78xx Series GC Help	
Hide Back Forward Print	
Hide Back Forward Print Contents Search Hide Back Forward Print Contents Search What's New? Loading Methods Method Editor	Automatic Liquid Sampler (ALS) Use the Front Injector and Back Injector panels to enter ALS (injector) parameters for the method. Injection • Syringe Size — This field displays the <u>configured</u> size of the installed syringe. • Injection Volume — Select the injection volume (in uL). The amounts listed depend on configured syringe size. • Number of Injections per Run — This parameter is available if Multiple Injections is selected from the Injection Type field. The entry field next to the Injection Volume field is used to enter the number of injections per run when performing large volume injections with multiple injections. • To perform a single injection, enter 1. • To perform multiple injections per run, enter the number of injections
Readiness     Instrument Configuration     Sample Prep     Tasks     Tasks     Tutorial	The total injection volume is displayed. When configured for multiple injections, the injector issues a Start Run command at the first injection only.
	Multiple Injection Delay — This parameter is available if Multiple Injections

Figure 2 Data system online help

When using the Agilent data system, press [F1] to access content-sensitive online help, or select Help from the Help menu.



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# • Part 2:

# **Safety and Regulatory Information**

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Installation, Operation, and Maintenance Part 2: Safety and Regulatory Information



Agilent 7650A Automatic Liquid Sampler Installation, Operation, and Maintenance

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This chapter provides important safety and regulatory information about the Agilent 7650A Automatic Liquid Sampler (ALS).



# Important Safety Warnings

There are several important safety notices that you should always keep in mind when using the 7650A ALS.

#### Many internal parts of the instrument carry dangerous voltages

With the instrument power switch on, potentially dangerous voltages can exist on:

- All electronics boards in the instrument
- The internal wires and cables connected to these boards

#### WARNING

All these parts are shielded by covers. With the covers in place, it should be difficult to accidentally make contact with dangerous voltages. Unless specifically instructed to, never remove a cover.

### WARNING

If the power cord or wiring from the instrument to the gas chromatograph insulation is frayed or worn, the cord must be replaced. Contact your Agilent service representative.

#### Electrostatic discharge is a threat to instrument electronics

The printed circuit (PC) boards in the instrument can be damaged by electrostatic discharge. Do not touch any of the boards unless it is absolutely necessary. If you must handle them, wear a grounded wrist strap and take other antistatic precautions. Wear a grounded wrist strap any time you must remove the electronics cover.

# Safety and Regulatory Certifications

The 7650A ALS conforms to the following safety standards:

- International Electrotechnical Commission (IEC): 61010-1
- EuroNorm (EN): 61010–1

The instrument conforms to the following regulations on Electromagnetic Compatibility (EMC) and Radio Frequency Interference (RFI):

- CISPR 11/EN 55011: Group 1, Class A
- IEC/EN 61326-1
- AUS/NZ C N10149

This ISM device complies with Canadian ICES-001. Cet appareil ISM est conforme a la norme NMB-001 du Canada.

# CE

The instrument is designed and manufactured under a quality system registered to ISO 9001.

### Information

The Agilent Technologies ALS meets the following IEC (International Electrotechnical Commission) classifications: Safety Class I, Transient Overvoltage Category II, Pollution Degree 2.

This unit has been designed and tested in accordance with recognized safety standards and is designed for use indoors. If the instrument is used in a manner not specified by the manufacturer, the protection provided by the instrument may be impaired. Whenever the safety protection of the ALS has been compromised, disconnect the unit from all power sources and secure the unit against unintended operation.

Refer servicing to qualified service personnel. Substituting parts or performing any unauthorized modification to the instrument may result in a safety hazard.

### **Symbols**

Warnings in the manual or on the instrument must be observed during all phases of operation, service, and repair of this instrument. Failure to comply with these precautions violates safety standards of design and the intended use of the instrument. Agilent Technologies assumes no liability for the customer's failure to comply with these requirements.

See accompanying instructions for more information.

Indicates a hot surface.

Indicates hazardous voltages.

Indicates earth (ground) terminal.

Indicates explosion hazard.

Indicates electrostatic discharge hazard.

### **Technical and environmental specifications**

- Indoor use only in ordinary atmospheres
- Altitude up to 4300 m
- Operating ambient temperatures between 15 degrees centigrade and 55 degrees centigrade
- Ambient operating humidity of 5 % to 95 %
- Pollution degree 2, Installation Cat II



### **Electromagnetic compatibility**

This device complies with the requirements of CISPR 11 and IEC 61326-1. Operation is subject to the following two conditions:

- 1 This device may not cause harmful radio frequency interference.
- **2** This device must accept any radio frequency 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.
- 7 Changes or modifications not expressly approved by Agilent Technologies could void the user's authority to operate the equipment.

### Sound Emission Certification for Federal Republic of Germany

#### Sound pressure

Sound pressure Lp < 82 dB(A) according to DIN-EN 27779 (Type test).

#### Schalldruckpegel

Schalldruckpegel LP < 82 dB(A) nach DIN-EN 27779 (Typprufung).

# Cleaning

To clean the external surfaces of the 7650A ALS, disconnect the power and wipe down with a damp, lint-free cloth. Refer to "Periodic Maintenance" on page 118 for more information.

# **Recycling the Product**

For recycling, contact your local Agilent sales office.



Agilent 7650A Automatic Liquid Sampler Installation, Operation, and Maintenance

# • Part 3:

# Installation

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This chapter assists you in determining whether the 7650A ALS is suitable for your Agilent GC, GC/MSD, or LTM-GC/MSD (instrument) and aids in identifying existing equipment.



# Hardware

The 7650A ALS is compatible with the following Agilent instruments:

- 7890A GC (front or back inlet)
- 7820A GC (front or back inlet)
- 5975E GC/MSD (front or back inlet)
- 5975T LTM-GC/MSD (front inlet)

The ALS supports the use of one ALS per instrument. More than one ALS per instrument is not supported.

## Firmware

To use the 7650A ALS, Agilent instruments require the minimum firmware revisions listed in Table 1.

Instrument	Minimum firmware version required
7890A GC	A.01.13
G3430 Controller (for 7890A GC)	A.02.11
7820A GC	A.01.12.003
5975E GC/MSD	5.02.12
5975T LTM-GC/MSD	A.03.03.005

**Table 1**Minimum firmware revisions for 7650A ALS use

To update the instrument firmware, see "Updating the Firmware" on page 49.



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# Installation

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The procedure for installing the 7650A ALS depends on the type of Agilent instrument involved. In all cases, you must remove any existing ALS components before installation. Your Agilent instrument may require a firmware update.



# **Preparing the Instrument**

This procedure explains how to prepare an Agilent GC, GC/MSD, or LTM-GC/MSD for the 7650A ALS.

# **WARNING** The inlet may be hot enough to cause skin burns. Allow the inlet to cool to ambient temperature before working near the inlet.

- 1 Set the instrument's inlets, detectors, and oven to room temperature.
- **2** After the inlets, detectors, and oven have cooled, turn off the instrument.
- **3** If installed, unplug any ALS cables and remove any injectors from the instrument.

For details, refer to the sampler's original documentation.

# **Preparing the ALS**

## **Remove the shipping screws**

- 1 Lay the ALS on a flat surface so the bottom of the ALS is accessible.
- **2** Using a T-10 Torx driver, remove the three red-colored T-10 Torx shipping screws from the bottom of the ALS, as shown in Figure 3.



Figure 3 Remove three shipping screws

## **Remove the shipping clamp**

The orange and blue shipping clamp is installed on the syringe carriage as shown in Figure 4.



Figure 4 Shipping clamp installed

To remove the shipping clamp:

1 Remove the shipping tape (Figure 5) and open the ALS door.



**Figure 5** Remove the shipping tape

**2** Using a T-10 Torx driver, completely loosen the T-10 Torx captive screw and remove the orange shipping clamp from the syringe carriage (Figure 6).



**Figure 6** Remove the orange shipping clamp

**3** Carefully remove the blue shipping clamp from the syringe carriage (Figure 7).



**Figure 7** Remove the blue shipping clamp

**4** Close the ALS door.

#### Installation

## Install the turret

To install the turret:

**1** Open the ALS door (Figure 8).



Figure 8 Open the ALS door

2 Align the tabs on the turret hub with the notch on the inside of the turret, and place the turret onto the turret hub (Figure 9).



Figure 9 Place the turret onto the turret hub

**3** Rotate the turret so that the injector opening faces toward the front of the ALS (Figure 10).



**Figure 10** Rotate the turret so that the injector opening faces toward the front of the ALS

**4** Place the turret hubcap onto the turret hub. Be sure to align the slots on the turret hubcap with the slots on the turret hub as shown in Figure 11.



Figure 11 Place the turret hubcap onto the turret hub

**5** While holding the turret in place, rotate the turret hubcap in a clockwise direction until it snaps into position on the turret hub. Make sure that the tabs on the turret hubcap align with the turret injector opening as shown in Figure 10.



Figure 12 Rotate the turret hubcap in a clockwise direction until it snaps into position
# **Installing the ALS**

## Install the ALS

This procedure explains how to install the 7650A ALS.

### CAUTION

In the following steps, use a flat blade screwdriver that snugly fits the slot in the top of the post. An undersized blade can damage the post top and prevent the ALS from mounting properly.

## CAUTION

Do not mount the ALS on a mounting post for any other injector (unless it is for a 7693A ALS); this can damage the ALS. Remove any incompatible posts and replace with the supplied G4513-20561 mounting post.

**1** Install the ALS mounting post (Figure 13) to the instrument:



TUR

Mounting post (G4513-20561)

• For a 7890A GC, 7820A GC, or 5975E GC/MSD, install the mounting post into the inlet cover in the front or back location (see Figure 14). The post must be turned all the way down.



**Figure 14** Mounting post locations on a 7820A GC (7890A GC is similar)



• For a 5975T LTM-GC/MSD, install the mounting post as shown in Figure 15. The post must be turned all the way down.

Figure 15 Mounting post installed on a 5975T LTM-GC/MSD

#### Installation

2 Connect the ALS communication cable to the front or back ALS port (7820A GC, Figure 16) or the sampler port (5975T LTM-GC/MSD, Figure 17).



Figure 16 Front and back ALS ports on the back of a 7820A GC



Figure 17 Sampler cable port on the back of a 5975T LTM-GC/MSD

**3** Position the ALS on the mounting post and the inlet cover's support guide (see Figure 18 or Figure 19).



Figure 18 ALS installed on a 7890A GC

Installation



Figure 19 ALS installed on a 7820A GC

Installation, Operation, and Maintenance Part 3: Installation

## **Check your work**

The ALS must be vertical and stable.

If the ALS does not sit upright on the instrument, check that the plumbing and cabling under the inlet cover are properly routed in their channels.

## **Connecting the Cables**

This section shows the cable routing for a 7650A ALS with a 7890A GC, a 7820A GC, a 5975E GC/MSD, and a 5975T LTM-GC/MSD.

### 7890A GC

Follow the cabling diagram in Figure 20 to properly wire the ALS to the 7890A GC.



Figure 20 Cables for 7890A GC

Connect the ALS to any available ALS Port on the GC using a  $G4514{\text{-}}60610$  cable.

## 7820A GC

Follow the cabling diagram in Figure 21 to properly wire the ALS to the 7820A GC.



Figure 21 Cables for 7820A GC

Connect the ALS to any available ALS Port on the GC using a  $G4514{\text{-}}60610$  cable.

## 5975E GC/MSD

Follow the cabling diagram in Figure 22 to properly wire the ALS to a 5975E GC/MSD.



Figure 22 Cables for 5975E GC/MSD

Connect the ALS to the GC/MSD with a G4514-60610 cable.

### 5975T LTM-GC/MSD

Follow the cabling diagram in Figure 23 to properly wire the ALS to the 5975T LTM-GC/MSD.



Figure 23 Cables for 5975T LTM-GC/MSD

Connect the ALS to the LTM-GC/MSD with a G4514-60610 cable.

## **Test the connections**

When the communication cables are connected, turn on the instrument power. After the start-up process ends:

- The Ready light on the ALS should be on.
- If the Align Mode light on the ALS is on, see "Aligning the ALS".

## **Updating the Firmware**

Use the Firmware Update utility in Agilent Instrument Utilities software to update the firmware.

The 7650A ALS requires the firmware revisions noted in Table 2. Before using the ALS, check the firmware revisions as described below.

You can download the latest firmware revisions from the Agilent web site at www.agilent.com/chem, or contact your local Agilent sales representative.

Failure to update the firmware will result in unrecognized components or fewer available features.

### View the current firmware version

To view the current firmware version for the instrument or installed ALS components:

**All Instruments** Power cycle the instrument. When it reboots, it displays the current firmware version.

**7890A GC** Press [Status] > [Clear], or press [Service Mode] > Diagnostics > Instrument status to display the current GC firmware version. To view the current ALS component firmware, press [Service Mode] > Diagnostics > ALS Status. Scroll through the menu to view the Front or Back Tower.

**7820A GC and 5975E GC/MSD** Using the software keypad, press [Status] > [Clear], or press [Service Mode] > Diagnostics > Instrument status to display the current instrument firmware version (Figure 24).



Figure 24 7820A GC firmware version displayed on the software remote controller

To view the current ALS component firmware, press [Service Mode] > Diagnostics > ALS Status. Scroll through the menu to view the Front/Back Tower firmware versions (Figure 25).



Figure 25 7650A ALS firmware version displayed on the software remote controller

**5975T LTM-GC/MSD** Press [Menu] to scroll to + Version or + LTM GC, and use the [Item] key to scroll to the desired component firmware information. Use an Agilent data system to view the ALS firmware information.

### Update the firmware

To update the firmware, use the Firmware Update utility (Figure 26) that is part of the Agilent Instrument Utilities software. Refer to the software help and user documentation for firmware update information.

Once you connect to an instrument in the software's **Firmware Update** screen, no other tasks can be performed on the instrument until you disconnect.

🖧 Agilent Instrument Utilities						
Agilent Instrument Utilities	Agilent Instrument Utilities	irrent User:	AGI	LENT		
👫 Lab at a Glance	Version B.1.06 Ci	irrent Instrum	ent:	Agilent 7820		Ψ
Configuration	Firmware Update					
Documentation	Instruments					
Calculators	Model Name	Address	P	rogress S	tatus	*
Calculators	Agilent 7820 Agilent 7820	000.000.0	00.000	-		
Instrument 🔅						
System Information						
Early Maintenance Feedback						_
Status Report						
						*
Help 🄅					Add New	Instrument
Ø About Firmware Update	Current Selection Erit Constants					
Ø Updating PID Constants						
Tasks	Disconnect					
② To download firmware	Component Current Version Available	Update	Restore	Add Firmware File	Progress	
O update GC and 7697A HS firmware	GC7820 MMON V1.01 1 Nov. 2004 A.01.11.002.Ru [7820A.01.11.002.Ru.bin]	▼ Update	Restore	Add		0%
(2) To update G1888 HS firmware	Back Injector: G4567A A.09.24 G4567A.09.24	▼ Update	Restore	Add		0%
To update PID constants						
To up date fit exercise (70204 CC)						
To update the constants (7820A GC)						
🥑 To update 5975T firmware						
O update WorkBench firmware	Show detailed version information					

**Figure 26** Firmware Update utility

## **Configuring the Instrument and Data System**

## **Configure the instrument**

When hardware installation is complete, configure your instrument for use with the 7650A ALS. See "ALS Configuration" for details.

## Configure the data system

### Configuration

Agilent data systems contain information about the sampling equipment in use. This must be updated to remove the old information and replace it with information about the new equipment that you have installed. See your data system documentation for details.

#### **Upgrading methods**

Before using methods created for a previous sampler system, be sure to edit them as needed to match the new hardware.

## **Making a Trial Run**

Once installation, configuration, updating, and calibration is complete, make a quick injection using the sampler to verify that it works properly.

- **1** Install an empty syringe in the ALS.
- **2** Place empty bottles in all of the Solvent and Waste positions, and place an empty capped sample vial in the sample 1 position in the turret. See "Placing Vials and Bottles in the Turret" for more information.
- **3** Make the sampler settings shown in Table 2. These are designed for a 7820A GC. If you are using a different supported instrument, use these settings as a guide.

7890A GCs 7820A GCs 5975T LTM-GC/MSDs Use the front keypad Use the software keypad Use your Agilent data system

Parameter	Setting			
Injection volume	1.00			
Viscosity delay	0			
Inject Dispense Speed	6000			
Airgap Volume	0.20			
Sample Pumps	6			
Sample Washes	0			
Solvent A post washes	1			
Solvent A pre washes	1			
Solvent B post washes	0			
Solvent B pre washes	0			
Sample Draw Speed	300			
Pre dwell time	0			
Post dwell	0			
Sample offset	0			
Injection Mode	Normal (1-layer injection)			
Tower LED	On			

 Table 2
 Trial run parameters

- **4** Set the oven program to 30 °C (or current room temperature) with a 0 °C/min ramp, a hold time of 0.1 minutes, an equilibrium time of 0.3 minutes, and an initial time of 0.3 minutes.
- **5** Store, load, then run the sequence.

If there are no faults, the ALS will make one injection from the first vial position.

If problems occur, see "Faults", "Error Messages", or "Correcting Syringe Problems".



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# Part 4:

# **Operation**

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# Introduction to Operation

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This chapter describes the features and capabilities of the 7650A ALS.



## Fast, Slow, and Variable Plunger Speeds

The default values for variable plunger speeds are based on a 10  $\mu$ L syringe. You should reduce the injection speed to accommodate larger syringe volumes. If you are performing a large volume injection and see a plunger error, try slowing the inject rate.

Plunger Speed (µL/min)	Syringe Volume (µL)									
	0.5	1	2	5	10	25	50	100	250	500
Draw (µL∕min)										
Fast	15	30	60	150	300	750	1500	3000	300	600
Slow	15	30	60	150	300	750	1500	3000	300	600
Dispense (µL∕min)										
Fast	300	600	1200	3000	6000	15000	30000	60000	1500	3000
Slow	300	600	1200	3000	6000	15000	30000	60000	1500	3000
Inject (µL∕min)										
Fast	300	600	1200	3000	6000	15000	30000	60000		
Slow	15	30	60	150	300	750	1500	3000	75	150

 Table 3
 Fast/slow plunger speed as a function of syringe volume

Variable Plunger Speed (µL/min)	Syringe Volume (µL)										
(x) = Default value	0.5	1	2	5	10	25	50	100	250	500	
Sample draw	1 - 30 (15)	1 - 60 (30)	1 - 120 (60)	1 - 300 (150)	1 - 600 (300)	2 - 1500 (750)	3 - 3000 (1500)	6 - 6000 (3000)	15 - 15000 (7500)	30 - 30000 (15000)	
Sample dispense	1 - 300 (300)	1 - 600 (600)	1 - 1200 (1200)	1 - 3000 (3000)	1 - 6000 (6000)	2 - 15000 (15000)	3 - 30000 (30000)	6 - 60000 (60000)	15 - 150000 (150000)	30 - 300000 (300000)	
Injection dispense	1 - 300 (300)	1 - 600 (600)	1 - 1200 (1200)	1 - 3000 (3000)	1 - 6000 (6000)	2 - 15000 (15000)	3 - 30000 (30000)	6 - 60000 (60000)	15 - 150000 (150000)	30 - 300000 (300000)	
Solvent draw	1 - 30 (15)	1 - 60 (30)	1 - 120 (60)	1 - 300 (150)	1 - 600 (300)	2 - 1500 (750)	3 - 3000 (1500)	6 - 6000 (3000)	15 - 15000 (7500)	30 - 30000 (15000)	
Solvent dispense	1 - 300 (300)	1 - 600 (600)	1 - 1200 (1200)	1 - 3000 (3000)	1 - 6000 (6000)	2 - 15000 (15000)	3 - 30000 (30000)	6 - 60000 (60000)	15 - 150000 (150000)	30 - 300000 (300000)	

Table 4Variable plunger speed as a function of syringe volume

# Capabilities

Table 5 summarizes the capabilities of the 7650A ALS.

Parameter	Range
Syringe size	1 to 500 μL
Wash mode	А, В
Solvent saving	10%, 20%, 30%, 40%, 80% of syringe size (µL)
Injection volume	1 - 50% of syringe size (μL)
Sample pumps	0 - 15
Viscosity delay	0 - 7 seconds
Air gap	0 - 10% of syringe size (μL)
Pre-injection sample washes	0 - 15
Post-injection solvent A washes	0 - 15
Post-injection solvent B washes	0 - 15
Plunger speed	Refer to Table 4 on page 59
Pre-injection solvent A washes	0 - 15
Pre-injection solvent B washes	0 - 15
Pre-injection dwell	0 - 1 minutes in .01 minute
Post-injection dwell	0 - 1 minutes in .01 minute
Sampling offset	On, Off
Variable sampling depth position	2 mm below, 30 mm above
Multiple injection mode, number of injections	1 - 99
Multiple injection mode, injection delay time	0 - 100 seconds

Table 57650A ALS capabilities

## **Fast Injection**

This is a method of introducing a sample to a heated inlet without the negative effect of needle fractionation.

If you are using the 7650A ALS for the first time, you may see some changes in the resulting chromatograms. Most of the changes are due to reducing the amount of vaporization from the needle during injection.

- The peak areas of your chromatograms may be smaller. Automatic fast injection delivers the desired setpoint volume of sample. Without fast injection, residual amounts of sample boil out of the needle and enter the inlet. This extra amount could measure up to 1  $\mu$ L.
- The peak areas of your chromatograms may show less differentiation between the low boiling and high boiling components.

Without fast injection, the sample introduced is richer in low boiling component than in high boiling components because of fractional distillation in the needle. Not only does residual sample in the needle enter the inlet, but the low boiling components boil off first. This is needle fractionation or discrimination. Figure 27 compares manual injection with automatic fast injection from the ALS for a 1- $\mu$ L sample of C<sub>10</sub> to C<sub>40</sub> paraffins in hexane.



Figure 27 Automated vs. manual injection

For more information on the performance of the ALS, order the following technical papers from your Agilent representative:

Publication No. 43-5953-1843: Snyder, W. Dale. Fast Injection with the 7673A Automatic Injector: Chemical Performance, Technical Paper 108, June 1985.

Publication No. 43-5953-1878: Snyder, W. Dale. Performance Advantage of the 7673A Automatic Injector Over Manual Injection, Technical Paper 109, August 1985.

Publication No. 43-5953-1879: Kolloff, R. H. C. Toney, and J. Butler. Automated On-Column Injection with Agilent 7673A Automatic Injector and 19245A On-Column Capillary Inlet: Accuracy and Precision, Technical Paper 110, August 1985.

## Sample Carryover

Carryover is the presence of peaks from an earlier injection in the present analysis.

The 7650A ALS uses solvent washes, sample washes, and sample pumps to control carryover. Each of these actions reduces the amount of sample left in the syringe. The effectiveness of each depends on your application.

### Solvent wash

The ALS draws solvent into the syringe from either the solvent A positions or solvent B positions, then discards the syringe contents into one or more waste bottles. Solvent washes can occur before taking a sample (pre-injection solvent wash) or immediately after the injection (post-injection solvent wash). The volume of the wash can be adjusted.

### Sample wash

During a sample wash, the ALS draws the next sample into the syringe and discards the contents into one or more waste bottles. Sample washes occur before the injection. When sample is limited, you can use a solvent prewash to wet the syringe before drawing sample. The volume of the wash can be adjusted.

### Sample pump

During a sample pump, the ALS draws sample into the syringe and returns it to the sample vial. Pumps occur after sample washes and immediately before the injection. Pumps serve to eliminate bubbles. If the needle contains solvent from a previous wash, the pump may add a small amount of solvent that mixes with the sample and can dilute a small volume.

### Number and type of washes

Under ideal conditions, four default volume (80%) washes reduce the carryover to one part in 10,000. The number and type of washes you need depends on:

- The amount of carryover that you can accept
- The viscosity and solubility of the analyte(s)
- The viscosity and volatility of the solvent(s)
- The degree of wear in the syringe barrel
- The wash volume

Chromatograms A and B (Figure 28) show the effect of carryover when 1  $\mu$ L from a vial of methanol is injected after 1  $\mu$ L from a vial of a solute dissolved in methanol. The peaks in chromatogram B are from the solute left in the syringe from the first injection.

Chromatogram C shows the result of washing the syringe with four 80% syringe volume solvent washes. The carryover peaks disappear.



**Figure 28** Sample carryover

## **Methods and Sequences**

The sampler is usually controlled by a sequence, which is a list of samples to be analyzed. It contains:

- Where to find the sample
- What method to use to analyze the sample
- How to measure and inject the sample
- How to generate a report of analysis

A method is a collection of setpoints (temperatures, times, etc.) that controls the operation of a gas chromatograph.

The combination of a sequence and the methods it specifies provides complete control over the analysis of your samples. However, the specific details differ depending on the hardware and software used, so you are referred to other documents for that information.

This discussion is limited to sampler-specific details. For setting up methods and sequences see your Agilent data system help or instrument documentation.

# The Sampler Cycle

Table 6 describes the basic operations of the 7650A ALS.

### Table 6Sampler cycle

Step	Action	Notes			
1 Wash the syringe with solvent.	<ul> <li>a Rotate a solvent bottle under the syringe.</li> <li>b Lower the syringe needle into the solvent.</li> <li>c Draw in solvent.</li> <li>d Raise the syringe needle out of the solvent bottle.</li> <li>e Rotate a waste bottle under the syringe.</li> <li>f Lower the syringe. Depress the plunger to discard solvent.</li> <li>g Raise the syringe needle out of the waste vial.</li> </ul>	<ul> <li>The syringe can be washed several times and with more than one solvent. This is controlled by the solvent pre-wash parameters.</li> </ul>			
2 Wash the syringe with sample.	<ul> <li>a Rotate the sample vial under the syringe.</li> <li>b Lower the syringe needle so that the needle pierces the vial septum and enters the sample.</li> <li>c Draw in sample.</li> <li>d Raise the syringe and needle out of the solvent bottle.</li> <li>e Rotate a waste bottle under the syringe.</li> <li>f Lower the syringe needle. Depress the plunger to discard sample.</li> <li>g Raise the syringe needle out of the waste vial.</li> </ul>	• The syringe can be rinsed with sample several times.			

### **Introduction to Operation**

Step		Action	Notes			
3	Load the syringe with sample.	<ul> <li>a Rotate the sample vial under the syringe.</li> <li>b Lower the syringe needle so that it pierces the vial septum.</li> <li>c Draw in sample.</li> <li>d With the needle still in the sample, depress the syringe plunger quickly.</li> <li>e After the final pump, draw in sample.</li> <li>f Raise the syringe needle out of the sample vial.</li> </ul>	<ul> <li>Actions c and d can be repeated several times. The purpose is to expel air bubbles from the syringe.</li> </ul>			
4	Inject the sample.	<ul> <li>a Rotate the turret to expose the inlet.</li> <li>b Lower the syringe needle so that it pierces the inlet septum.</li> <li>c Depress the syringe plunger to make the injection.</li> <li>d Raise the syringe needle out of the inlet.</li> </ul>	• Start signals are sent to the instrument and the data processor at the moment of injection.			
5	Wash the syringe with solvent.	• Same as step 1, but according to the post-injection parameters.				
6	Perform repeat injections.	<ul> <li>If so programmed, wait for the instrument to become Ready and repeat the cycle from step 1.</li> </ul>				

#### **Table 6**Sampler cycle (continued)

If your method switches between inner and outer row turret positions, the ALS may take more time than it does when moving from vial to vial in the same row.



This chapter describes how to configure the 7650A ALS.



## **Configuring the 7650A ALS**

### 7890A and 7820A GCs

For a 7890A GC, press **[Config] [Front Injector]** or **[Config] [Back Injector]** on the GC keypad to display the front or back injector configuration parameters. For a 7820A GC, press **[Config] [Injector]** on the GC software keypad to display the front or back ALS configuration parameters.

```
CONFIGURE FRONT INJECTOR
Front tower INJ1
Syringe size 10.0
```

<code>Front/Back tower- Toggle between INJ1 (Front Tower) and INJ2 (Back Tower) depending on the tower installation location using the On/Yes and Off/No keys.</code>

Syringe size- Enter a syringe size between 0.5 and 500  $\mu$ L.

#### NOTE

The GC assumes the syringe volume represents full plunger stroke. If the maximum volume marked on the syringe barrel is halfway up the barrel, you will need to enter **double** that volume (the label in this case is half of the full plunger stroke).

#### **5975E GC/MSD**

The 5975E GC/MSD ALS configuration is the same as the 7820A GC. See "7890A and 7820A GCs" on page 70.

## 5975T LTM-GC/MSD

Configure the ALS using your Agilent data system. Refer to your data system help for details.



This chapter describes how to set the 7650A ALS parameters using different controlling devices.

The descriptions in this chapter refer to the features available with the instrument firmware and does not necessarily describe the capabilities available with Agilent data systems. Refer to the Agilent data system help for further information.



# **Setting the 7650A ALS Parameters**

The available parameters depend on the specific configuration of your instrument and ALS. Press the **[Info]** key to view the possible setpoint ranges for each parameter. Refer to Table 4 on page 59 and Table 5 on page 60 for ALS capabilities.
### 7890A and 7820A GCs

For a 7890A GC, press [Front Injector] or [Back Injector] on the GC keypad. For a 7820A GC, press [Injector] on the software keypad. Scroll to the desired setpoint detailed below.

Enter a setpoint value, use [Mode/Type] to change the selection, or turn the setpoint on or off.

FRONT INJECTOR	
Injection volume	1.0
Viscosity delay	0
Inject Dispense Speed	FAST
Sample pumps	6
Sample washes	2
Sample Wash Volume Solvent A post washes	8.0
Solvent A wash volume	8
Solvent B post washes	0
Solvent B pre washes	0
Sample Draw Speed	FAST
Sample Disp Speed	FAST
Solvent Draw Speed	FAST
Solvent Disp Speed	FAST
Pre dwell time Post dwell	0.00
Sample offset	Off
Injection Reps	1

Injection volume-Sample volume to be injected. Enter the injection volume in  $\mu$ L up to 50% of the configured syringe size. (Using a 10  $\mu$ L syringe, entries would be 0.1, 0.2, 0.3, and so forth up to 5  $\mu$ L.) The GC will round the volume to the next allowable parameter.

Viscosity delay—How many seconds the plunger pauses at the top of the pump and injection strokes to wait for the syringe to fill. For viscous samples, the pause allows the sample to flow into the vacuum created in the syringe.

Inject Dispense Speed-How many microliters per minute to inject. Parameter value entered here rounds to the nearest acceptable parameter value. For example, 7000  $\mu$ L/min rounds to 7009  $\mu$ L/min.

Airgap Volume—The amount of air separating the sample from the end of the needle.

Sample pumps—How many times the syringe plunger is moved up and down with the needle in the sample to expel air bubbles and improve reproducibility.

Sample washes—How many times the syringe is rinsed with sample before the injection. The ALS lowers the syringe needle into the sample vial, draws up sample, and empties it into one of the waste bottles.

Sample Wash Volume-How many microliters used for the sample wash.

Solvent A post washes-How many times the syringe is rinsed with solvent from the solvent A bottles.

Solvent A pre washes—How many times the syringe is rinsed with solvent from the solvent A bottles.

Solvent A wash volume-How many microliters used for the sample A wash.

Solvent B post washes-How many times the syringe is rinsed with solvent from the solvent B bottles.

Solvent B pre washes—How many times the syringe is rinsed with solvent from the solvent B bottles.

Solvent B wash volume-How many microliters used for the sample B wash.

Sample Draw Speed—The speed of the syringe plunger during sample injection.

Sample Disp Speed-If using a variable plunger speed, the speed of the sample injection.

Solvent Draw Speed-If using a variable plunger speed, the speed of the syringe plunger during solvent injection.

Solvent Disp Speed-If using a variable plunger speed, the speed of the solvent injection.

Pre dwell time-How long, in minutes, the needle remains in the inlet before the injection.

Post dwell-How long, in minutes, the needle remains in the inlet after the injection.

Sample offset-Enables variable sampling depth.

Injection mode-The type of injection mode.

Injection Reps-If Injection mode is LVI (large volume injection) with multiple reps, the amount of repetitions.

Injection Delay-If Injection mode is LVI (large volume injection) with multiple reps, the length of delay between repetitions.

L2 volume—If Injection mode is a 2-layer or 3-layer Sandwich Injection, the amount of sample used in layer 2.

L2 Airgap volume—If Injection mode is a 2-layer or 3-layer Sandwich Injection, the amount of air used between sample layer 1 and sample layer 2.

L3 volume—If Injection mode is a 3-layer Sandwich Injection, the amount of sample used in layer 3.

L3 Airgap volume—If Injection mode is a 3-layer Sandwich Injection, the amount of air used between sample layer 2 and sample layer 3.

Tower LED-Control the LED light inside the injection tower.

#### **5975E GC/MSD**

The 5975E GC/MSD ALS parameter settings are the same as the 7820A GC. See "7890A and 7820A GCs" on page 73.

#### 5975T LTM-GC/MSD

Set all parameters from your Agilent data system. Refer to your data system help for details.

**ALS Parameters** 



Agilent 7650A Automatic Liquid Sampler Installation, Operation, and Maintenance

# **Syringes and Needles**

Selecting a Syringe78Inspecting a Syringe80Installing a Syringe81Removing a Syringe85Replacing a Syringe Needle86

The 7650A ALS uses syringes as the sample-handling device. This chapter describes their properties and uses.



# **Selecting a Syringe**

1 Select the syringe type based on the inlet you are using and the volume of sample you want to inject (Table 7 and Table 8).

Nominal size (uL)	Maximum injection size (uL)
1	0.5
2	1
5	2.5
10	5
25	12.5
50	25
100	50

#### Table 7Standard syringes

<b>T</b> I I A	DTEE	
lahle X	PIFF-finned	nlunder svrindes
	I II L UPPOU	plunger synniges

Nominal size (uL)	Maximum injection size (uL)
10	5
25	12.5
50	25
100	50
250	125
500	250

- **2** Select a syringe. Refer to the Agilent catalog for consumables and supplies for part numbers and ordering information.
- **3** Select the appropriate syringe needle gauge (Table 9).

Inlet type	Column type	Needle gauge
Purged-packed, split or splitless (including MMI and PTV)	Any Applicable	23 gauge 26s gauge or 23/26s gauge tapered

Use syringe needles with a conical tip. Do not use sharp-tipped needles. They tear the inlet septum and cause leaks. Also, a sharp-tipped needle tends to wipe off on the septum as it exits resulting in a large solvent tail on the chromatogram (Figure 29 and Figure 30).









Figure 30 Needle shapes

# **Inspecting a Syringe**

Before installing a syringe:

1 Roll the syringe on the edge of a clean flat surface. If the tip of the needle moves in a circle, straighten the shaft by bending it slightly near where it connects to the syringe barrel and check it again (Figure 31).



Figure 31 Inspecting the syringe

**2** Check for a rough needle. The needle surface may have closely spaced concentric ridges that act like a miniature file and abrade pieces of the septum into the inlet or vial. The ridges are easy to see under 10X magnification.

If there are ridges, polish the needle by pulling it through a folded piece of fine emery paper between your finger and thumb until the ridges are gone. Be careful not to modify the tip of the syringe.

**3** Check for a sticky plunger. Slide the plunger of the syringe up and down a few times. It should move smoothly without sticking or binding. If it is sticky, remove the plunger and clean it with solvent.

# **Installing a Syringe**



To install a syringe (Figure 32):

Figure 32Installing a syringe

- **1** Unplug the ALS communication cable, and if desired, mount the ALS on a parking post, or lay the ALS on a work bench.
- **2** Open the ALS door.
- **3** Slide the syringe carriage to the top position.
- 4 Open the syringe latch by swinging it in a counterclockwise direction.
- **5** Lift the plunger carrier to the top position.
- **6** Carefully pass the syringe needle through the guide hole in the needle support foot.

7 Align the syringe flange with the flange guide and press the syringe into place, keeping the needle end in the guide hole of the needle support foot. Make sure that the flat edge of the syringe flange faces out (Figure 33).

### NOTE

Failure to correctly install the syringe flange into the flange guide will result in damage to the syringe plunger.



Figure 33 Syringe flange orientation

- 8 Close the syringe latch by swinging it clockwise until it snaps in place.
- **9** Loosen the plunger screw entirely by turning it counterclockwise until the stop is reached.
- **10** Slide the plunger carrier down until it is completely over the syringe plunger, and tighten the plunger thumb screw until finger-tight.
- **11** Manually move the plunger carrier up and down. If the syringe plunger does not move along with the carrier, repeat the previous steps until installed correctly. Be sure the plunger thumb screw is secure and tight. If the carrier is not completely attached to the syringe plunger it may become detached after a few injections.

### CAUTION

Repeating this movement can damage the syringe.

**12** Verify that the needle is inside the guide hole of the needle support foot. The needle should be straight and pass freely through the needle guide hole.

If the needle is bent or is outside the guide hole, remove the syringe and reinstall. See Figure 34 for a properly installed syringe.



Figure 34 Syringe carriage and needle support with syringe installed

**13** Close the ALS door.

- **14** Do the following only if the ALS was removed from the mounting post during installation:
  - **a** If necessary, plug in the ALS communication cable.
  - **b** Install the ALS on the mounting post. See "Installing the ALS" on page 37 for details.

# **Removing a Syringe**

To remove a syringe:

- **1** Unplug the ALS communication cable, and if desired, mount the ALS on a parking post.
- **2** Open the ALS door.
- **3** Slide the syringe carriage to the top position.
- **4** Completely loosen the plunger thumb screw until it reaches the stop, and lift the plunger carrier off of the syringe plunger.
- 5 Open the syringe latch by swinging it in a counterclockwise direction.

### CAUTION

Be careful not to bend the syringe needle. Only pull the syringe out of the carriage until clear. The needle bends easily when still seated in the needle support guide.

**6** Carefully pull the top of the syringe out of the flange guide, then lift the needle out of the needle support foot.

To install a syringe, see "Installing a Syringe" on page 119 for details.

# **Replacing a Syringe Needle**

The stainless steel needles used for 250-µm and 320-µm injections must be inserted into a glass syringe barrel. Select the correct size needle for the column you will use.

Needles for 250- $\mu$ m injections have silver-colored stops. Needles for 320- $\mu$ m injections have gold-colored stops. See your Agilent consumables and supplies catalog or the Agilent website (www.agilent.com/chem) for a list of syringes and needles.

To insert a needle into a syringe barrel (Figure 35):



- 1 Unscrew the syringe barrel cap and remove the spring.
- **2** Make sure the needle has a PTFE disk (Figure 35). If the syringe barrel does not have the PTFE disk, use the instructions in the syringe box to wrap the needle yourself.

- **3** Slide the spring and the cap down over the needle.
- 4 Insert the needle into the syringe barrel.
- **5** Screw the cap back on the syringe barrel.

**Syringes and Needles** 



Agilent 7650A Automatic Liquid Sampler Installation, Operation, and Maintenance

# **Vials and Bottles**

Preparing a Sample Vial 90 Select a sample vial 90 Select a vial septa 91 Fill a sample vial 93 Label a sample vial 92 Cap a sample vial 94 Preparing Solvent and Waste Bottles 96 Select the bottles 96 Fill the solvent bottles 97 Prepare the waste bottles 97 Placing Vials and Bottles in the Turret 98 How Many Sample Vials Can I Run? 100 Sandwich Injections 104 Example 2-layer sandwich injection 106 Example 3-layer sandwich injection 108

This chapter describes sample vials, solvent bottles, and waste bottles, including how to place them in the turret. It also discusses how to estimate the maximum number of samples that can be run before solvent bottles must be refilled or waste bottles must be emptied.



# **Preparing a Sample Vial**

### Select a sample vial

Use clear or amber glass sample vials with crimp caps, or screw-cap vials. Use amber glass vials for light-sensitive samples. Refer to your Agilent catalog for consumables and supplies for acceptable vial types. Incompatible sample vials cause turret errors.

Figure 36 shows the critical dimensions for sample vials used with the ALS. These dimensions do not make up a complete set of specifications.

Body Diameter (BD) =  $11.7 \pm 0.2$ Cap Diameter (CD) = BD × 1.03 maximum All dimensions in millimeters



### Select a vial septa

There are two types of septa used with crimp caps and screw-on caps, each with different resealing characteristics and different resistance to solvents.

- One type is natural rubber formulation coated with PTFE on the sample side. This septum is suitable for samples with a pH range of 4.0 to 7.5. They are less resistant to solvents after puncture and are more easily cored than silicone rubber septa. Coring may deposit septum pieces in the vial and affect your chromatograms.
- Another is high-quality, low-extractable silicone rubber septa, coated with PTFE on one or both sides. They are more resistant to solvents after puncture and to coring by the needle.

Refer to your Agilent catalog for consumables and supplies for more information.

Figure 37 shows the diameter for vial cap apertures.



Figure 37 Vial cap aperture specifications

### Label a sample vial

Some vials are available with a write-on spot for easy marking. If you choose to make and apply your own labels, Agilent Technologies recommends the positioning and maximum label thickness shown in Figure 38.





CAUTION

Correct sample vial dimensions are critical for proper operation. Vials and labels that do not meet these specifications may cause sampler errors. Service calls and repairs found to be due to vials and microvials that do not meet these specifications are not covered under warranty or the service contract.

### Fill a sample vial

Figure 39 shows the recommended fill volumes for sample vials of:

- 1 mL for the 2-mL vial
- 50  $\mu$ L for the 100- $\mu$ L vial

The air space in the vial is necessary to avoid forming a vacuum when sample is withdrawn. This could affect reproducibility.

CAUTION

Do not inject air into the vials to prevent this vacuum. This often damages the cap seal and may damage syringe needles.



\* Needle position based on default sampling depth

**Figure 39** Recommended fill volumes for sample vials

When developing your method, keep the following in mind:

- If you need to test a large amount of sample over repeated injections, divide the sample among several vials to obtain reliable results.
- When sample volume in the vial is low, contaminants from the previous sample injection or solvent washes may have a greater impact on the sample.

If you change suppliers, you may need to redevelop your method. Differing manufacturing practices for vial hardware sometimes cause variances in your results.

### Cap a sample vial



If using a sample vial with a screw cap top, be sure to completely tighten the screw cap before use.

Adjuster screw	Handle ——	
Adjuster screw		R
Adjuster screw Jaws Crimp cap Adjuster screw		
Jaws Crimp cap A Sample vial	Adjuster screw ———	,♥
Crimp cap  Sample vial	Jaws	
Sample vial	Crimp cap	
	Sample vial ————	—

**Figure 40** Crimping caps

To install the airtight crimp caps:

- **1** Clean the inside surfaces of the crimper jaws.
- **2** Place the crimp cap over the top of the vial.
- **3** Lift the vial into the crimper. Squeeze the handle until it reaches the adjuster screw.



Figure 41 shows acceptable and unacceptable vial caps.

Figure 41 Acceptable and unacceptable caps

Check each vial for proper crimping:

- 1 Be sure there are no folds or wrinkles on the part of the cap that wraps under the neck of the vial. To remove folds or wrinkles, turn the vial about 10° and crimp it again. Adjust the crimper for a looser crimp by turning the adjusting screw clockwise.
- **2** The cap should be finger-tight. If the cap is loose, adjust the crimper for a tighter crimp by turning the adjusting screw counterclockwise. Crimp the cap again. If the cap is too tight, the septum will distort and the vial may leak.
- **3** Be sure that each cap has a flat septum centered over the top of the vial.
  - If the septum is not flat, remove the cap, turn the crimper adjusting screw clockwise, and try again.
  - If the cap is not centered, remove the cap and make sure the new cap is flat on the top of the vial before you squeeze the crimper.

## **Preparing Solvent and Waste Bottles**

The solvent bottles hold solvent for rinsing the syringe between injections. The 7650A ALS dispenses the solvent washes and sample washes into waste bottles. The number of samples that can be analyzed may be limited by the wash or waste bottle capacity.

### Select the bottles

Solvent and waste bottles can be closed with either diffusion caps (a plastic cap with a hole; it retards evaporation while letting the needle enter freely) or septa. Agilent Technologies recommends diffusion caps (Figure 42) over septa for two reasons:

- The diffusion cap allows multiple entrances into a bottle without contaminating the liquid inside the bottle with small pieces of septum material.
- For common solvents, the rate of diffusion out of the bottle is less with a diffusion cap than with a septum that has been punctured multiple times with a standard syringe needle.



Figure 42 Four milliliter bottle used for solvent or waste

#### Fill the solvent bottles

Rinse and fill each solvent bottle with 4 mL of fresh solvent. The liquid level will be near the shoulder of the bottle. Good laboratory practice dictates using no more than 2.0 mL of the 4 mL solvent for syringe washes. The needle tip draws solvent 18.5 mm from the bottom of the vial

(Figure 43).



Figure 43 Needle tip position when withdrawing solvent

### Prepare the waste bottles

Empty and rinse each waste bottle after each multiple vial run. The syringe can discard about 4 mL of waste into the waste bottle (Figure 44).





#### **Vials and Bottles**

## **Placing Vials and Bottles in the Turret**

The turret provided with the 7650A ALS has 50 sample positions, four solvent positions, and four waste positions (Figure 45).



Figure 45 Turret labels

The labeled positions are defined in Table 10.

Position	Label	Bottle/Vial	
1 through 25	1 through 25	Sample vials, row 1	
26 through 50	26 through 50	Sample vials, row 2	
L2	24 and 49	Configurable sample vial positions Layer 2 vials	
L3	25 and 50	Configurable sample vial positions Layer 3 vials	
W	W	Waste bottles	
A	А	Solvent A bottles	
В	В	Solvent B bottles	

#### Table 10Turret labels

# How Many Sample Vials Can I Run?

The number of sample vials that you can run at one time is determined by the following parameters for your application:

- The number of sample injections per sample vial.
- The syringe size used: 1  $\mu$ L to 500  $\mu$ L.
- The syringe wash volume (Table 11).
- The number of solvent washes (both pre- and post-injection) required from each solvent bottle.
- The number of sample washes and solvent washes per sample injection that the ALS discards into each waste bottle.
- Number of waste bottles.

Syringe Size (µL)	80% syringe volume µL	40% syringe volume µL	30% syringe volume µL	20% syringe volume µL	10% syringe volume µL
1	0.8	0.4	0.3	0.2	0.1
2	1.6	0.8	0.6	0.4	0.2
5	4	2	1.5	1	0.5
10	8	4	3	2	1
25	20	10	7.5	5	2.5
50	40	20	15	10	5
100	80	40	30	20	10
250	200	100	75	50	25
500	400	200	150	100	50

Table 11Syringe wash volumes

### Solvent bottle equation

### CAUTION

The number of sample vials given by these equations are estimates. Solvent characteristics such as evaporation rate and surface tension may affect the capacity of the bottles.

This equation estimates the maximum number of sample vials you can run from **one** solvent bottle.

Maximum number of sample vials per solvent bottle

$$= \frac{2000}{V_{W} \times N_{SI} \times N_{SW}}$$

where:

 $V_W$  = Volume of Wash in  $\mu L$  taken from Table 11

N<sub>SI</sub> = Number of sample injections taken from each sample vial

 $N_{SW}$  = Number of Solvent washes (pre and post) per sample injected

#### Waste bottle equation

This equation estimates the maximum number of sample vials you can run per waste bottle.

Maximum number of sample vials per waste bottles used (W)

$$= \frac{V_{Waste}}{W_{Wash} \times N_{SI} \times N_{SS}}$$

where:

 $V_{Waste}$  = Volume of waste bottles used in µL. Use 4000.

 $V_{\text{Wash}}$  = Volume of wash in  $\mu$ L taken from Table 11.

 $N_{SI}$  = Number of sample injections taken from each sample vial.

 $N_{\rm SS}$  = Total number of pre- and post-solvent A or solvent B washes and sample washes per sample injected. When using both solvent A and B, the sample wash is divided evenly between both waste bottles for each row.

### CAUTION

We strongly recommend that *all* waste positions contain bottles at *all* times, regardless of which ones are actually used in the current configuration. This protects against accidental discharge of solvent into the turret body, which could damage it.

### **Reduced solvent and sample usage**

Solvent and sample washes by default use 80% of the syringe capacity. Solvent and sample can be conserved by reducing this volume. This is done by configuring the solvent saving setting.

### CAUTION

PTFE-tipped syringes must be used to lubricate the barrel wall. Standard syringes will fail quickly with limited lubrication.

The solvent wash can be set to use less than the usual 80%. This modifies the solvent wash process as follows:

- 1 The syringe draws in solvent to the percent specified, which can be as little as 10% of the syringe size.
- 2 The syringe and needle rise out of the solvent bottle.
- **3** The plunger rises to the 80% mark, rinsing the syringe barrel with solvent, followed by air.
- 4 The solvent and air are discharged into a waste bottle.

Confirm that sample carryover (see "Sample Carryover") is not a problem with the reduced solvent washes.

# **Sandwich Injections**

The 7650A ALS has the ability to draw liquid from multiple vials to create multilayered, or sandwich, injections. Each layer of sample, internal standard, or solvent can be separated by a layer of air (from 0% to 10% of the syringe size). Figure 46 and Figure 47 show examples of 2- and 3- layer sandwich injections.



Layer 1 (L1) Layer 2 (L2)

Layer 3 (L3)

Figure 47 3-layer sandwich injection



When loading vials for sandwich injections, refer to Figure 48 and note the following:

**Figure 48** Turret vial locations

- **Row 1** (outer row) The turret adds L2 to position 24 and L3 to position 25. L1 is always the active sample vial for position 1 to 23 (or 1 to 24 if L3 is not used).
- **Row 2** (inner row) The turret adds L2 to position 49 and L3 to position 50. L1 is always the active sample vial for position 26 to 48 (or 26 to 49 if L3 is not used).

For the following examples, we assume the use of a 7820A GC with an ALS installed in the front inlet location. Refer to "Setting the ALS Parameters" for more information.

#### Example 2-layer sandwich injection

Suppose you want to create a 2-layered sandwich injection using 2.0  $\mu$ L of sample and 15  $\mu$ L of solvent separated by 0.10  $\mu$ L air gaps (Figure 49).



**Figure 49** Example 2-layer sandwich injection with 2.0 µL of sample and 15 µL of solvent

- **1** Place the sample vial (L1) in turret position **1**.
- **2** Place the solvent vial (L2) in turret position L2 (positions 24 or 49).
- **3** Press [Front Injector] on the software keyboard.
- 4 Scroll to Injection Mode and select 2-LAYER.
- **5** Set the following parameters for the ALS:
  - Injection volume-2.0 µL
  - Airgap Volume-0.10 µL
  - L2 volume-15 µL
  - L2 Airgap Volume—0.10 µL

Note that for 2- and 3-layer injection modes, **Injection volume** and **Airgap volume** represent layer 1 (L1) parameters.

- **6** Store the method parameters:
  - a Press [Method] on the software keyboard.
  - **b** Scroll to an available method number.
  - c Press [Store] to select the method number.
  - d Press [Yes] to confirm.

- 7 Set up the sandwich injection sequence:
  - a Press [Seq] on the software keyboard.
  - **b** Scroll to Method # under Subseq 1 and press [On/Yes].
  - **c** Enter the method number from step 6 using the software keyboard, and press **[Enter]** to confirm the entry.
  - **d** Scroll to **Samples** and press [1] [.] [1] to set the vial range on the turret, and press [Enter] to confirm the entry.
- 8 Run the sandwich injection sequence:
  - a Press [Seq control] on the software keyboard.
  - **b** Scroll to **Start sequence** and press **[Enter]**. The sandwich injection sequence begins.

#### **Example 3-layer sandwich injection**

Suppose you want to create a 3-layered sandwich injection using 10  $\mu$ L of solvent A, 2.0  $\mu$ L of sample, and 15  $\mu$ L of solvent B separated by 0.10  $\mu$ L air gaps (Figure 50).



**Figure 50** Example 3-layer sandwich injection with 10 μL of solvent A, 2.0 μL of sample, and 15 μL of solvent B

- **1** Place the solvent A vial (L1) in turret position **1**.
- **2** Place the sample vial (L2) in turret position L2.
- **3** Place the solvent B vial (L3) in turret position L3. Refer to Figure 48 on page 105 for turret position information.
- 4 Press [Front Injector] on the software keyboard.
- 5 Scroll to Injection Mode and select 3-LAYER.
- **6** Set the following parameters for the ALS:
  - Injection volume—10 µL
  - Airgap Volume-0.10 µL
  - L2 volume-2 μL
  - L2 Airgap Volume-0.10 μL
  - L3 volume-15 µL
  - L3 Airgap Volume-0.10 µL

Note that for 2- and 3-layer injection modes, **Injection volume** and **Airgap volume** represent layer 1 (L1) parameters.
- 7 Store the method parameters:
  - a Press [Method] on the software keyboard.
  - **b** Scroll to an available method number.
  - c Press [Store] to select the method number.
  - d Press [Yes] to confirm.
- 8 Set up the sandwich injection sequence:
  - a Press [Seq] on the software keyboard.
  - **b** Scroll to Method # under Subseq 1 and press [On/Yes].
  - **c** Enter the method number from step 7 using the software keyboard, and press **[Enter]** to confirm the entry.
  - **d** Scroll to **Samples** and press [1] [.] [1] to set the vial range on the turret, and press [Enter] to confirm the entry.
- **9** Run the sandwich injection sequence:
  - a Press [Seq Control] on the software keyboard.
  - **b** Scroll to **Start sequence** and press **[Enter]**. The sandwich injection sequence begins.

**Vials and Bottles** 



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# **Running Samples**

Running a Sample 112 Injection volume 112 Interrupting a Run or Sequence 113 Sampler response to interruptions 113 Restarting an interrupted sequence 113

This chapter describes the process for running one or more samples.



## **Running a Sample**

### WARNING

# When running a sample, keep your hands away from the syringe needle. The needle is sharp and may contain hazardous chemicals.

To operate your automatic liquid sampler:

- 1 Install a clean syringe. See "Installing a Syringe" on page 81.
- 2 Fill the solvent bottles. See "Preparing Solvent and Waste Bottles" on page 96.
- **3** Place the solvent and waste bottles in the turret. See "Placing Vials and Bottles in the Turret" on page 98.
- **4** Load the sample vials into the turret. See "Preparing a Sample Vial" on page 90.
- **5** Prepare the instrument sequence. See your instrument or data system documents.
- **6** Run the sequence by pressing the start button on the instrument or in the data system. When the instrument is ready, the ALS begins the injections.

### **Injection volume**

The injection volume depends on the sample size and the syringe size.

- Syringe size may be 1  $\mu L,$  2  $\mu L,$  5  $\mu L,$  10  $\mu L,$  25  $\mu L,$  50  $\mu L,$  or 100  $\mu L$  when using the standard syringe carriage.
- Syringe size may be 250  $\mu$ L or 500  $\mu$ L when using the enhanced sample handling syringe carriage.
- Sample size may be 1-50% of the syringe size in 1% increments.

## Interrupting a Run or Sequence

The following events interrupt a run:

- Power failures-The power to the controlling instrument fails.
- Stop commands-[**Stop**] on the instrument is pressed, or the Stop Run/Abort option is selected from the Agilent data system.
- Safety or operator faults-The sampler recognizes these fault(s):
  - The ALS door was opened
  - Turret error
  - Plunger error
  - The ALS was moved on the instrument during injection

#### Sampler response to interruptions

If the interruption is caused by a problem that the sampler recognizes, a message appears on the instrument or Agilent data system. For more information, see "Error Messages" on page 159.

- Power failure-Aborts the run. Restart the sequence (see below).
- Stop commands-Interrupts the run. Restart the sequence (see below).
- Safety or operator faults—Aborts the run. Restart the sequence (see below).

#### **Restarting an interrupted sequence**

To restart an aborted sequence from the point of interruption:

- **1** Resolve the problem that caused the interruption.
- **2** The ALS automatically starts the run with the next vial in the sequence.

This allows normal operation to continue after an interruption without delaying the entire sequence.

**Running Samples** 



Agilent 7650A Automatic Liquid Sampler Installation, Operation, and Maintenance

# Part 5:

# **Maintenance and Troubleshooting**

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Agilent 7650A Automatic Liquid Sampler Installation, Operation, and Maintenance

# Maintenance

Periodic Maintenance 118 Installing a Syringe 119 Removing a Syringe 123 Changing the Turret 124 Replacing the Needle Support Guide 131 Adapting for Syringes Over 100 µL 133 Replacing the Syringe Carriage 134 Replacing a Syringe Needle 140 Aligning the 7650A ALS 142 Installing the Parking Post 144

The information in this chapter will assist you in keeping the sampler running smoothly.



## **Periodic Maintenance**

This section contains some suggestions for ensuring good performance of your 7650A ALS. The maintenance interval varies with the use of the instrument.

### CAUTION

Do not use any lubricants on the ALS. They may affect the chemical performance and damage the instrument.

### CAUTION

When cleaning the instrument, as described below, use a damp (not wet!), lint-free cloth. Do not use chemical cleaners.

On an occasional basis:

- ✓ Align the ALS. See "Aligning the 7650A ALS" on page 142 for details.
- ✓ Clean the turret and other surfaces.
- ✓ Wipe away any excess spill remains from the heat deflector underneath the turret. See "Changing the Turret" on page 124 for details.
- ✓ Clean the needle support guide and nearby surfaces on the ALS. Dust and dirt accumulate in these areas and can be picked up by the syringe needle and carried into the inlet.
- ✓ Inspect the needle support guide for signs of wear, and replace if necessary. See "Replacing the Needle Support Guide" on page 131 for details.
- ✓ Clean the ALS surfaces and door.
- ✓ Vacuum any dust found on or around the vents on the ALS.
- ✓ Make sure the ALS mounting post is tight.
- ✓ Be sure all cables are securely connected.

## **Installing a Syringe**



To install a syringe (Figure 51):

Figure 51Installing a syringe

- **1** Unplug the ALS communication cable, and if desired, mount the ALS on a parking post, or lay the ALS on a work bench.
- **2** Open the ALS door.
- **3** Slide the syringe carriage to the top position.
- 4 Open the syringe latch by swinging it in a counterclockwise direction.
- **5** Lift the plunger carrier to the top position.
- **6** Carefully pass the syringe needle through the guide hole in the needle support foot.
- 7 Align the syringe flange with the flange guide and press the syringe into place, keeping the needle end in the guide hole of the needle

support foot. Make sure that the flat edge of the syringe flange faces out (Figure 52).

### NOTE

Failure to correctly install the syringe flange into the flange guide will result in damage to the syringe plunger.



Figure 52 Syringe flange orientation

- 8 Close the syringe latch by swinging it clockwise until it snaps in place.
- **9** Loosen the plunger screw entirely by turning it counterclockwise until the stop is reached.
- **10** Slide the plunger carrier down until it is completely over the syringe plunger, and tighten the plunger thumb screw until finger-tight.
- **11** Manually move the plunger carrier up and down. If the syringe plunger does not move along with the carrier, repeat the previous steps until installed correctly. Be sure the plunger thumb screw is secure and tight. If the carrier is not completely attached to the syringe plunger it may become detached after a few injections.

## CAUTION

Repeating this movement can damage the syringe.

**12** Verify that the needle is inside the guide hole of the needle support foot. The needle should be straight and pass freely through the needle guide hole.

If the needle is bent or is outside the guide hole, remove the syringe and reinstall. See Figure 53 for a properly installed syringe.



Figure 53 Syringe carriage and needle support with syringe installed

**13** Close the ALS door.

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- **14** Do the following only if the ALS was removed from the mounting post during installation:
  - **a** If necessary, plug in the ALS communication cable.
  - **b** Install the ALS on the mounting post. See "Installing the ALS" on page 37 for details.

## **Removing a Syringe**

To remove a syringe:

- **1** Unplug the ALS communication cable, and if desired, mount the ALS on a parking post.
- **2** Open the ALS door.
- **3** Slide the syringe carriage to the top position.
- **4** Completely loosen the plunger thumb screw until it reaches the stop, and lift the plunger carrier off of the syringe plunger.
- **5** Open the syringe latch by swinging it in a counterclockwise direction.

### CAUTION

Be careful not to bend the syringe needle. Only pull the syringe out of the carriage until clear. The needle bends easily when still seated in the needle support guide.

**6** Carefully pull the top of the syringe out of the flange guide, then lift the needle out of the needle support foot.

To install a syringe, see "Installing a Syringe" on page 119 for details.

# **Changing the Turret**

The turret provided with the 7650A ALS has 50 sample positions, four solvent positions, and four waste positions (Figure 54).



Figure 54 Turret labels

The labeled positions are defined in Table 12.

Position	Label	Bottle/Vial
1 through 25	1 through 25	Sample vials, row 1
26 through 50	26 through 50	Sample vials, row 2
L2	24 and 49	Configurable sample vial positions Layer 2 vials
L3	25 and 50	Configurable sample vial positions Layer 3 vials
W	W	Waste bottles
A	А	Solvent A bottles
В	В	Solvent B bottles

 Table 12
 Turret labels

To change your turret, use the following instructions.

### **Remove the turret**

- **1** Remove all vials from the turret.
- **2** Open the ALS door.
- **3** Remove the syringe. See "Removing a Syringe" on page 123.
- **4** While holding the turret steady with one hand, rotate the turret hubcap counterclockwise until it releases from the turret motor hub (Figure 55).



Figure 55 Rotate the turret hubcap counterclockwise

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**5** Lift the turret hubcap up to remove it from the ALS.

Figure 56 Removing the turret

**6** Carefully lift the turret from the motor hub and remove it from the ALS (Figure 57). Avoid contact with the heat deflector and ALS casing when removing the turret.



Figure 57Removing the turret

## Install the turret

To install the turret:

**1** Open the ALS door (Figure 58).



Figure 58 Open the ALS door

**2** Align the tabs on the turret hub with the notch on the inside of the turret, and place the turret onto the turret hub (Figure 59).



Figure 59 Place the turret onto the turret hub

**3** Rotate the turret so that the injector opening faces toward the front of the ALS (Figure 60).



**Figure 60** Rotate the turret so that the injector opening faces toward the front of the ALS

**4** Place the turret hubcap onto the turret hub. Be sure to align the slots on the turret hubcap with the slots on the turret hub as shown in Figure 61.



Figure 61 Place the turret hubcap onto the turret hub

Installation, Operation, and Maintenance Part 5: Maintenance and Troubleshooting 5 While holding the turret in place, rotate the turret hubcap in a clockwise direction until it snaps into position on the turret hub. Make sure that the tabs on the turret hubcap align with the turret injector opening as shown in Figure 60.





- 6 Install the syringe. See "Installing a Syringe" for more information.
- 7 Close the ALS door.
- 8 Remove the ALS from the parking post and install the ALS on the mounting post. See ""Installing the 7650A ALS" for details.
- **9** Plug in the ALS communication cable.
- **10** When powered on, the ALS verifies the turret installation. A fault light will show if the turret is not installed correctly.
- 11 Align the ALS. See "Aligning the 7650A ALS" on page 142 for details.

## **Replacing the Needle Support Guide**

You must use the standard needle support guide (Figure 63).



Figure 63 Replacing the needle support guide

Replace the needle support guide when the support guide shows signs of wear:

- **1** Open the ALS door.
- 2 Remove the syringe. See "Removing a Syringe" on page 123 for details.
- **3** Slide the syringe carriage up to the top position.
- **4** Completely remove the T-10 Torx screw from the support guide. Be careful to not let the screw fall into the turret assembly.
- **5** Slide off the support guide.
- **6** Slide on the new support guide.

#### Maintenance

- 7 Replace the T-10 Torx screw and tighten.
- 8 Install the appropriate syringe. See "Installing a Syringe" on page 119 for details.
- **9** Close the ALS door.
- 10 Align the ALS. See "Aligning the 7650A ALS" on page 142 for details.

## Adapting for Syringes Over 100 µL

The 7650A ALS can perform large volume injections using syringe sizes over 100  $\mu$ L. To adapt the ALS and instrument for large volume injections, the G4521A Enhanced Sample Handling Syringe Carriage must be installed. To install the G4521A Enhanced Sample Handling Syringe Carriage, follow these steps:

- 1 If necessary, remove the current syringe from the ALS. See "Removing a Syringe" on page 123 for details.
- 2 Replace the standard syringe carriage with the G4521A Enhanced Sample Handling Syringe Carriage. See "Replacing the Syringe Carriage" on page 134 for details.
- **3** Install the appropriate syringe. See "Installing a Syringe" on page 119 for details.
- **4** If necessary, reinstall the ALS on the instrument. See "Installing the ALS" on page 37 for details.
- 5 Align the ALS. See "Aligning the 7650A ALS" on page 142 for details.

# **Replacing the Syringe Carriage**

Use the Standard Syringe Carriage for injections up to 100  $\mu$ L, and the G4521A Enhanced Sample Handling Syringe Carriage for injections over 100  $\mu$ L (Figure 64).



Standard Syringe Carriage

**Enhanced Sample Handling Syringe Carriage** 

**Figure 64** Standard and Enhanced Sample Handling Syringe Carriages with differences in appearance shown

The G4521A Enhanced Sample Handling Syringe Carriage can only handle syringes that are 100  $\mu L$  and greater.

To replace the syringe carriage assembly:

- **1** Remove all vials and bottles from the turret, and disconnect the ALS communication cable from the instrument.
- **2** If desired, remove the ALS from the mounting post and mount it on a parking post.
- **3** Open the ALS door.
- 4 Remove the syringe. See "Removing a Syringe" on page 123 for details.
- **5** Remove the turret. See "Changing the Turret" on page 124 for details.
- **6** Slide the syringe carriage down until its cable is accessible below the tower casing, and detach the cable from the assembly.
- 7 Completely loosen and remove the T-10 Torx screw at the top of the syringe carriage assembly (Figure 65).



Figure 65 Remove the T-10 Torx screw at the top of the syringe carriage assembly

#### Maintenance



8 Slide the syringe carriage completely up until the flange and latch are accessible.

**Figure 66** Removing the syringe carriage assembly

- T-10 Torx screw
- **9** Completely loosen and remove the T-10 Torx screw above the syringe latch (Figure 67).

Figure 67 Remove the T-10 Torx screw

- **10** Carefully remove the syringe carriage from the injector carriage.
- **11** Slowly position the replacement syringe carriage on the injector carriage. The syringe carriage will hang in place when positioned correctly.
- 12 Completely tighten the T-10 Torx screw above the syringe latch.

#### Maintenance



**13** Slide the syringe carriage completely down until the syringe carriage cable jack is accessible below the ALS casing (Figure 68).

**Figure 68** Syringe carriage cable jack

- T-10 Torx screw
- 14 Completely tighten the T-10 Torx screw at the top of the syringe carriage assembly (Figure 69).

Figure 69 Tighten the T-10 Torx screw at the top of the syringe carriage

- **15** Connect the cable to the syringe carriage assembly.
- 16 Slide the syringe carriage completely up.
- 17 Make sure you have the standard needle support guide installed. See "Replacing the Needle Support Guide" on page 131 for details.
- **18** Install the turret. See "Changing the Turret" on page 124 for details.
- 19 Install the syringe. See "Installing a Syringe" on page 119 for details.
- **20** Close the ALS door.
- **21** If necessary, plug in the ALS communication cable and install the ALS on the mounting post. See ""Installing the 7650A ALS" for details.
- 22 Align the ALS. See "Aligning the 7650A ALS" on page 142 for details.

## **Replacing a Syringe Needle**

The stainless steel needles used for  $250-\mu m$  and  $320-\mu m$  injections must be inserted into a glass syringe barrel. Select the correct size needle for the column you will use.

Needles for 250- $\mu$ m injections have silver-colored stops. Needles for 320- $\mu$ m injections have gold-colored stops. See your Agilent consumables and supplies catalog or the Agilent website (www.agilent.com/chem) for a list of syringes and needles.

To insert a needle into a syringe barrel (Figure 70):



- 1 Unscrew the syringe barrel cap and remove the spring.
- **2** Make sure the needle has a PTFE disk (Figure 70). If the syringe barrel does not have the PTFE disk, use the instructions in the syringe box to wrap the needle yourself.

- **3** Slide the spring and the cap down over the needle.
- 4 Insert the needle into the syringe barrel.
- **5** Screw the cap back on the syringe barrel.

# Aligning the 7650A ALS

This section explains how to perform the 7650A ALS alignment procedure. The ALS has been factory-aligned prior to shipment. The alignment procedure should only be run if the ALS hardware configuration has changed or if the Align Mode light comes on.

#### NOTE

Agilent does not recommend performing this procedure unless the Align Mode light turns on.

If the top, orange Align Mode light is on, the ALS will not operate again until the alignment procedure has successfully completed.

To align the ALS:

- **1** Remove all vials from the turret.
- **2** Open the ALS door and remove the syringe. See "Removing a Syringe" on page 123 for details.
- **3** Close the ALS door.
- **4** Use a long, narrow object to press the recessed Alignment button above the indicator lights. Avoid contact with anything behind or around the button inside the recessed hole (Figure 71).



The orange Align mode light is on during the alignment procedure:

- **a** The turret rotates to verify that the syringe was removed, then rotates to determine what type of turret is installed.
- **b** The syringe carriage moves all the way down, homes, then moves back up again.
- **c** The syringe carriage steps down until it touches the turret. This sets its position relative to the turret.
- **d** The plunger moves to calibrate stops.
- **e** The syringe carriage moves down and the turret rotates to set its position relative to the syringe carriage position.

#### NOTE

If any of these steps fail, the process will halt and the fault light will shine. If it halts at step **b**, confirm that the turret is installed correctly and press the Alignment button again. If it fails again, unplug the ALS, reconnect it, and try one more time before obtaining Agilent service.

**5** When the ALS stops and the green Ready light is on, the calibration is finished.

If this alignment was user-initiated (orange Align Mode light was off before pressing the Alignment button) and alignment fails, unplug the ALS, reconnect it, and it will reset to the old alignment values.

- **6** Open the ALS door and install the syringe. See "Installing a Syringe" on page 119 for details.
- 7 Close the ALS door.

# **Installing the Parking Post**

If desired, install the parking post on the 7890A GC (Figure 72) or 7820A GC (similar). If you have a 5975T LTM-GC/MSD, skip this section.



Figure 72 Parking post positions on a 7890A GC


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# Troubleshooting

Symptom: Fast Instrument Run Times Longer than Expected 146 Symptom: Variability 147 Symptom: Contamination or Ghost Peaks 149 Symptom: Smaller or Larger Peaks Than Expected 150 Symptom: Sample Carryover 152 Symptom: No Signal/No Peaks 153 Correcting Syringe Problems 154

The information in this chapter will assist you in keeping the sampler running smoothly.

If the chromatogram is not satisfactory, clearly something is wrong. Use this chapter to determine the probable cause and, in many cases, the cure for the problem.

This chapter deals only with sampler-related problems. However, many of the symptoms described here could also come from other sources, particularly the stability of the instrument temperature and its gas supplies.

If you cannot correct the problem, obtain Agilent service.



# Symptom: Fast Instrument Run Times Longer than Expected

Possible cause	Action
Sample vial usage is consistently alternating from turret row 1 and turret row 2.	Use simple sequencing if possible (vial position 1, followed by positions 2, 3, 4, etc.) or use vials in the same turret row to maximize productivity.

# Symptom: Variability



Figure 73 Retention times or areas are not reproducible

### Table 14Variability problems

Possible cause	Action	
Inlet septum is leaking.	<ul> <li>If the septum is leaking, replace it. If the septum you replaced experienced less than 200 injections, check for the following possible problems to prevent premature septum failure: <ul> <li>The septum retainer nut is too tight.</li> <li>The syringe needle is not straight.</li> <li>The syringe is not installed correctly.</li> </ul> </li> </ul>	
Syringe is worn or dirty.	If the syringe looks dirty or the plunger is sticking, clean the syringe with an appropriate solvent or follow the syringe manufacturer's cleaning instructions.	
Sample volume is too low or too high.	Check sample level. If the sample vials are not filled correctly evaporation or contamination may affect the analysis. The sample level hold will be approximately half the volume of the vial. See "Fill a sample vial" on page 93.	

Possible cause	Action	
Vial caps are loose.	Check vial caps. If you can turn the vial crimp cap by hand, they are too loose. Loose caps may cause volatile samples to change concentration over time. See "Cap a sample vial" on page 94. Screw caps, if not sufficiently tight, can come loose in the mixer and even come off.	
Sample is not stable.	<ul> <li>Check the sample stability. Some samples change with heat or ultraviolet light. There are several ways to reduce changes to unstable samples:</li> <li>Use amber sample vials.</li> <li>Store the samples in a protected environment.</li> </ul>	
Sample size varies.	Install a new syringe. If the sample size varies, the syringe is probably not precise or the plunger is worn. Variation may be due to syringes with removable needles because of dead volume or needle-to-needle variation.	
Air bubbles are in the needle.	<ul> <li>If air bubbles are in the needle, user variable speed and a sample draw speed slow enough to avoid bubble formation. See "Setting the 7650A ALS Parameters" on page 72.</li> <li>If this does not help and the sample is viscous, try the following: <ul> <li>Increase the viscosity delay time.</li> <li>Dilute the sample in an appropriate low-viscosity solvent.</li> </ul> </li> </ul>	

 Table 14
 Variability problems (continued)

# Symptom: Contamination or Ghost Peaks

Possible cause	Action	
Vial cap septum is dissolving in solvent. Ghost peaks sometimes appear when small pieces of septum material dissolve in the sample. Make several blank runs to determine the presence or absence of the ghost peaks.	<ul> <li>Check for the following:</li> <li>Be sure the vial septum is flat. If the vial septum is not flat, the needle tends to core the septum and drop pieces into the sample. See "Cap a sample vial" on page 94.</li> <li>Check the needle. If the syringe needle has burrs, it could cut pieces of the septum and push them into the sample.</li> <li>Check the vial septum. If the vial septum is not resistant enough to the solvent you are using, try a more resistant type.</li> </ul>	
Sample vials are contaminated.	Ghost peaks are sometimes caused by contaminated sample vials. Try new or clean vials to see if ghost peaks disappear. Store new vials in a contaminant-free location.	
Inlet septum is giving off volatiles.	Make several blank runs with a small piece of aluminum foil backing the inlet septum. If the contamination peaks disappear, they were probably due to the septum. Try replacing the septum you usually use with another type.	
Column is contaminated. High molecular weight samples that contain residues may cause the syringe, the inlet liner, or the first few inches of column to become contaminated.	<ul> <li>Do the following:</li> <li>Replace or clean and deactivate the inlet liner.</li> <li>Examine the first few inches of a capillary column for foreign material by holding a light behind it. If possible, remove the contaminated section.</li> </ul>	
Sample is not stable.	Some samples change with heat or ultraviolet light. Check the sample stability. There are several ways to reduce the change: • Use amber sample vials. • Store the samples in a protected environment	

#### Table 15 Contamination or ghost peak problems

## Symptom: Smaller or Larger Peaks Than Expected





#### Table 16Peak size problems

Possible cause	Action
You are comparing a chromatogram without needle fractionation against one with needle fractionation.	Check your injection mode. In the normal injection mode, the sampler uses fast injection to deliver a representative amount of the sample. Fast injection minimize needle fractionation. Chromatograms from manual injection or slower auto injection devices show higher levels of low molecular weight materials versus higher molecular weight materials because the volatiles boil out of the needle faster than the higher weight materials.

Possible cause	Action	
You are using a packed inlet and a 530-µm column.	Check your inlet. Capillary columns used with packed inlets have some inherent sample discrimination characteristics.	
There is a leak in the system.	<ul> <li>Replace the septum and check the fitting for leaks. If the leaking septum has experienced less than 200 injections.</li> <li>To prevent future premature failures, be sure that: <ul> <li>The septum retainer nut is not too tight.</li> <li>The syringe needle is straight.</li> <li>The syringe is installed correctly.</li> </ul> </li> </ul>	
Sample is not stable.	Some samples change with heat or ultraviolet light. Check the sample stability. There are several ways to reduce the change: • Use amber sample vials. • Store the samples in a protected environment.	
Vial caps are loose.	Check the vial caps. Loose vial caps can cause selective loss of lighter materials from a sample. The cap should not rotate easily if installed properly. See "Cap a sample vial" on page 94.	

Table 16	Peak size problems	(continued)

# Symptom: Sample Carryover



Figure 75 Blank run showing carryover peaks

### Table 17Carryover problems

Possible cause	Action Check the run parameters for the number of sample and solvent washes. The number of washes needed depends on your application. See "Sample Carryover" on page 63.	
Number or type of washes is insufficient.		
You ran out of solvent.	Check the solvent bottles. If the solvent level is below 2.5 mL, the syringe cannot reach the solvent. Replace the remaining solvent with 4 to 4.5 mL of fresh solvent. See "Preparing Solvent and Waste Bottles" on page 96. Check the waste bottles. If the waste level is near the neck of the bottle, replace it with an empty bottle.	
Syringe is worn or dirty.	If the syringe look dirty or the plunger is sticking, clean the syringe with an appropriate solvent or follow the syringe manufacturer's cleaning instructions. If the syringe seems worn, replace it.	
Samples (vial-to-vial) are of immiscible types.	In this situation, the sample and solvent washes may not rinse the syringe properly. Increase the number of wash cycles or use a solvent that rinses a variety of sample types.	

# Symptom: No Signal/No Peaks

Possible cause	Action Verify that the syringe plunger is secured by the plunger screw. If the plunger screw is loose, tighten it. See"Installing a Syringe" on page 81. Check the syringe needle for plugging. If the syringe is plugged, replace or clean the syringe.	
Syringe plunger is malfunctioning.		
Sample level is too low in vial.	If there is no or very little sample in the vial, the needle may not be able to reach it. See "Fill a sample vial" on page 93. Alternately, you may edit your method to adjust the needle sampling depth. See Sampling offset in "Setting the 7650A ALS Parameters" on page 72.	
Sample is viscous.	<ul> <li>If the sample is viscous, try the following:</li> <li>Increase the viscosity delay time.</li> <li>Dilute the sample in an appropriate low-viscosity solvent.</li> </ul>	

#### Table 18Signal/Peak problems

## **Correcting Syringe Problems**

### WARNING

When troubleshooting the ALS, keep your hands away from the syringe needle. The needle is sharp and may contain hazardous chemicals.

Several things can cause syringe needles to bend. When you find one, check for the following conditions before installing a replacement:

- ✓ Was the syringe installed properly in the syringe carriage?
- ✓ Are you using the correct syringe type? Is the combined length of the syringe barrel and needle 126.5 mm? For more information, see "Selecting a Syringe" on page 78.
- ✓ Is the needle support guide clean? Remove any residue or septum deposits. For more information, see "Periodic Maintenance" on page 118.
- ✓ Is the inlet septum nut to tight? For more information, see your instrument operating documentation.
- ✓ Is the septum of the crimp cap centered over the sample vial? For more information, see "Cap a sample vial" on page 94.
- ✓ Are the inside diameters of the sample vial, microvial insert, and vial cap septum at least 5 mm? For more information, see "Preparing a Sample Vial" on page 90.



Sometimes things do not work as expected. In most cases, the nature of the problem is signalled by status lights on the 7650A ALS or by error messages on the instrument's display. If this happens, use this chapter to discover the probable cause and the corrective action to take.



### Faults

The 7650A ALS status is indicated by the three lights on the front panel (Figure 76).



Figure 76 7650A ALS status lights

During normal operation, the green Ready light is on. If the ALS is busy, the green Ready light is flashing.

If another combination of lights is on, a fault has occurred.

Use the information in this chapter to try to solve or identify the problem before obtaining Agilent service.

Lights	Possible cause	Action
All lights are off.	<ul> <li>The line voltage to the instrument is off.</li> <li>The ALS cable or connection to the instrument is bad.</li> <li>Your instrument requires service.</li> </ul>	<ol> <li>Verify the ALS is properly connected to the instrument.</li> <li>Check the power source for your instrument.</li> <li>Obtain Agilent service.</li> </ol>

Table 19ALS status lights

Lights	Possible cause	Action
Fault light is on.	<ul> <li>The ALS door is open.</li> <li>ALS is not properly mounted to the instrument.</li> </ul>	<ol> <li>Ensure that the ALS door is closed.</li> <li>If the fault light stays on, obtain Agilent service.</li> <li>Ensure that the ALS is mounted properly. For more information, see "Installing the ALS" on page 37.</li> <li>Ensure the correct mounting post is installed. See "Installing the ALS" on page 37.</li> <li>If the Fault light stays on, obtain Agilent service.</li> </ol>
Fault light is flashing two times.	• Syringe error.	• Re-install or replace the syringe.
Fault light is flashing three times.	Turret error.	Re-install the turret.
Fault light is flashing four times.	Plunger error.	
Fault light is flashing five times.	Shifter error.	<ul> <li>Restart the instrument. If the error reoccurs, obtain Agilent service.</li> </ul>
Align Mode light is on.	<ul> <li>The system was not initialized.</li> <li>There is an ALS memory error.</li> </ul>	<ol> <li>Verify the turret is properly installed. See "Changing the Turret" on page 124.</li> <li>Perform the alignment procedure to initialize the system. See "Aligning the 7650A ALS" on page 142.</li> <li>If alignment fails, obtain Agilent service.</li> </ol>
Align Mode light is flashing.	• Customer pushed the align mode button.	<ul> <li>The alignment and calibration process is currently underway. Let the process finish.</li> </ul>

Table 19	ALS status	lights	(continued)	)
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Lights	Possible cause	Action
All lights are on.	<ul> <li>There is a board failure.</li> <li>There is a firmware revision conflict.</li> </ul>	<ol> <li>Check all cable connections.</li> <li>Turn the instrument off, then on again.</li> <li>If the lights remain on, obtain Agilent service.</li> </ol>

 Table 19
 ALS status lights (continued)

### **Error Messages**

Table 20 lists the sampler error messages as reported on the instrument. If you receive an error message that is not shown below, record it. Make sure that your instrument is properly configured and that your sample vials and equipment match your method and/or sequence. If the problem continues, report your error message to Agilent service.

#### Table 20Error messages

Message	Probable cause	Suggested action
Front (or Back) door open or injector not mounted		• See "Faults" on page 156.
Front (or Back) injector com error	<ul> <li>There is a communications error between the ALS and the instrument.</li> </ul>	Obtain Agilent service.
Front (or Back) injector incomplete injection	<ul> <li>The syringe needle is bent.</li> <li>The plunger or syringe carriage is operating incorrectly during injection.</li> </ul>	<ol> <li>See "Correcting Syringe Problems" on page 154.</li> <li>Remove the syringe from the ALS and check the plunger for stickiness or binding. Replace the syringe if necessary.</li> <li>Restart the sequence.</li> <li>If the error occurs again, obtain Agilent service.</li> </ol>
Front (or Back) injector reset	• There is an interruption in the power supply from the instrument.	Obtain Agilent service.
Front (or Back) plunger error	<ul> <li>The syringe plunger is sticking or not securely connected to the plunger carrier.</li> <li>The plunger solenoid is binding.</li> <li>The plunger carrier encoder is inoperable.</li> </ul>	<ol> <li>Remove the syringe and check it for plunger stickiness or binding. Replace the syringe if necessary. For more information, see "Inspecting a Syringe" on page 80.</li> <li>Check the viscosity of the sample against the viscosity parameter. Reset the viscosity parameter if necessary.</li> <li>Restart the sequence.</li> <li>If the error occurs again, obtain Agilent service.</li> </ol>

Message	Probable cause	Suggested action
Front (or Back) syringe error	<ul> <li>The syringe carriage motor is defective.</li> <li>The syringe is not currently installed or is an incorrect type.</li> <li>The syringe carriage sensor is inoperable.</li> </ul>	<ol> <li>Ensure the syringe is installed correctly. For more information, see "Installing a Syringe" on page 81.</li> <li>Ensure the syringe meets specifications.</li> <li>If the syringe needle is bent, see "Correcting Syringe Problems" on page 154.</li> <li>Restart the sequence.</li> <li>If the error occurs again, obtain Agilent service.</li> </ol>
Front (or Back) turret error	<ul> <li>Something has interfered with the turret rotation.</li> <li>The turret motor/encoder assembly is inoperable.</li> <li>The turret type was changed while the power was on and the turret alignment procedure was not performed.</li> <li>Turret is loose.</li> </ul>	<ol> <li>Clear any obstructions.</li> <li>Check the Align Mode light. If it is on, perform the alignment procedure. See "Aligning the 7650A ALS" on page 142.</li> <li>Tighten the top of the turret.</li> <li>If the error occurs again, obtain Agilent service.</li> </ol>
Injector not present	<ul> <li>There is a board failure in the ALS or instrument.</li> <li>The ALS communication cable is bad or not securely connected to the instrument.</li> <li>There is a cable failure in the instrument.</li> <li>Your method specifies an incorrect inlet location (method mismatch).</li> </ul>	<ol> <li>Make sure that the ALS communication cable connection is secure.</li> <li>Check your method to make sure it uses the appropriate ALS inlet location.</li> <li>If the error remains, obtain Agilent service.</li> </ol>
Injector offline	<ul> <li>There is a board failure in the ALS or instrument.</li> <li>The ALS communication cable is bad or not connected.</li> <li>There is a cable failure in the instrument.</li> </ul>	<ol> <li>Make sure that the ALS communication cable connection is secure.</li> <li>If the error remains, obtain Agilent service.</li> </ol>

### Table 20Error messages (continued)

Message	Probable cause	Suggested action
Invalid sequence	<ul> <li>The sequence is set up for the wrong injection device.</li> <li>Hardware required by the sequence is not installed and configured.</li> <li>The instrument configuration was changed during sequence execution.</li> <li>The ALS communication cable is bad or not connected properly.</li> </ul>	<ol> <li>Make sure that the connection to the instrument is secure.</li> <li>Verify the sequence parameters against the instrument configuration.</li> <li>If the error remains, obtain Agilent service.</li> </ol>
No injector	<ul> <li>The cabling connection to the instrument became loose during a run.</li> <li>An ALS board or instrument board failed during a run.</li> </ul>	<ol> <li>Make sure that the connection to the instrument is secure.</li> <li>If the error remains, obtain Agilent service.</li> </ol>
Prerun >10 min	• The instrument is Not Ready.	• Check for Not Ready and other instrument messages to determine the cause.

### Table 20Error messages (continued)



The following pages list the replacement parts for the 7650A ALS. Also refer to the Agilent website at www.agilent.com/chem for the most up-to-date replacement parts and firmware listings.



## **7650A ALS Replacement Parts**

Table 21 and Figure 77 list and show the replacement parts for the 7650A ALS module.

ltem	Description	Part no.	Qty/Assy
1	Injector tower (new/exchange)	G4567A	1
2	Syringe carriage (standard)	G4513-60550	1
3	Syringe carriage (enhanced sample handling)	G4521-63000	1
4	Turret assembly	G4567-40192	1
5	Turret cap	G4567-40510	1
6	ALS mounting post	G4513-20561	1
7	Needle support guide (standard)	G4513-40525	1
8	Communication cable	G4514-60610	1

Table 217650A ALS replacement parts



**Figure 77** 7650A ALS replacement parts

Installation, Operation, and Maintenance Part 5: Maintenance and Troubleshooting



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