Model U6K Universal Liquid Chromatograph Injector **Instruction Manual** Waters Associates



Waters Associates

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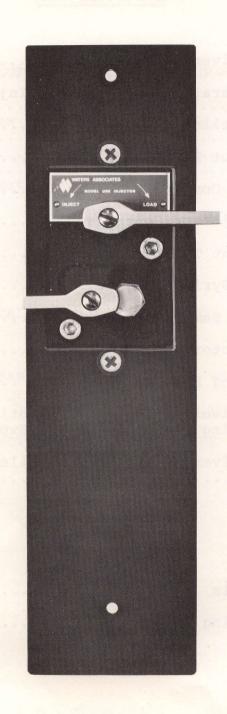


Figure 1-1. Model U6K Universal Injector

SECTION 1. GENERAL INFORMATION

1.1 INTRODUCTION

1.1.1 PURPOSE OF MANUAL

This manual provides the user with the instructions necessary to understand, operate, and maintain the Model U6K Universal Liquid Chromatograph Injector (Figure 1-1) manufactured by Waters Associates, Inc.

1.1.2 FEATURES OF THE U6K INJECTOR

The Waters Associates Model U6K Universal Liquid Chromatograph Injector (referred to as the U6K Injector) is a specially-designed injector, that enables the user to load samples and make injections, at system pressure up to 6000 psi (420 bars) without interruption of the solvent flow. The U6K injector can accommodate the full range of analytical and preparative injections, from a fraction of a microliter to 2 milliliters, without making any changes to the injector.

Reproducible sample loading is performed at atmospheric pressure, while solvent is simultaneously being delivered to the system at full pressure. When an injection is made, an integral chart recorder automatically marks the injection point on the chromatogram. The automatic chart marking capability is standard when using the U6K Injector with a detector which has a remote chart marking circuit (such as the Waters Associates Model 440 Absorbance Detector and R-400 Series Refractive Index Detectors). A chart marker accessory can be used for marking the sample injection point when the detector does not have a remote chart marking circuit. Peak spreading is comparable to that of a conventional septum injector.

The U6K is a septumless injector, which employs a patented configuration for loading the sample by back-filling the sample loading loop. Any syringe, microliter, or milliliter, with a 1.90 in. (48 mm) long x 0.020-in. (0.51 mm) - (25 gauge) outside-diameter needle can be used to load the injector. The syringe capacity is chosen according to the desired injection volume. The syringe, not the loop, determines sample size. For example, in analytical work, the volume loaded into the loop for injection will only occupy a few microliters of the entire loop volume. A nominal 2 milliliter sample loading loop is supplied as standard. Capacities for even larger-scale preparative work are provided by simple loop replacement.

The U6K Injector fits all Waters ALC-200, ALC/GPC-200, and ALC/GPC-500 Series Liquid Chromatographs. It is available as an installed unit or as a free-standing component.

1.2 FUNCTIONAL DESCRIPTION

Sample loading and operation are depicted in the series of drawings in Figure 1-2. Reference will also be made to the upper and lower handles of Figure 1-1. Beginning with Figure 1-2a, valves A, B, and C are closed. Solvent flow is from the pump outlet directly through a restrictor to the column. The small arrows indicate solvent flow. The other channel, which is the sample loading loop, is isolated from the flow stream and contains solvent at atmospheric pressure. The Sample Loading Plug is in place.

In Figure 1-2b, the Sample Loading Plug has been removed and the syringe has been inserted into the sample loading loop. To remove the plug, the lower handle in Figure 1-1 must be turned. In doing so, vent valve C is opened. This opens the sample loading loop to the atmosphere making it possible to displace solvent, which is already in the loop, with sample as it is delivered from the syringe. The valve design provides access to the most forward part of the sample loading loop enabling the initial volume of sample to be loade at the very front of the loop. As the sample size increases, it extends from the front toward the back of the loop, displacing an equivalent volume of solvent out through the vent valve C. The sample can occupy any fraction of the total loop volume since sample size is controlled by the user, not the loop. The standard sample loop size supplied with the instrument permits injections up to as large as 2 ml; however, there is no restriction on the use of either a larger or smaller size loop if necessary. After the sample has been loaded into the loop, the syringe is withdrawn and the Sample Loading Plug is replaced. To retain the Sample Loading Plug, the lower handle in Figure 1-1 is returned to its original position. This also closes vent valve C, sealing the sample loading loop. It is now possible to pressurize the loop.

At this point, the upper handle in Figure 1-1 is turned to the 'INJECT' position, opening valves A and B, and momentarily closing the two chart marker switches, causing the inject point to be marked on the chromatogram. This configuration is indicated in Figure 1-2c. Now, because it is the path of least resistance, solvent from the pump preferentially passes through the loop, carrying the sample onto the column. After allowing sufficient operating time to displace all of the sample out of the loop and onto the column, the sample handle can be turned to the 'LOAD' position, closing valves A and B. The sample loading operation can now be repeated without affecting the operation of the system.

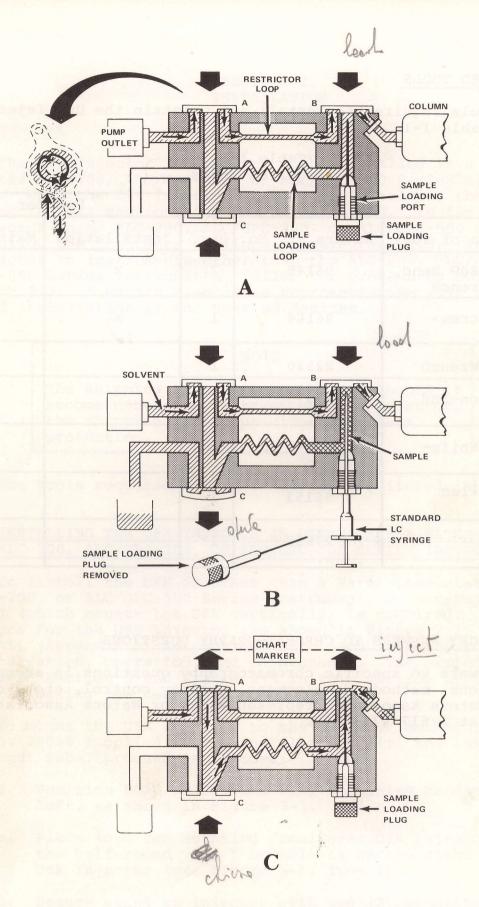


Figure 1-2. Schematic Operation of Universal Injector

1.3 REQUIRED TOOLS

The tools required to install and maintain the U6K Injector are listed in Table 1-1.

Table 1-1.
Required Tools

			Use for	
Description of Tool	Waters Pt. No.	Qty.	Installation	Maintenance
5/16 in., 80° Bend, Open-End Wrench	96148	2	х	х
Phillips Screw- Driver	96166	1	x	х
#4 Spline Wrench	22530	1		Х
1/2 in. Open-End Wrench	27375	1		х
6-1/2 in. Knife- Edge File	96149	1		x
3/16 in. Allen wrench	96151	1		х
1/4 in. Allen wrench	27953	1	PAUMA I DANS	х

1.4 HOW TO GET ANSWERS TO CHROMATOGRAPHY QUESTIONS

For answers to specific chromatography questions in areas such as applications, methods development, quality control, etc., contact your local Waters Associates representative or Waters Associates home office at 1-617-478-2000.

SECTION 3. START-UP/OPERATION

3.1 CONTROLS

Figure 3-1 illustrates and outlines the functions of the Model U6K Injector controls.

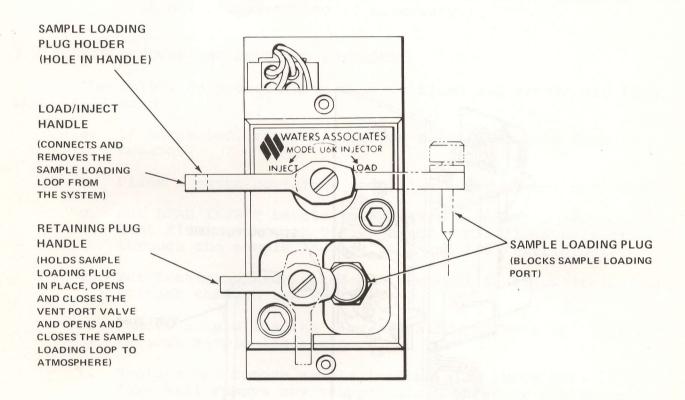


Figure 3-1. Model U6K Injector Controls

3.2 U6K INJECTOR START-UP PROCEDURE

During testing at the factory, the last solvent pumped through the U6K Injector was methanol. This enables the U6K user to start the injector with any organic solvent miscible with methanol. If using a solvent with limited solubility in methanol, an intermediate solvent (see paragraph 3.4) must be used to flush the U6K prior to use. To prepare the U6K Injector for system operation, perform the steps outlined in paragraphs 3.2.1 and 3.2.2.

3.2.1 VENT TUBE CONNECTION

To connect the vent tube, refer to Figure 3-2 and proceed as follows:

a. Slide vent tube (Part No. 24036 supplied in start-up kit) over vent output tube, and position end of vent output tube so that it is in line with the center of the sample loading port. This configuration will prevent a siphoning effect from draining the sample loading loop.

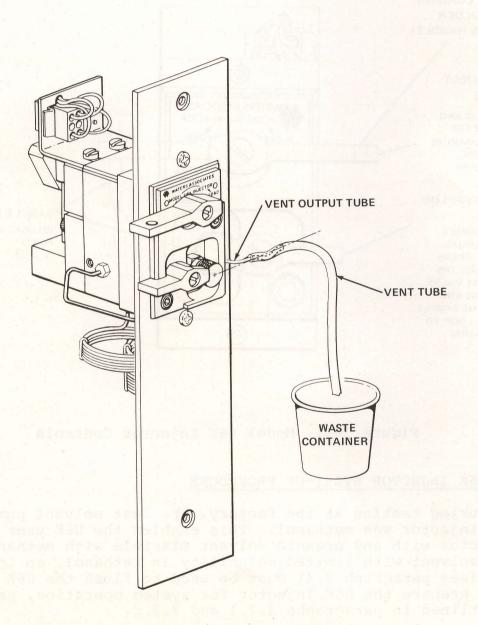


Figure 3-2. Model U6K Vent Tube Connection

CAUTION

If a tight-fitting vent tube (i.e., 1/16-in. (1.6 mm) inside diameter) is used, a siphoning effect may develop which will drain the sample loading loop.

b. Run the vent tube down to a waste container. Do not allow end of vent tube to contact liquid in waste container. (Cut off excess tubing if necessary.)

3.2.2 AIR REMOVAL AND FLUSHING PROCEDURE

The following procedure is used to flush and remove air from the U6K Injector:

- a. If connected, remove 'injector-to-column' tube from column.
- b. Place a waste container under 'injector-to-column' tube.
- c. Set LOAD/INJECT handle to the INJECT position and start solvent flowing through U6K Injector (this flows solvent through the sample loading loop and the restrictor).
- d. Set Retaining Ring handle to vertical position (this flows solvent through the vent).
- e. Remove sample loading plug and allow solvent to flow through sample loading port.
- f. Replace and remove sample loading plug three more times. This will remove any trapped air bubbles or contamination on plug wire.
- g. Allow approximately 6 milliliters of solvent to flow through the U6K Injector.
- h. Replace the sample loading plug.
- i. Stop the solvent flow and connect the 'injector-to-column' tube to the column.
- j. Set LOAD/INJECT handle to LOAD position.
- k. Set Retaining Ring handle to horizontal position.
- 1. The U6K Injector is now ready for system operation (see paragraph 3.3 for proper operating procedures).

3.3 OPERATING PROCEDURES

To operate the U6K Injector, proceed as follows:

CAUTION

To prevent damage to the TFE plug seal (Figure 5-2, Item 13), it is important to use a syringe with the proper size and type needle (Figure 3-3). In addition, the syringe needle and plug wire (Figure 5-2, Item 10) must be free from bent or burred tips.

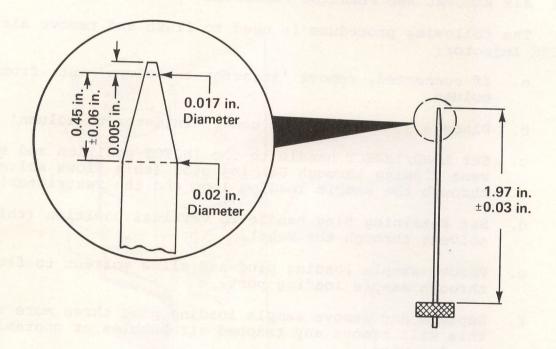


Figure 3-3. Recommended Syringe Needle

Start

a. Set LOAD/INJECT handle (Figure 3-4) to LOAD (right) position.

Loop Sample

b. Set Plug Retaining handle (Figure 3-4) to opened (vertical) position.

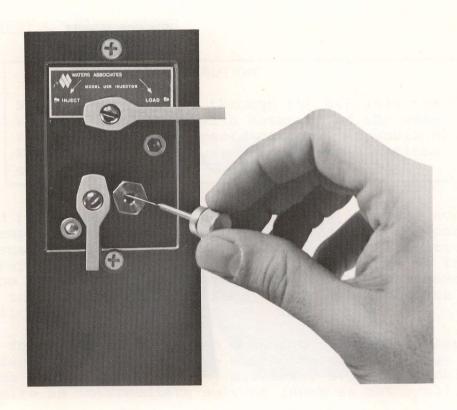


Figure 3-4. Removing U6K Sample Loading Plug

c. Remove Sample Loading Plug (Figure 3-4). The hole in the LOAD/INJECT handle is provided as a convenient holder for the Sample Loading Plug during sample loading and prevents making an injection without the Sample Loading Plug in place.

CAUTION

Care should be taken not to contaminate the wire at the end of the Sample Loading Plug.

- d. Insert syringe into sample loading port until syringe bottoms (see Figure 3-5).
- e. Load desired volume of sample into U6K Injector.
- f. Remove syringe and replace Sample Loading Plug.
- g. Set Plug Retaining handle to closed (horizontal) position.

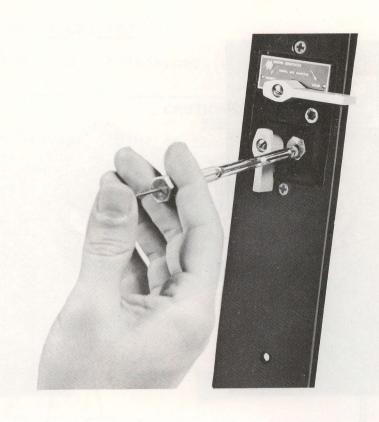


Figure 3-5. Loading Injector with Sample

Inject Sample

h. Set LOAD/INJECT handle to INJECT (left) position.

After Injection

i. Position LOAD/INJECT handle as follows:

NOTE

ALLOW SUFFICIENT TIME FOR SAMPLE TO BE MOVED ONTO THE COLUMN

 If running a gradient or if the next sample is not ready to be loaded leave LOAD/INJECT handle in INJECT position.

or

2. If operating in RECYCLE or if the next sample is ready to be loaded return LOAD/INJECT handle to LOAD position.

3.4 SOLVENT CONSIDERATIONS

CAUTION

Never pump solvents through the U6K Injector which are reactive with material of the wetted surfaces (see specifications, Appendix A). For example, never use strong concentrations of Halide ions.

Good practice in liquid chromatography involves avoiding solvents or samples containing foreign particles. If the purity of your sample is suspect, a Waters Associates Sample Clarification Kit should be used. Particles of approximately 0.5 micrometers or larger can be removed from either aqueous or organic solvents with these kits. Additional information on these Sample Clarification Kits is given in Waters Publication DS 043.

The following applies when changing solvents: Changes involving two miscible solvents may be made directly. Changes involving two solvents that are not totally miscible, e.g., from chloroform to water, require an intermediate solvent (such as methanol).

3.4.1 CHANGING SOLVENT

To change miscible solvents, proceed as follows:

NOTE

The following steps must be performed for each intermediate solvent required.

- a. Set LOAD/INJECT handle (Figure 3-1) to INJECT (left)position.
- b. Flow new solvent through injector long enough to insure that the previous solvent is flushed out of the system. Approximately three injector volumes (6 milliliters) are required.
- c. Set Retaining Plug handle (Figure 3-1) to vertical position and remove sample loading plug. Allow approximately 3 ml of solvent to flow from vent tube and out of sample loading port.
- d. Set LOAD/INJECT handle to LOAD (right) position.
- e. Set Retaining Plug handle to horizontal position.
- f. The U6K Injector is now ready to operate with the new solvent.