## 305 Pump User's Guide

# SOLUTIONS TO A COLUTIONS AT WORK FOR YOU

# 305 Piston Pump User's Guide

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### **Safety**

Read this section carefully before installing and operating the pump.

For safe and correct use of the pump, it is essential that both operating and service personnel follow generally accepted safety procedures as well as the safety instructions given in this document, the 305 Pump User's Guide.

The instrument described in this document is 305 high pressure piston pump for use in single or multi-pump applications. It can control Gilson Model 306 slave pumps in multi-pump applications. It should only be used in the laboratory or similar indoor environment, by qualified personnel. If the instrument is used in a manner not specified by Gilson, the protection provided by the instrument may be impaired.

Ensure that the ventilation fan on the Piston Pump operates and is not obstructed when the instrument is installed.

Voltages present inside the instrument are potentially dangerous. If there is a problem with the instrument, the power cable should be removed until qualified service personnel have repaired it. This is to prevent anyone from inadvertently using the instrument, thus causing possible harm to themselves, or damage to the instrument itself.

The leakage current of this instrument is within the limits allowed by safety standards for laboratory equipment. An efficient ground connection is imperative for the physical protection of the user.

Power supply cord reference 7080316106 is for use in France and Germany. Power supply cord reference 7080316105 is for use in USA and Canada. For other countries contact your local Gilson distributor. You must only use the type of fuse described and specified in this document: 2.0 Amp type "T" slow blow for use where the power supply is between 100 V and 120 V, 1.0 Amp type "T" slow blow fuse for use where the power supply is between 220 V and 240 V.

However, adequate protection including clothing and ventilation must be provided if dangerous liquids are used. In case of incidental spillage, carefully wipe with a dry cloth, taking into account the nature of the spilled liquid and the necessary safety precautions.

Cleaning, installation, dismantling, maintenance, adjustment and repair should only be performed by personnel trained in such work, and who are aware of the possible dangers involved. This instrument must not be sterilized, using an autoclave, or any other mechanical device. When you need to clean this instrument, use one of the three following methods:

1	- a	clean	drv	cloth
1	- a	Cicari	QI y	cioui,

- 2 a cloth dampened with water,
- 3 a cloth dampened with soapy water.

If a cloth dampened with soapy water is used to clean the pump, only domestic soap may be used. No other form of detergent or chemical may be used.

These electronic and hazard symbols appear on the pump:

Symbol	Explanation
~	Alternating current
	PROTECTIVE CONDUCTOR TERMINAL
I	On (Supply switch)
0	Off (Supply switch)
4	Caution, risk of electric shock
Î	Caution (refer to User's Guide)

Introduction 1

Gilson's 305 Master pump conform to the standard specified in the 'Declaration of Conformity' certificate (reference LT801354) supplied with the instrument.

The 305 Master pump is designed as a system controller. It can operate as a stand-alone isocratic pump or as a system controller, to deliver fluids (liquids or liquefied gas when specially equipped). As a system controller, the 305 Master pump controls a complete pumping system, elution pumps and injection pump.

The 305 Master pump can operate in three different modes. These modes are:

Flow: The 305 pump provides a constant flow rate. The pump starts when the Run

key is pressed and stops when the Stop key is pressed. The flow mode is for

isocratic use only.

Dispense: The 305 dispenses a specified volume. The pump starts when the Start key is

pressed and stops when the specified volume has been dispensed. The

dispense mode is for isocratic use only.

Program: The 305 controls a multi-pump system with up to 2 slave elution pumps and

1 slave injection pump. In this mode, the 305 Master pump can create gradients of flow rate and composition, open and close outputs to control

other instruments and wait for signals from other instruments.

The operation of each mode is explained in Chapter 4.

The present software version is 3.01.

#### **Using this Manual**

The 305 piston pump is a precision instrument which is simple and easy to use. To gain the maximum from the instrument, you should:

- Read the description of the instrument in chapter 2.
- Install the instrument as shown in chapter 3.
- Follow the operating instructions given in chapter 4.

#### **Unpacking**

The 305 piston pump is packed in a single carton. Upon receipt of your instrument, carefully unpack the unit and inspect it for possible damage. This should be done immediately. Check the contents of the carton against the parts list to verify that all parts are included and undamaged. The parts list is given in Appendix A. Do this now, even if the unit will not be used immediately. Report any damage to the responsible carrier immediately. Read the description in chapter 2 to become familiar with the instrument, its different parts and their names.

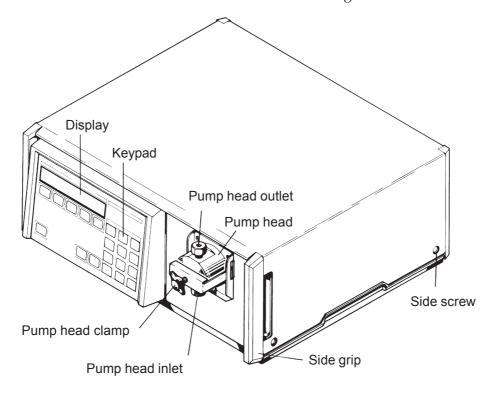
Introduction 1

Description 2

This chapter describes the physical layout of the 305 piston pump. It describes the main body of the 305 and the position of the electrical connectors on the rear panel.

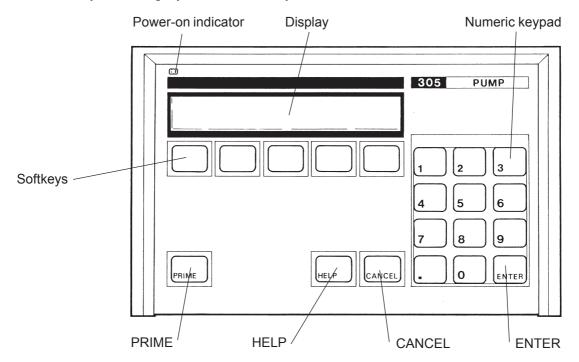
#### **Front view**

The figure below shows a front view of the 305 Master pump with a pump head mounted. There is a keypad which consists of a display, a numeric keypad and soft keys for programming the 305. The pump head is mounted on the right hand side.



#### **Keypad**

The figure below shows the keypad with the numeric keys, the display and the soft keys.



**Display**: two 24-character lines are used to dispaly parameters commands and messages.

**Softkeys**: their functions are determined by the software and may change from menu to menu. The present functions are displayed above each softkey.

**PRIME**: the pump runs at its maximum flowrate until you press the STOP stoftkey.

**HELP**: dispays advice and instructions at any time, with no effect on the operation of the pump.

**CANCEL**: cancels your last entry before it has been stored in the memory.

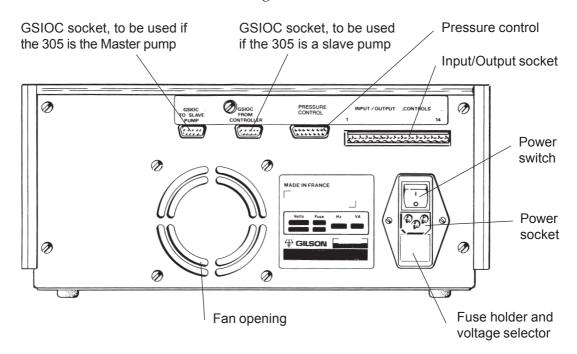
**ENTER**: confirms a selection or parameter value and stores it in the memory.

**Numeric keypad**: this is used to key in values during programming. The parameter being modified is always underlined with a flashing cursor.

#### **Rear View**

The figure below shows a rear view of the 305 with the electrical connectors. The function of each connector is as follows:

- **GSIOC TO SLAVE PUMP** Connection to a slave pump.
- **GSIOC FROM CONTROLLER** Connection to a computer.
- PRESSURE CONTROL
  Connection to the manometric module.
- INPUT/OUTPUT CONTROL Connector for the 305 inputs and outputs.
- Power switch On/off power switch.
- Power receptacle
  Voltage selector and fuse holder.



Installation 3

This chapter describes how to install the 305 Master pump. It is recommended that you follow the installation instructions in the order that they are presented in the manual.

#### **Electrical Installation - Power**

For safety reasons, the 305 is shipped without the fuses installed and with the voltage selector in the 220/240 Volt position. You must:

- Insert the correct fuses.
- Set the voltage selector to your local voltage.

#### **Inserting the Fuses**

Ensure that the power cord is not connected before starting to install the fuses. Follow the procedure below to install the **two** fuses.

- The voltage selector and fuse holder is located under neath the power socket. See opposite figure.
   Pull the voltage selector out of the power receptacle. This is done by gently levering the selector out using a small screwdriver.
- Pull out the drawer as shown in the figure opposite. Insert the first fuse into the clips.
- Push the drawer back into position.
- Pull out the drawer for the second fuse which is on the other side of the voltage selector. Insert the second fuse into the clips.

The instrument requires two fuses to be installed. The type of fuses required are: 2.0 Amp type "T" slow blow for 100 -120 V, 1.0 Amp type "T" slow blow for 220-240 V.

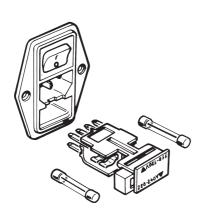
For safety reasons, piston pumps are delivered without fuses installed. Fuses must be installed by the user upon delivery.

#### Selecting the Voltage

The 305 can be set to operate at 100/120 volts or 220/240 volts. The different voltages are selected depending on the orientation of the fuse holder.

#### To set the voltage to 100/120 volts:

Insert the fuse holder with the numbers 110/120 on the bottom, facing the small white arrow.



#### To set the voltage to 220/240 volts:

Insert the fuse holder with the numbers 220/240 on the bottom, facing the small white arrow.

For safety reasons, do not connect the power cord until you have finished assembling the instrument.



#### **Mechanical Installation**

This section explains how to install the pump head, the mast clamp and mast. The pump head and mast clamp for each pump should be installed before positioning the modules.

#### **Pump Head Installation**

The pump head is shipped in a hard case to protect it during transit. Unpack the pump head from its case and check that all of the parts are included. Follow the procedure below to install the pump head.

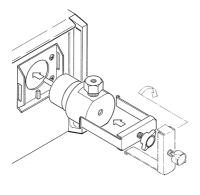
- Insert the pump head into the front aperture of the pump. See the opposite figure. The notch at the bottom of the pump head body must be fitted onto the matching pin on the pump, just below the aperture. This notch ensures that the inlet port is on the bottom and the outlet port is on the top.
- Holding the pump head in place with one hand, set the clamp diagonally over the head.
- Turn the clamp clockwise into position in the slots on both sides of the pump head.
- Tighten the thumb screw until the clamp holds the pump head securely. Make sure that the clamp ends are secured in their slots on both sides.

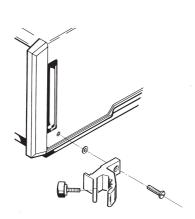
See the pump head User's Guide for more information on the pump head.

#### **Mast Installation**

The mast is used to stabilise a system when several modules are stacked on top of each other. It can also used to hold the prime/purge valve and a manual injection valve. The mast clamp should be installed before positioning the 305 Master pump in a system. The mast is added after all of the modules have been put in place. Follow the procedure below to install the mast clamp.

- Remove the side screw holding the module cover. See the opposite figure.
- Screw on the mast clamp.





Fix one clamp onto each pump in the system. After all the modules have been positioned, the stainless steel mast can be secured within the clamps. The lower end of the mast should be level with the bottom of the lowest pump.

# 305 Master 306 Det.

# 305 Master 306 pump B 231

#### **Positioning the Modules**

Before putting each module in position, make sure that each module is ready, i.e. that the fuses have been installed and that any mechanical installation is finished.

The physical positioning of each of the modules in your system will depend on your type of system. Some suggested layouts are given below. These layouts have been desigend to make the hydraulic and electrical connections as simple as possible.

#### Manual Injection System

This is a binary gradient system with two pumps, one manometric module, one mixer and one detector. The pumping system consists of one 305 Master pump and one 306 slave pump. The different modules should be located as shown in the figure opposite.

The detector is located at the bottom of the stack and the pumps and other modules are positioned over it. The 305 Master pump should be the top pump. This makes the hydraulic connections to the manometric module as short as possible which gives the best results. It is also easy to read the display and to use the keypad.

#### **Auto-Analytical System**

This is a binary gradient system with two pumps, one manometric module, one mixer, one detector and one auto-sampler.

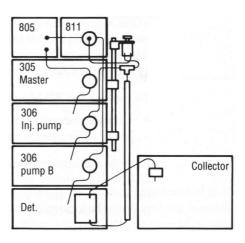
The pumping system consists of one 305 Master pump and one 306 slave pump. The 305 Master pump should be the top pump. This makes the hydraulic connections to the manometric module as short as possible which gives the best results. It is also easy to read the display and to use the keypad. The figure opposite shows the layout with a Gilson 231-402 auto-sampler. The next figure shows the layout with a Gilson 232-402 auto-sampler.

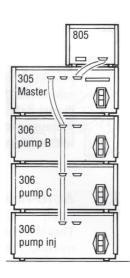
#### **Auto-Preparative System**

This is a binary gradient system with two elution pumps, one injection pump, one manometric module, one mixer, one detector and one fraction collector.

In this configuration, automatic injection is performed by the injection pump, located below the master pump.

The pumping system consists of one 305 Master pump and two 306 slave pumps. The 305 Master pump should be the top pump. This makes the hydraulic connections to the manometric module as short as possible which gives the best results. It is also easy to read the display and to use the keypad (see the opposite figure).





# Slave pump I.D. Number B 2 C 3 Inj 4

#### **Control Connections**

After positioning all of the modules in the system, it is necessary to connect each module to the system controller. Two types of electrical connection must be made.

- The manometric module must be connected to the 305 Master pump.
- All of the slave pumps must be connected to the Master pump using the GSIOC cable provided.

#### **Connecting the Manometric Module**

The manometric module has two functions, to dampen the pulsations of the pump and to supply the current pressure value to the Master pump. The Master pump needs this information to accurately control the flow rate and to ensure that the system pressure is not above or below the control limits entered in the program. The opposite figure shows the connection for the manometric module.

# **Connecting the 305 to other Gilson Modules**

The 305 Master pump and the other modules in the system communicate using the Gilson Serial Input/Output Channel (GSIOC). Each slave module controlled by the 305 Master has a GSIOC connector on its rear panel and is connected to the 305 Master using the GSIOC cable provided. It is possible to connect the 305 Master pump to two slave modules using this cable. If there are more than two slave modules, two GSIOC cables can be connected together. The opposite figure shows the GSIOC connections for a system with 3 elution pumps and on injection pump.

To connect the 305 Master pump to the slave pumps: Connect the socket marked GSIOC TO SLAVE on the rear panel of the 305 Master pump to the slave pumps using the GSIOC cables provided.

Each slave pump and the injection pump must have the GSIOC ID number set as appropriate, see the opposite table. Ensure that their respective ID numbers have been correctly set. This can be checked by Installation 3

looking at the rear of the 306 pumps and ensuring that the correct switch (2, 3 or 4) has been switched into the bottom position. The switches are labelled REMOTE, and are numbered 1 to 8 from left to right.

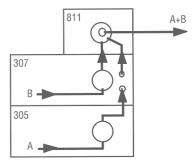
#### Connecting the 305 to a 307

The 305 Master pump can directly control a 307 pump without the addition of a manometric module and with pressure display on the 305 screen.

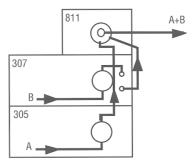
In this case, the value 2 must be entered in the 307 software as a GSIOC identification number for the 307 pump. If this is omitted, the pressure value dispalyed by the 305 will remain at zero. The remedy is to first correct the 307 identification, then to switch off and on the two pumps.

Hydraulically, the 307 should normally be used for solvent B, and set-up 1 is recommended (rather than set-up 2) for the following reasons:

- As it is usually the case in Gilson gradient systems, solvent B, of higher elution strength than A (and normally more expensive), does not enter the pulse dampener. This makes purge easier.
- Tubing is shorter and fewer elbows are required.



Set-up 1



Set-up 2

#### **Hydraulic Connections**

The hydraulic connections for the 305 pump head are made using the tubing provided in the standard accessory package. Connect the 305 pump head input with the inlet tubing assembly provided with the pump head. The connections to the 305 pump head output should be made using stainless steel, titanium tubing or plastic tubing.

Installation 3

#### **Input/Output Connections**

For coordination with surrounding equipment, electrical contacts are used.

The input/Output connector is a 14-pin terminal block connector. Connections are made to the inputs and outputs using the connector supplied in the standard accessory package.

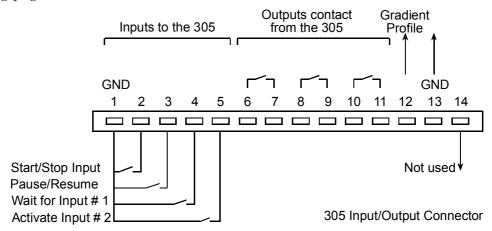
The function and pin numbers for each input and output are as follows.

#### **Operation of the Inputs**

To activate an input, you must connect it to ground or 0 V. This is usually done using a relay output, with one side connected to 0 V (Pin 1) and the other side connected to the input. When the output is closed, the input is connected to 0 V and is activated. Each of the four inputs are described in detail in the following pages.

Inputs	Outputs
Start/Stop	Out # 1
Pause/Resume	Out # 2
IN # 1	Out # 3
IN # 2	

Pin#	Function
1	Ground
2	Start/Stop Input
3	Pause/Resume Input
4	IN # 1 Input
5	IN # 2 Input
6	Out # 3 Output
7	Out # 3 Output
8	Out # 2 Output
9	Out # 2 Output
10	Out # 1 Output
11	Out # 1 Output
12	<b>Gradient Profile Out</b>
13	Gradient Profile GND
14	Not connected



#### The Start/Stop Input

The start/stop input is used to start and stop the 305 pump using an external relay contact.

This input is only activated when the input changes from open to closed or from closed to open. The operation for each mode is given opposite.

Mode	Input	Result
Flow	Closed Open	Start Flow Stop Flow
Dispense	Closed Open	Start Dispense Stop Dispense
Program	Closed Open	Start Program Stop Program

In the Program mode, Stop does not stop the flow, it only stops the program from continuing. The flow will continue with the flow rate and composition which existed when the stop input was activated. The program will restart from the beginning when the contact is opened. At the end of each operation, the solvent consumption will be displayed. In the Dispense mode, the Start input can be activated with a pulse.

#### The Pause/Resume Input

Mode	Input	Result
Flow	0.000	Pause Flow Resume Flow after Pause
Dispense		Pause Flow Resume Flow after Pause
Program		Pause Program Restart Program

The Pause/Resume input is used to pause and restart the 305 pump using an external contact.

This input is only activated when the input changes from open to closed or from closed to open. The operation of each mode is shown in the table opposite.

In the Program mode, this input can be configured to obtain a pause with or without flow. This choice is offered inside the software branch I/O, within the screen:

I. Pause/Prog is w. flow

The default value is with (w) flow, press change to obtain a pause without (w.o) flow. During a pause with flow, the flow will continue with the flow rate and composition which existed when the pause input was activated. The program will continue from the same point in the from the same point in the program when the resume contact is opened. The pause without input is of particular interest when the 305 is used as a metering pump to feed a reagent in high pressure reactors. In this application, the 305 can wait for correct values of external parameters (temperature, pressure, composition) before adding more reagent.

#### The IN # 1 Input

Timed event	Input	Result
Wait input # 1 Closed	Open	Wait IN # 1 is closed
Wait input # 1 Closed	Closed	Continue with program
Wait input # 1 Open	Open	Continue with program
Wait input # 1 Open	Closed	Wait IN # 1 is open

For more information, see Programming the inputs in Chapter 4.

This input only operates in the Program mode. It is fully programmable. It can be used to make the program wait until a piece of equipment is ready.

An example for this input is when the pumping system is waiting for a signal from a sample injector or a fraction collector.

Installation 3

#### The IN # 2 Input

This input is activated when closed. Activating this input while Program mode is selected, causes File 13 to run. If nothing is programmed in File 13 or if File 13 is already running, this input will be ignored. This input can be used to start a special program if an external signal is received, for example a warning signal from a temperature measuring system.

Mode	Input	Result
Program	Open	No effect
Program	Closed	Start File 13

#### **Output Controls**

The 305 Master pump has three output relays.

Each output relay consists of two terminals.

These terminals can be connected together (contacts closed) or not connected together (contacts open).

All of the outputs are electrically isolated from each other and from ground. These contacts can be used to control other equipment, e.g. to turn on or off another piece of equipment.

For more information, see Programming the outputs in Chapter 4.

Installation 3

Operation 4

After switching ON the 305, the display shows the following information for one second:

Model 305 VX.X Manometric Module: M805

This indicates the pump version, software version, VX.X, and the Manometric Module which is connected. If the Manometric Module is not properly connected, **None** is displayed. After this step, one of the Ready-to-Run screens is displayed.

#### **Priming the Pump Head**

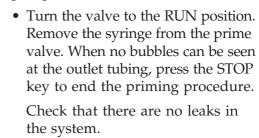
Do not run the pump when the pump head is dry. This can result in severe pump head damage.

Check that the solvent bottle is filled with HPLC grade, degassed solvent or buffer. Immerse the inlet tubing filter into the solvent reservoir. Make sure that all of the hydraulic connections are properly made.

Before priming, all electrical connections must be made, and all hydraulic connections in place. All pumps present in the system, A, B, C and Inj can be primed in the Program Mode using the 305 Prime command. However, the pumps must be connected and declared present in the "Setup Pump Hardware" procedure, page 4-5.

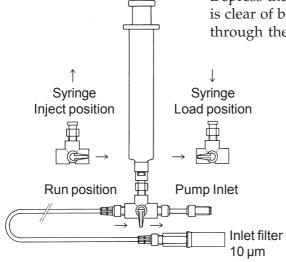
For 5SC, 10SC, 10WSC and 10WTi pump heads, use the syringe supplied with the pump head to prime the pump as follows:

- Attach the syringe to the luer fitting of the low pressure prime valve (refer to the figure below).
- Draw liquid into the syringe with the low pressure prime valve in the SYRINGE LOAD position.
- Turn the valve to the SYRINGE-INJECT position. Press the PRIME key on the front panel of the 305, the screen will indicate which pumps are present, and you simply select which pump you wish to prime by pressing the **Pump** key until the desired pump has been selected. Press Run and the pump will start running at its maximum speed. Depress the syringe (if used) until the pump inlet is clear of bubbles and some liquid has passed through the pump outlet.



For the 25SC, 50SC, 100SC, 25WTi and 200WTi pump heads,

• prime the pump directly without syringe or valve.



#### **Using the Keypad**

The keypad consists of numeric keys, dedicated keys such as Enter and Prime, 5 white soft keys and a 2 line 24 character display. The function of each part of the keypad is as follows.

Numeric keys: Used to enter numeric values(to

define parameters).

PRIME: Runs the pump at maximum flow

rate.

HELP: Displays help messages.

CANCEL: Cancels a value before it is entered

into the memory.

ENTER: Enter a value into the memory.

The 24-character display is used to indicate the flow rates, solvent compositions etc... The bottom line of the display is used to present the soft key options above the 5 white soft keys. Pressing one of the white soft keys selects the option displayed directly above it. The following soft key options will occur frequently and should be noted.

Quit: Return to the Ready-to-Run screen.

Next: Brings you to the next screen.

Prev: Brings you to the previous screen.

Time is expressed in minutes and hundredths of a minute. For example a time display of 2.50 min is 2 minutes and 30 seconds.

The words "key in" mean enter in a numerical value. A flashing cursor on the screen underlines the current parameter to be entered/modified.

The symbol # is used in this guide to show the factory set value (the default value).

The symbol • is used in this guide to show a key which must be pressed or a value which must be entered.

#### **Setting Up the Pump**

The software for the 305 Master pump can be explained with the help of the chart in Figure 15. The chart has 4 software branches.

Pump: This is used to enter data about each pump

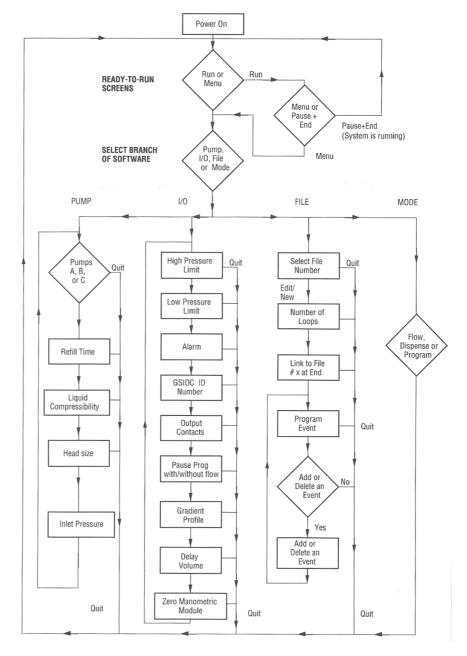
in the system, this means pumps A, B, C and the Injection Pump, if present.

I/O: This is used to define the Input/Output

functions.

File: This is used to write a method program.

Mode: This is used to select the mode of operation.



With a new pumping system, you must enter data about each pump connected, i.e. Refill Time, solvent Compressibility and pump Head Size and Inlet Pressure. This is done using the Pump branch of the software. Data concerning the overall pumping system, for example high pressure limit, low pressure limit etc. is done using the I/O branch of the software. The File branch is used for writing files of method programs. The Mode branch is used to operate the system in one of three possible modes, Flow, Dispense, or Program Mode.

The specifications for **Pump** and **I/O** must be set for a new system setup, or when the physical setup of the pumping system is changed, for example, changing a pump head size or removing or introducing a pump.

#### **Set Up Pump Hardware (PUMP)**

A pumping system consists of a minimum of 1 pump and a maximum of 3 elution pumps and 1 injection pump. Pump A, the first elution pump (the 305 Master pump), is always present.

The sequence of screens and soft key command options for the setup of pump hardware is shown in page 4-6.

To go to the pump setup menu:

- press Menu
- press Pump

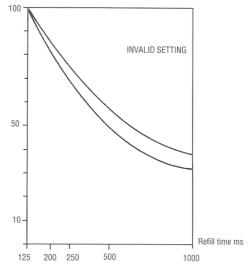
Use this menu to enter data about pump A.

- Select pump A by pressing the soft key below A
- The sequence for defining parameters is:
  - 1. Refill time
  - 2. Liquid Compressibility
  - 3. Head size
  - 4. Inlet Pressure (for liquefied gas)

A value for each parameter is keyed in using the keypad and is stored in memory by pressing Enter. This automatically brings you to the next menu screen. If you do not want to change the value already stored in memory, press the **Next** soft key or press Enter.

The maximum flow rate depends on the Head Size and the refill time. If the refill time is too long, a message **Invalid settings** flashes when you run the program. The refill time or flow rate must be lowered. The figure below shows a curve of refill times versus flow rate.



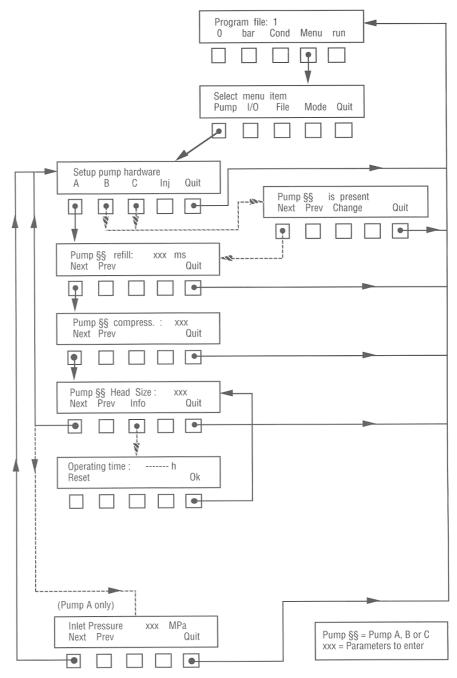


#### Pump A Refill time

The refill time is the time required for the piston return stroke. Normally it is set at the lowest value (125 ms) to give the fastest refill time. If cavitation or degassing occurs, then a higher value must be used. The minimum value is 125 ms ( the default value) and the maximum value is 1000 ms.

- # The default value is 125ms.
- Key in the refill time and press Enter.

This brings the menu screen onto compressibility.



# **Pump A Compressibility**

This data is used to calculate the flow rate compensation for the compressibility of the solvent. The minimum value is 0 and the maximum value is 2000 Mbar<sup>-1</sup>. Compressibility values for commonly used solvents at atmospheric pressure are listed in Appendix E. The values for the most common solvents are:

Solvent	X <sub>0</sub> (Mbar <sup>-1</sup> )
Water	46
Methanol	123
Acetonitrile	99

# The default value is 46 for water.

• Key in the value for the solvent being used with pump A and press Enter.

This menu will not appear if there is no manometric module connected to the system.

### **Pump A Head Size**

This parameter is the size of the pump head fitted to the pump. The pump head size is marked on the face of the pump head. Possible values are 5, 10, 25, 50, 100 and 200.

# The default value is 200.

• Key in the value for the pump head size on pump A and press Enter.

The three parameters for pump A are now entered.

Press the **Info** soft key to display the operating time for the pump head. This time can be reset to zero by pressing the **Reset** soft key. This should be reset every time the pump head has routine maintenance or when a new pump head is installed. Pressing **Ok** returns to the pump head size display.

Press the **Next** soft key to return to the **Setup pump** hardware menu to setup the parameters for other pumps in the system. Press the **Quit** soft key to return to the first Ready-to-Run screen. The next menu to program is the **Input/Output** parameter setup.

### **Inlet Pressure**

The Inlet Pressure (P0) for liquefied gas is the pressure at the inlet of the pump head of the 305. For example, when using carbon dioxide at a temperature of 22°C, the value should be defined as 6 MPa. The minimum value is 0 for solvents used in Liquid Chromatography and the maximum allowed value is 10 MPa. The default value is 0 MPa.

A table of inlet pressure values for  $CO_2$  is shown below.

Ambient temperature (°C)	15	20	22	25	30	31 (T <sub>c</sub> )
Pressure P0 (MPa)	5.1	5.8	6.0	6.5	7.2	7.4 (P <sub>c</sub> )

- Key in the desired inlet pressure according to the ambient temperature.
- Press Enter.

The four parameters for pump A are now entered.

# **Entering the Parameters for Other Pumps in the System**

You must tell the 305 Master pump if the slave pumps B, C, and Inject are present in your system. By default, pumps B, C, and Inject are set as present. This can be changed by pressing the **Change** soft key when defining the relevant pump.

If a pump is defined as present, the parameters Refill Time, Compressibility and Head Size must be defined.

If pump B is defined as absent, the Pump C is automatically set as absent.

### Summary:

- press the **Menu** soft key.
- press the **Pump** soft key.
- Select pump (A, B, C or Inj). (Define pumps B, C or Inj as present or absent.)
- Key in the desired refill time. (Minimum time 125 ms, maximum time 1000 ms.)
- Key in the solvent compressibility. (Minimum value 0, maximum value 2000 Mbar<sup>-1</sup>.)
- Key in the relevant Pump Head Size. (5, 10, 25, 50, 100, or 200.)
- Key in the Inlet Pressure. (Minimum value 0 MPa, maximum value 10 MPa.)

When all pumps have been defined, press the **Quit** soft key.

# Input/Output Parameter Set Up (I/O)

The I/O menu is used to enter data about parameters associated with the complete system. To go to the Input/Output parameter setup procedure:

- press the Menu soft key
- press the I/O soft key

The sequence for defining parameters is:

- 1. High pressure limit.
- 2. Low pressure limit.
- 3. Alarm On/Off.
- 4. GSIOC ID number.
- 5. Output Contacts.
- 6. Pause/Program with or without flow.
- 7. Gradient profile selection.
- 8. Delay Volume selection.
- 9. Zero Manometric Module.

The sequence of menu screens and soft key options is shown on page 4-6.

# **High Pressure Limit**

If the pressure reading from the manometric module rises above the defined limit, the pump will stop. The sequence following a high pressure error is described page 4-10.

The pressure can be displayed in three different units; bar, MPa, or kpsi. Change the units displayed by pressing the soft key below the units currently indicated on the display.

The maximum value is the maximum pressure limit for the manometric module connected in the system. The 305 Master pump knows which manometric module is connected and will refuse any value which is outside the range for that model.

- # The default value is the maximum pressure allowed by the manometric module.
- Key in the pressure limit that applies to your system and press Enter.

### **Low Pressure Limit**

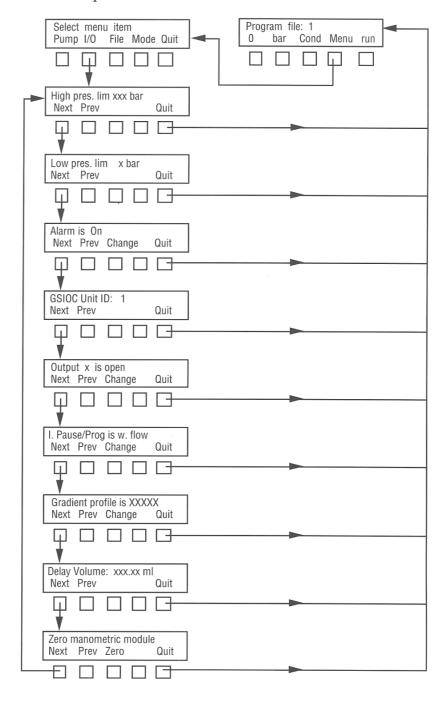
If the pressure reading from the manometric module drops below the defined limit, the pump will stop.

The sequence following a low pressure error is described page 4-10. The minimum value is 0.

Manometric Module	Maximum pressure (bar)
805	600
806	320
807	80

This menu will not appear if there is no manometric module connected to the system. This menu will not appear if there is no manometric module connected to the system.

- # The default value is 0.
- Key in the value that applies to your system and press Enter.



### **Alarm**

The alarm is a buzzer which sounds every time there is an error or an invalid setting entered or encountered. It can be programmed to be either **On** or **Off**. This function only controls the operation of the buzzer, it does not affect the operation of the pump when there is an error.

If the alarm is set to be **On**, the warning buzzer will sound every time an error is encountered. An error can be a pressure limit encountered, an invalid setting, or a pump absent. This setting can be changed from **On** to **Off** by pressing the soft key under **Change**.

# The default setting is Off.

 Select the option you want and press the Next soft key to go to the next menu.

### **GSIOC Unit ID Number**

Each pump in a multipump system has to have an identification number to distinguish it from other pumps and equipment connected to the GSIOC communications channel.

When using a 305 Master pump, you must have the identification numbers detailed in the table above for the slave pumps. The Master pump can have any number between 0 and 63, however the default value of 1 is recommended for simplicity.

- # The default value is 1.
- Key in the identity number you require for this pump and press Enter.

Pump.

Do not set the Master pump ID

number to 2, 3 or 4 as these numbers are reserved for the

Slave Pumps and Injection

### Output Contacts (Ouptut #xx is Open / Closed)

There are three relay outputs in the 305 Master pump numbered 1, 2, and 3. These outputs can be used to control other instruments. They can be programmed to open and close during a method run. They can also be opened and closed manually using soft key commands.

To change the state of an output, key in the number of the output, for example output # 2. When you press Enter, the display will show the present state of output 2, i.e. **Output # 2 open/closed**. Press the soft key **Change** to change the state of the output.

Setting the outputs manually is useful to check that output # 1 turns on the integrator for example. However, when repeating the same operation many times, it is better to program the output operations as part of a method program. In this way, the outputs will follow the same sequence each time the method program is run. Refer to programming the outputs in Section 3 of this Chapter.

By using this procedure, each of the three outputs can be manually set to be open or closed. The outputs will remain in this state until a method program is run and a change in the outputs is programmed.

- # The default state is **open.**
- Press **Next** and go to the next menu.

### Pause Prog With/Without Flow

This option runs in Program mode only. It enables the system to be controlled from an external sensor. For example, a temperature sensor can be used to pause the system if the temperature is outside a desired range. The program and flow can be paused together, or the program can be paused whilst the flow rate continues. External parameters such as temperature, pressure, or composition can be monitored to control the system.

### **Gradient Profile**

This function only operates in the program mode, and enables a selected gradient profile to be output to a recorder from the analog output of the 305. The gradient profile can be selected as being a solvent composition, %B or %C or the flow rate (Flow). The solvent composition can be useful for biochromatographic applications for plotting pH gradients and salinity (salt concentration) gradients. The flow rate can be useful for verifying detector stability.

The analog output gives 1 V full scale. The full scale represents 100%B, 100%C or 100% of the maximum possible (not programmed) flow rate of the system. This output is from connectors 12 and 13 on the I/O connector at the back of the 305. Pin 13 is the ground (0 V) connection.

Options are: Flow, %B, %C.

- # The default setting is **Flow**.
- Press the Change soft key to select your desired output and press Enter.

# **Delay Volume**

When the selected gradient profile is a solvent composition (%B for instance), the Delay Volume can be used to synchronise on the recorder the plot of the programmed profile with the plot of the detected profile (baseline drift). In this case the delay volume is generally defined, for a first approximation, as being the total volume between the mixer inlet and the detection cell inlet. The value is adjusted from experimental observation to obtain the desired synchronisation.

The 305 software divides the Delay Volume by the total flow rate to calculate the total delay time applied to the analog output Gradient Profile. The size of the Delay Volume can be specified between 0.01 mL and 999 mL.

# The default value is 0, i.e. no delay volume specified.

• Key in the required delay volume and press Enter.

### **Zero Manometric Module**

The **Zero** soft key is used to set the manometric module value to zero when there is zero pressure in the system. This ensures accurate pressure readings when the pumps are running. Before pressing the **Zero** soft key, make sure that all pumps have stopped and that the pressure has dropped to zero. Otherwise further pressure indications will be incorrect. This can be easily done by opening the prime/purge valve whilst the pumps are not operating. If the operation is successful the message **Pressure reading is zero** is displayed. If the operation is not successful, the message **Not done - check pressure readings** is displayed.

You have now completed the Input/Output parameter setup. Press **Quit** to leave this branch of the software. This will bring you back to one of the Ready-to-Run screens.

### Summary:

- Press the **Menu** soft key.
- Press the **I/O** soft key.
- Set the High Pressure Limit (Maximum value depends on Manometric Module).
- Set the Low Pressure Limit.
- Set the Alarm to be On or Off.

The Delay Volume option will only appear if %B or %C has been selected for Gradient Profile

- Set the GSIOC ID number for the 305 Master pump.
- Program outputs to be Open/Closed.
- Set pause in program to be with or without flow .
- Select the Gradient profile output to be Flow, %B, or %C.
- Specify the Delay Volume. (Maximum value 999 mL)
- Zero the Manometric Module.
- When all I/O functions have been defined as required, press the **Quit** soft key.

After entering the data about the pumping system, the pump is ready to run. The 305 Master pump can operate in three different modes. These modes are:

Flow: The 305 pump provides a constant flow rate. The pump starts when the Run key is pressed and stops when the Stop key is pressed. The flow mode is for isocratic use only.

Dispense: The 305 dispenses a specified volume. The pump starts when the Start key is pressed and stops when the specified volume has been dispensed. The dispense mode is for isocratic use only.

Program: The 305 controls a multi-pump system with up to 2 slave elution pumps and 1 slave injection pump. In this mode, the 305 Master pump can create gradients of flow rate and composition, open and close outputs to control other instruments and wait for signals from other instruments.

The operation of each mode is explained.

# **Mode Selection**

# **Running the Pump in Flow Mode**

In Flow mode, the pump provides a constant flow rate, commencing when the **Run** soft key or start input is activated. The pump stops when the **Stop** soft key or stop input is activated.

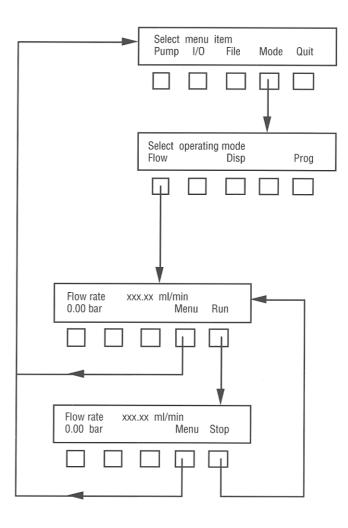
To go to the Flow mode:

- press the **Menu** soft key.
- press the **Mode** soft key.
- press the **Flow** soft key.

This brings you to the Flow mode Ready-to-Run screen. The sequence of screens and soft key command options for using the flow mode can be seen in the opposite figure.

The flow rate can be set between 0.01% and 100% of the pump head size fitted to the 305 Master pump. A flow rate value will not be accepted if it is larger than the pump head size. If the selected flow rate is incompatible with the refill time or compressibility, the message **Invalid settings** flashes after pressing run. In this case, you must reduce the refill time or the flow rate.

- Key in the flow rate in mL/min and press Enter.
- You can change the pressure units displayed at any time by pressing the soft key under the pressure units currently displayed (in this example the units displayed are in bar).



**Mode Selection** 

• The pump will start either when the **Run** soft key is pressed, or when the start input is activated.

• The pump will stop when the **Stop** soft key is pressed, or when the stop input is activated.

# **Modifying the Flow Rate**

The flow rate can be modified at any time during a run by keying in a new value. It is possible to review and change the pump and I/O setup parameters *except* the pump head size during a run. Press the **Menu** soft key and follow the setup procedures described pages 4-10 to 4-15.

# Operation of the Pressure Limits in the Flow Mode

The maximum pressure limit depends on the defined Refill Time and Compressibility. If the parameters defined in the I/O setup procedure are not compatible with the flow rate entered, the message **Invalid settings** will flash on the screen after the **Run** soft key is pressed, or the start input activated. In this case you must lower the refill time or the flow rate.

If there is a high pressure error, the pump will stop and the message **High pressure limit** will flash on the screen. The alarm will sound if it is programmed to be on. The pump will start again when the pressure drops below the defined limit. This cycle will continue indefinitely.

If there is a low pressure error, the pump will stop and the message **Low pressure limit** will flash on the screen. The alarm will sound if it is programmed to be on. The pump will stay in this condition until the **Stop** soft key is pressed.

The flow mode can be simulated in the Program mode, with the advantage of having safety error files and having the option of including timed events.

# Running the Pump in Dispense Mode

In this mode the pump can be used to deliver a specified volume beginning when the **Run** soft key or start input is activated, and finishing when the specified volume of liquid has been delivered. The parameters to enter are dispense volume and dispense flow rate or time of dispense.

To go to the dispense mode:

- press the Menu soft key.
- press the **Mode** soft key.

This brings you to the Dispense mode Ready-to Run screen. The sequence of screens and soft key command options for using the flow mode can be seen in the Figure on the next page.

Two parameters are displayed on the top line of the screen, the dispense volume, and the dispense time. A flashing cursor appears under the dispense volume setting, key in the required dispense volume and press Enter. The flashing cursor now moves to the dispense time display, key in the dispensing time and press Enter. If you wish to define the dispense volume and dispense flow rate instead, press the **Rate** soft key. The top line will then change to display the dispense volume and flow rate.

- Key in the dispense volume and press Enter.
- Key in the dispense time (or rate) and press Enter.

The limits for each of the parameters are as follows:

Maximum dispense volume:

100 x head size (mL).

Minimum dispense volume: 0.0001 x head size (mL).

Maximum dispense flow rate:  $1 \times \text{head size (mL/min)}$ .

Minimum dispense flow rate: 0.0001 x head size (mL/min).

Maximum dispense time: 9999 minutes.

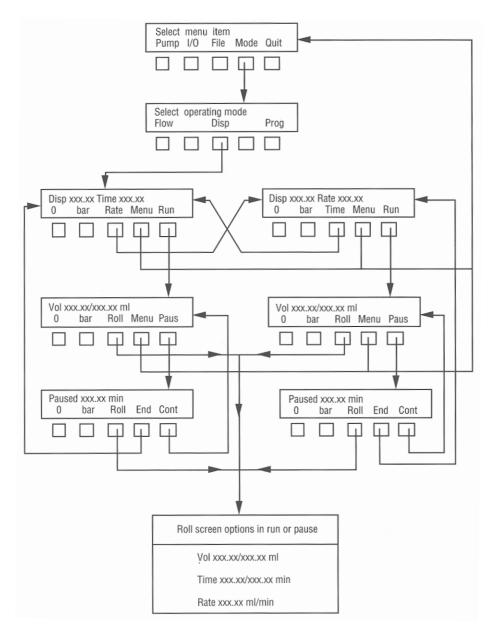
The maximum dispense flow rate depends on the Refill Time and Compressibility. If the parameters are not compatible with the dispense flow rate or volume entered, the message **Invalid settings** will flash on the screen after the **Run** soft key is pressed. In this case you must reduce the refill time or the flow rate.

If the dispense flow rate or volume is not compatible with the head size, the software will not accept the value and you must key in a new value. If the head size is changed to a size that is too small after the dispense volume and flow rate have been entered, the **Run** soft key will not appear in the menu when you return to the dispense Ready-to-Run screen.

- press the **Run** soft key to start the delivery of the liquid. The delivery will stop when the specified volume has been delivered.
  - After pressing the **Run** soft key, the display changes to give **Roll-Pause** options for the soft keys.
- press the Paus soft key to interrupt the dispense operation. The display changes to give End-Continue options.
- press the **End** soft key to terminate the delivery without dispensing any more liquid.
- press the Cont soft key to continue delivering the specified volume.
- press the **Roll** soft key to review the programmed dispense volume and the volume already dispensed.
- press the Roll soft key again to view the programmed time for the dispense and the time already elapsed.
- press the **Roll** soft key again to review the flow rate.

# Modifying the Values

It is not possible to change the dispense volume or delivery rate during a run. All of the setup parameters (including I/O parameters) *except* the head size can be reviewed and modified during a run. Press the Menu soft key and follow the setup procedures as described in pages 4-10 and 4-15.



# **Operation of the Pressure Limits**

As a safety feature of the system, it is possible to define high pressure and low pressure limits so that the operation will stop when the system pressure falls outside these limits.

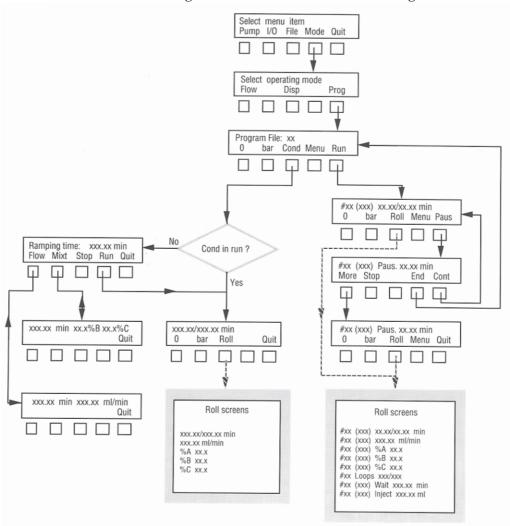
If the high pressure limit is exceeded, for whatever reason (column clogged, wrong valve closed etc.), the pump will stop and the message **High pressure limit** will flash on the screen. The alarm will sound if it is programmed to be on. The pump will start again when the pressure drops below the limit. This cycle will continue indefinitely.

If the pressure falls below the low pressure limit, the pump will stop and the message **Low pressure limit** will flash on the screen. The alarm will sound if it is programmed to be on. The pump will stay in this condition until the **End** soft key is pressed.

The dispense mode can be simulated in the Program mode with the added advantage of having Safety Files (described page 4-31), and being able to program timed events.

# Running the Pump in Program Mode

In this mode, the 305 Master pump can create both flow rate and composition gradients, program timed events, and control an injection pump. The program mode can also simulate the flow and dispense modes, with the advantage of safety error files and the ability to program timed events. The sequence of screens and soft key command options for programming the flow mode can be seen in Figure below.



At the end of a run in Program mode, the Solvent Consumption can be obtained by selecting the appropriate roll screen.

The 305 controls up to two other elution pumps and one injection pump through the GSIOC cable. Other instruments such as auto-samplers and fraction collectors can be connected to the 305 Master pump using the Input/Output contacts on the rear panel of the 305.

Before creating a method program in the 305 Master pump, it is necessary to understand how the method programs are stored in memory.

# **Memory Layout**

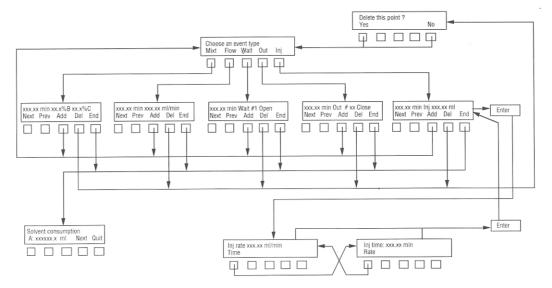
There are 14 files, numbered 1 to 14. Each file can store 1 program. Files 1 to 10 are user files, available for method programs. Files 11 to 14 are reserved for safety/error programs.

A file contains timed events. A timed event is a flow rate, a solvent composition, the operation of an input or output or the operation of an injection pump. One file stores a maximum of 25 timed events which can make up the method program.

The Safety Files, 11 to 14, do not contain pre-stored programs you can write each Safety File according to your own requirements, for example, to output a control signal to an external device. Write each safety program in the same way as a method program. This allows you to program the sequence of events that will happen when an error occurs. If an error occurs during a run, the method program stops and the relevant safety program starts. If no safety program has been written, the default operation in the event of an error is described in each relevant Safety File description. See pages 4-31 to 4-33 for the exact operation of Safety Files.

# File Selection

One complete method is stored in a file. In order to read/edit/write a method, you have to go to a file. The sequence of screens and soft key command options for programming a file can be seen in the figure below.



To go to a file:

- press the **Menu** soft key.
- press the **File** soft key. This brings you to the **Select file** menu.

Key in the number of the file, for example 1, and press Enter.

There are five soft key options available:

- Directory
- Copy
- Delete
- Edit/New
- Quit

# **Directory**

Press this key to go to each stored file, e.g. File 1, File 3 etc. This displays all of the files where method programs are stored. If no programs are stored, as in the case of a new pump, **Select file #**—will be displayed. Key in the file number that you want to use and press Enter.

# Copy

Press this key to make a copy of a complete file. This is useful if you want to make a small modification to an existing program and keep a copy of the original program. After pressing **copy**, key in the file number where the copy will be stored and press Enter. The software indicates if the file where you want to store the copy is empty (**New**) or if there is a program already stored there (**Exists**). You have the choice of completing the copy procedure, **Yes**, or ending the procedure without making a copy, **No**. The copy of the program can then be modified without destroying the original.

### **Delete**

Press this key to delete a complete file. The deleted file is the file currently shown on the upper line of the display. After pressing **Del**, **Delete file # xx ?** is displayed. This is a safeguard against accidental erasure. There are two options: **Yes** deletes the file, **No** brings you back to the original menu.

### **Edit/New**

If there is no program stored in the file number that is displayed on the top line, the display will be **New**, if there is a program stored in the file, the display will be **Edit**. Both of these options will bring you to the first step in writing/editing a method program.

### Quit

Pressing **Quit** brings you back to the Ready-to-Run Screens.

Pressing the **Edit/New** key brings you to the first menu in the programming sequence, **Number of loops**. You are now ready to write a method program.

# **Programming a Method**

A complete method program is written by programming flow rates, solvent compositions and operation of the outputs. The method program run time starts at time 0.00, i.e. when the start key is pressed, and ends at the time of the last timed event. For example, if you program the last event at time 20.00, then the run time is 20 minutes.

You must program every event for your method, starting at time 0.00. For example, at time 0.00 you must program the flow rate and composition. If you do not program anything at 0.00 minutes, the pump will assume the current flow rate and composition. For a pump which is stopped, the software will assume a flow rate of 0 mL/min and a composition of 100% A, 0% B, and 0% C. If the pump is running, it will assume the current flow rate and composition for time 0.00. It will then operate on a gradient between these values and the first flow rate in your program and the first solvent composition in your program.

# Menu: Number of Loops

The number of loops is the number of times that the program will repeat itself before stopping. The minimum value is 1 and the maximum value is 999.

- # The default value is 1.
- Key in the number of loops and press Enter.

This brings you to the When finished, use menu.

# Menu: When Finished, Use

At the end of a program, you can link the current file to any of the 14 files. If you do not want to link to any other file, press the soft key below **None** on the display.

If you link to the current file, i.e. link File 3 to File 3, the program will continue to run until the **Pause** key is pressed. If both looping and linking are programmed, the software will complete the programmed number of loops, then link to the new file.

If you link files together which have different values for the setup parameters, the pump will not start. After pressing **Run** the display will give you the message **Note!** Setup has changed since file creation. Pressing **Ok** gives you the choice of which setup parameters to keep by asking the question **Keep original setup?** Yes - No. If you choose Yes, the values which are stored in the first file in the sequence of files will be loaded into all of the files which are linked together. If you choose No, the values which are currently stored in the pumps setup parameter memory will be loaded into all of the files which are linked together.

# The default value is None.

• Key in the number of the file you want to link to and press Enter, or press the None soft key.

This brings you to the Choose an event type screen.

# Menu: Choose an Event Type

There are five different types of timed events. You choose one of the different events by pressing the soft key below it. The five timed event types are:

- Mixture Program the composition, %A, %B and %C.
- Flowrate
  Program the flow rate, mL/minute.
- Wait Wait for an input.
- Out Activate an output.
- Inject Activate the injection pump.

At the end of each operation, the solvent consumption will be displayed.

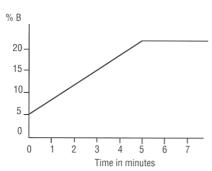
### **Mixt**

Set the composition of the solvents, e.g. 70 % solvent A, 20 % solvent B and 10 % solvent C. There is a maximum of three pieces of data for this menu:

- Time at which composition occurs
- % of solvent B
- % of solvent C.

One complete method program is stored in a file. The values for the six parameters; Refill Time, Compressibility, Pump Head Size, Inlet Pressure, High Pressure Limit and Low Pressure Limit for that method program are also stored in the same file. If you link two or more files together, you must ensure that they all have the same values for these parameters.

In the case of linking an error file which has different setup parameters to the method file, the parameters for the method file automatically replace the values which were written in the error file.



There will be a linear gradient between two solvent composition points. With the example given, the %B will increase linearly from 5% to 20% in 5 minutes. If you change the gradient point during a run, the new gradient will be between the next programmed gradient value and the value which existed at the instant the gradient was modified.

If you do not program a composition at 0.00 min., the pump assumes the current composition. For a stopped pump, this will be 100% A. There will then be a gradient between 100% A and the first programmed solvent composition point. Programming the example above will give a composition gradient as shown in Figure.

The percentage of solvent A is calculated as: % solvent A = 100 - % B - %C.

The example opposite is for a binary gradient. At 0 minutes, there is 95% solvent A and 5% solvent B. At 5 minutes, there is 80% solvent A and 20% solvent B.

Example: 
$$0.00 \text{ min } 5\%B$$
 (%A = 95%)  
5.00 min 20%B (%A = 80%)

To program the example:

- press the **Mixt** soft key.
- key in the time for the first composition point, 0.00, and press Enter.
- key in the value for the %B, 5, and press Enter.
- press the **Add** soft key to add a second timed event to the program.
- key in the time for the second composition point, 5.00, and press Enter.
- key in the value for the %B, 20, and press Enter.

Composition parameters can be set down to 0.1% increments. If you key in a value which is too high (sum A+B+C>100%) the last entry will not be accepted by the software and you must key in the last value again, or correct your composition specification.

### **Flow**

In this menu, you can set the flow rate, for example, 2 mL/min. There are two parameters to specify for this menu.

- Time at which flow rate occurs.
- Flow rate in mL/min.

Example: 0.00 min 0.00 mL/min 5.00 min 3.0 mL/min

To program this example:

- press the **Add** soft key.
- press the **Flow** soft key.
- key in the time for the first flow rate, 0.00, and press Enter.

Operation 4

- key in the value for the flow rate, 0, and press Enter.
- press the Add soft key to add a second timed event.
- key in the time for the second flow rate, 5, and press Enter.
- key in the second flow rate, 3, and press Enter.

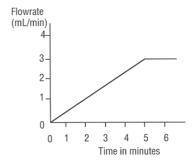
If you try to enter a flow rate value which is too high for the pump head, the entry is refused by the software. The maximum value accepted in a gradient system is the value of the smallest pump head size in the system.

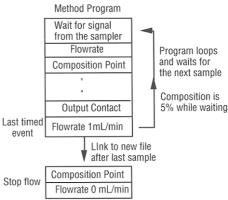
If you do not program a flow rate at 0.00 min., the software will assume the current flow rate for the time 0.00 min., i.e. for a stopped pump, a flow rate of 0 mL/min. There will then be a gradient between 0 mL/min. and the first programmed flow rate. With this example the flow rate will be as shown in the opposite figure.

To stop the flow of solvent at the end of a run, you must program a flow rate of 0 mL/min. Otherwise, the pump will continue to run with the last programmed flowrate, even after the last timed event. If there is more than one pump in the system, you must program a composition point containing all of the pumps followed by a flow rate of 0 mL/min.

If the system is set up to do more than one sample, the initial composition for the method can be maintained between method runs by programming this composition at the end of the program, for example 5% B. At the end of all the samples, link to another file to completely stop the flow of liquid. See the opposite figure.

There will be a linear gradient between any two programmed flow rates. With the above example, the flow rate will linearly increase from 0.00 mL/min to 3.0 mL/min in 5.00 minutes.





Stopping the flow after repeated samples

### Wait

In this menu you can make the program wait until Input # 1 is activated. This is used to stop the program until another piece of equipment is ready, for example an auto-sampler or a fraction collector. There are two pieces of data for this menu.

- Time at which the pump will wait for an input
- Waiting for an open contact or a closed contact

Example 4: 2.00 min Wait #1 Closed.

To program this example:

- press the Add soft key.
- press the Wait soft key.
- key in the time for the wait to begin, 2.00, and press Enter.
- press the **close** soft key.

The program waits at time 2.00 minutes until input #1 is closed. If input #1 is already closed at time 2.00 mins, the program will continue. If input #1 is not closed, the program will wait. During the time that the program is waiting, the display will show the total run time and the time that it has been waiting.

If a pump is waiting for an input, pressing **Cancel** will simulate an input and the program will continue.

### Out

In this menu, you can program each of the outputs in the system to open or close. The out-puts are numbered 1, 2 and 3 and are used to send signals to other equipment in the system, for example an auto sampler or a chart recorder. By having the output operations as part of the method program, the same sequence of contacts repeats every time the method program is run.

There are three pieces of data for this menu.

- Time at which output is operated
- Output number
- Output opened, closed or pulsed

Each of the outputs can be made to open, close or pulse. Pulse means that the output will change its current state for 0.6 of a second.

Example: Screen 1 3.00 min Output 2 Screen 2 Open Close Pulse

To program this example:

- press the Add soft key.
- press the **Out** soft key.
- key in the time for the output to operate, 3.00, and press Enter.
- key in the output number, 2, and press Enter.

The display will change to **Open**, **Close** or **Pulse**.

• press the **Close** soft key.

# Inj

In this menu you can injection a sample using the inject pump. There are three pieces of data for this menu.

- Time at which the injection pump is started
- Injection volume
- Flowrate for the injection

Example: Screen 1 2.00 min Inj 6.00 mL Screen 2 Inj rate 0.5 mL/min

To program this example:

- press the **Add** soft key.
- press the **Inj** soft key.
- key in the time at which the injection will start, 2.00, and press Enter.
- key in the injection volume, 6.00, and press Enter.
- key in the value for the injection rate, 0.5, and press Enter.

The injection rate parameter can be changed to injection time by pressing the **Time** soft key. There is a limit of one injection point for each method program. The maximum injection volume is the pump head volume multiplied by 100. If you program a second injection point, it will write over the existing injection point.

Preparative injection: The 305 can be used in the Program mode for repetitive injection of the same sample.

At the selected time, the Master 305 starts the injection pump and automatically lowers the elution rate in order to keep the total flow rate, and hence the pressure, constant. The following relationship is applied:

 $F'_{e} = F_{e} - F_{i}$  where

F'<sub>e</sub>: the total elution flow rate during injection

F<sub>e</sub>: the total elution flow rate before injection

 $F_i$ : the injection flow rate

A stop-flow injection, generally desired, is then obtained by selecting  $Fi \ge Fe$ .

# Reading/Writing/Editing Timed Events

The five different types of timed event have been explained above. At the end of each event, the menu gives you five options. These options are explained below.

**Next:** displays the next timed event in the sequence. If there are no more events, it displays **End of file**.

**Prev**: displays the previous timed event in the sequence. If there are no more previous events, it displays **Beginning of file**.

Add: brings you to the Choose an event type menu to allow you to add another event to the present program.

**Del**: deletes one timed event in the program. As a safeguard it asks you, **Delete this point**? Press **Yes** to delete the event or **No** to return to the original menu.

End: leaves the software for programming timed events. The pump displays Solvent consumption in mL for pump A for one complete run of the method program.

Pressing the **Next** soft key displays in turn the consumption for the other pumps in the system. The minimum consumption is < 0.1 mL. The maximum is > 999 mL.

Press Quit to go to the Ready-to-Run Screen. You have now finished creating a program method file. Before running a method program, you should program each of the four safety/error files.

# **Programming the Safety Files**

The Safety Files are a useful safety feature which can be used to control the pumping system if a malfunction is detected. It is possible to detect if the

system pressure is too high or too low, if there has been a power failure, or if an external sensor has detected an illegal value (temperature too high, for example). In this case, the method program is stopped, and the relevant Safety File is run. The File number and function of each File is listed in this table.

File	Name	Function
11	Low	File runs after a low pressure error
12	High	File runs after a high pressure error
13	Input	File runs if input # 2 is activated
14	Power	File runs after a power failure

These programs are created in exactly the same way as a method program. To program the low pressure safety file, select file 11 and enter the sequence of flow rates and input/output operations that you require when there is a low pressure error. Similarly, enter a program for the high pressure safety file, Input# 2 safety file and the power failure safety file. The Input # 2 contact can be connected to an external safety device such as a temperature or pressure measurement system.

An example of simple programs for the high and low pressure safety files are given in program lists 2 and 3 at the end of this section.

# **Low Pressure Safety File (File 11)**

If the pressure goes below the low pressure limit the sequence of events is as follows:

- If the low pressure error file (11) contains no program, the method program will stop and can not resume until the operator presses the **Pause** soft key followed by **End**.
- If the low pressure error file contains a program, the method program will link to this file and will run it. After file 11 is finished, if there is a link to another file, this new file will start to run. If there is another low pressure error during file 11, the pump will stop and can not resume until the operator presses the **Pause** soft key followed by **End**.

For an example of a Low Pressure safety file refer to page 4-39, Program List 2.

# **High Pressure Safety File (File 12)**

If the pressure goes above the high pressure limit, the sequence of events is as follows:

- If the high pressure safety file (12) contains no program, the pumps stop at the instant the pressure rises above the limit. If the pressure drops below the limit again, the pump will restart. This cycle will continue indefinitely.
- If the high pressure safety file contains a program, the pumps stop and wait for the pressure to drop below the high pressure limit contained in File 12. When the pressure is below this limit, file 12 will begin to run. After file 12 is finished, if there is a link to another file, this new file will start to run. If there is another high pressure error during file 12, the pump will stop until the pressure drops below the limit and will then restart. This cycle will continue indefinitely.

For an example of a High Pressure safety file, refer to page 4-39, Program List 3.

# Input # 2 Safety File (File 13)

When input # 2 is activated, it causes the following sequence of events:

- If there is no program in File 13, activating input # 2 will have no effect and the method program will continue.
- If there is a program stored in File 13, when input # 2 is activated, File 13 will start. File 13 will start to run even if the system was not previously running. If there is a link to another file at the end of the program, the linked file will start to run.

# **Power Failure Safety File (File 14)**

The power failure safety file will only operate if there is a power failure while a method program is running. The sequence of events after a power failure is as follows:

• If there is no program stored in File 14: after the power is restored, the program will go back to the start of the method file and wait for a start input.

If there is more than one file in the method, i.e. one file is linked to another, the program will go back to the beginning of the first file in the method.

• If there is a program stored in File 14: after the power is restored, the program in file 14 will be run. If there is a link to another program at the end of file 14, the linked file will start to run. If the alarm is on, it will sound.

If there is a power failure in the Flow or Dispense modes, it has the same effect as if the pump was turned off and turned on again. The screen presented will be the Ready-to-Run Screen of the last used mode.

# **Running a Method Program**

At the end of programming the method and error files, the software returns to a Ready-to-Run Screen. The top left corner will indicate which Ready-to-Run screen you are in. To go to the Program mode Ready-to-Run Screen:

- press Menu.
- press Mode.
- press Prog.

The pressure can be displayed in 3 units, bar, MPa or kpsi. To change from one unit to another, press the soft key directly below bar. The units will change to MPa. Pressing the soft key again will change the units to kpsi. Pressing the soft key again will change the units back to bar.

You can go to any file by keying in a new number and pressing Enter. The Ready-to-Run Screen displays the present file number, the pressure, and gives you 3 soft key options, **Cond**, **Menu** and **Run**.

# **Cond Soft Key**

It brings you to the part of the software which is used to condition the column. You program a ramp time, the flow rate and gradient. The initial conditions are the existing conditions for the pump, i.e. for a pump which is not running 0 mL/min and 100% solvent A. The flow rate and composition programmed should be the flow rate and composition that is required at the start of the method program that you are going to use. The solvent flow rate and composition are then ramped from 0 mL/min and 100% A to the initial composition and flow rate for your method program.

- press Cond.
- enter the ramping time and press Enter.
- press Flow.
- enter the flow rate for the end of the ramp time.
- enter the % B for the end of the ramp time (if pump B is present).
- enter the % C for the end of the ramp time (if pump C is present).

Press **Run** to start the conditioning of the column. The pump will ramp to the flow rate and solvent composition that is programmed in the time programmed. The values for time, flow rate and solvent composition can be changed during the conditioning. At the end of the ramp time, the flow rate and solvent composition will remain constant. Note that you can leave the conditioning of the column and create a program without interfering with the conditioning of the column.

After pressing **Run**, the display changes to give you the choice of **Roll**, **Stop** or **Quit**.

Pressing Roll displays in turn the time for the ramp, the flow rate, the proportion of solvent A, B and C in turn. The proportion of solvents B and C will not appear if they do not exist.

This part of the software is also used at the end of a method program to clean the column. The initial conditions will be the final flow rate and composition which existed at the end of your method program. You can ramp the flow rate down to 0 mL/min and create a solvent gradient that will completely clean the column.

The **Quit** soft key brings you to the **Select Menu Item** menu. This gives you access to the pump setup parameters, the pump I/O parameters and the method file. Press this key to verify or change the value of any parameters in these sections.

# **Run Soft Key**

It starts the program running. When this key is pressed, the display changes. The figure in the top left hand corner is the file number. The number in parentheses is the loop number. If there is more than one loop in your program, the present loop number will be displayed here. The first time displayed is the actual running time. The second time displayed is the total running time for the method program.

When a program is written, the current values for the six parameters Pump Head Size, Compressibility, Refill time, Inlet Pressure, High Pressure Limit and Low Pressure Limit are stored as part of the method program. When the file is being run again, if any of these parameters has been changed, the following message will appear after **Run** has been pressed, **Note! Setup has changed since file creation Ok**. Press **Ok**. You can choose to keep the setup parameters which were stored with the method program or you can choose to keep the setup parameters which exist currently. Press **Yes** to keep the setup parameters which existed when the file was created. Press **No** to keep the current setup parameters. After pressing one of these keys the pump will automatically start running the file.

If a program has a link to a file which has nothing stored in it, the program will not start when **Run** is pressed. The message **Link file does not exist** is displayed and the alarm will sound.

After pressing Run, the bottom line of the display changes to **Roll/Menu/Pause**.

- Pressing **Roll** will display in turn:
  - current file/loop number/running time and program time
  - current file/loop number/current flow rate
  - the % of solvent A
  - the % of solvent B
  - the % of solvent C
  - the time that the 305 is waiting on an input
  - the programmed injection volume

Pressing **Roll** once more brings you back to the Run screen.

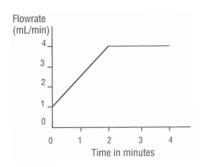
- The **Menu** soft key has the same function as before.
- Press **Pause** to freeze the program. The pumps will continue to run, keeping a constant flow rate and composition. The pumps will stay in this state until another key is pressed. When **Pause** is pressed the screen changes to **Stop/End/Cont**.
  - **Stop** will cause the pumps to stop. The method file remains in the Pause state.
  - **End** will cause the termination of the method file without stopping the pumps. The pumps will continue with the flow rate that existed when the pumps were paused. The screen returns to the Program mode Ready-to-Run Screen.
  - Cont will cause the program to continue from the point where it was paused.

The input Start/Stop has the same effect as the **End** soft key in the Program mode.

To terminate a program completely and stop the flow, you must press **Stop** followed by **End**. If a method program has terminated and the pumping system is still running, press **Cond** followed by **Stop**.

During a method run, the setup parameters and the method program can be modified. Modify a parameter in the same way that you program it. If the flow rate or composition is modified during a run, the new value will take effect from the instant it is programmed. If a flow rate is modified while the pump is running, the new flow gradient will be between the next programmed point and the flow rate which existed at the instant the modification was made.

After a method program has finished, the display returns to the Ready-to-Run screen.



# **Programming Examples**

Two example programs are given below. Before running these example programs, ensure that the hydraulic circuit is properly connected.

# **Example 1**

In the example given in List 1, the flow rate will start at 1 mL/min and increase to 4 mL/min after 2 minutes. The composition will be constant at 100% solvent A. An output will close at 0.00 minutes to start the integrator. The method will only operate once and will not link to another file when it is finished. Enter this program following the steps given Program list 1.

Note that it is necessary to program a flow rate of 0 mL/min at the end of the program to stop the flow, the flow does not automatically stop when the program is finished. It will continue with the last programmed flow rate. To stop the flow at 4.00 minutes you must program a flow rate of 4 mL/min at 3.99 minutes and 0.00 mL/min. at 4.00 minutes.

# **Note on Programming Sheet**

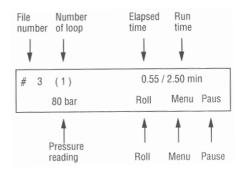
Appendix G contains a programming sheet which can be copied. An example of how to use the programming sheet is also given. The sequence you should follow is:

- Fill in the File Number and name
- Fill in the SETUP parameters for your system
- Draw the flow rate and composition gradients on the graphs provided, remembering to mark the axes
- Fill in the table for Input/Output operations
- Write the programming steps. Finish one section before going on to the next, i.e. program all of the flow rate time points before going on to the composition time points. The 305 software will arrange all of the time points in the correct sequence.

After writing the method program, it is necessary to write the programs for the safety/error files.

Simple programs are given in list two and three for the Low pressure error file and the High pressure error file. In both programs, the flow rate is programmed to be 0 mL/min. This is a simple example of an error file.

Enter these two error programs before running Example 1. After entering the two pressure error programs, the system is ready to run. Press Run. The display will look as opposite. Press Roll to display in turn the flow rate, %A, %B, %C, return to the run screen. When the method program is finished, the Ready-to-Run screen is displayed.



### **Program List 1**

Key to press	Notes
Menu	
File	
1/ENTER	Select file 1
New/Edit	Go to file
1/ ENTER	1 Loop
None	No link at end of method
Out	Program the output
0/ ENTER	At 0.00 minutes
1/ENTER	Output # 1
Close	Close output # 1
Add	Add the next timed event
Flow	Enter the flow rate
0/ENTER	At 0.00 minutes
1/ENTER	Flowrate = 1mL/min.
Add	Add the next timed event.
Flow	Enter the flow rate
2/ENTER	At 2.00 minutes.
4/ENTER	Flowrate = 4mL/min
End	Finish writing method
	Solvent A consumption: 5mL
Quit	Go to Ready-to-Run screen

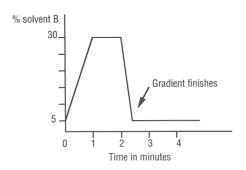
### **Program List 2**

Key to press	Notes
Menu	
File	
11/ENTER	Low pressure safety file
New/Edit	Go to file
1/ENTER	1 Loop
None	No link to other files
Flow/ENTER	Enter the flow rate
0/ENTER	At 0.00 minutes
0/ENTER	Flow rate = 0mL/min
End	Finish writing safety file
Quit	

### **Program List 3**

Key to press	Notes
Menu	
File	
12/ENTER	High pressure safety file
New/Edit	Go to file
1/ENTER	1 Loop
None	No link to other files
Flow/ENTER	Enter the flow rate
0/ENTER	At 0.00 minutes
0/ENTER	Flow rate = 0 mL/min
End	Finish writing safety file
Quit	

# Example 2

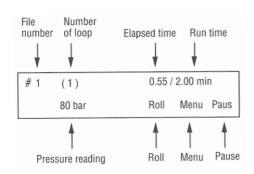


The operation of the method in Example 2 will be as follows.

File # 4 contains the method program. The flow rate remains constant throughout the program. At 0.00 minutes the mobile phase will have a constant flow rate of 3 mL/min and a composition of 95% A, 5% B and 0% C. The composition will change to 70% A, 30% B and 0% C in a time of 1 minute. The composition will stay constant for 1 minute and then return to 95% A, 5% B and 0% C in a time of 0.5 minutes. The composition will remain at 5% B until 4.00 minutes. The program will loop and repeat the same method 3 times and will then link to File 5.

File 5 will contain a program to set the flow rate to 0 mL/min. The Low pressure and High pressure safety files that were used in the first example can also be used for this example. It is not necessary to reprogram them.

Enter this program by following the steps given in Program list 4 and 5.



The system is now ready to run. Press **Run**. The display will look as opposite. Press Roll to see the % of A and B changing. After 2.5 minutes, the program will loop to start the method a second time. The Loop Number will change to 2.

At the end of three loops, the program will link to File 5. File # 5 is displayed. At the end of File # 5 the Display will go back to File # 4. Note that in this program, File # 5 only lasts for a short time.

To improve on this basic program, add a wait for input # 1 timed event at 0.00 min. This means that the gradient will not start until it receives a signal from another piece of equipment, for example a sampler. Only after the sample has been injected will the gradient start. Program a wait Input # 1 is closed. To start the program with the wait programmed, connect Pin 1 to Pin 4 on the Input/Output connector or press Cancel.

### **Program List 4**

Key to press Notes

# Menu File 3/ENTER File # 3 New/Edit 3/ENTER 3 Loops 4/ENTER Link to file # 4 when finished Flow Enter the flowrate

Flow Enter the flowrate

0/ENTER At 0.00 minutes

3/ENTER Flowrate = 3mL/min

Add Add the next timed event

Mixt Enter the composition

0/ENTER At 0.00 minutes

5/ENTER 5% solvent B

0/ENTER 0% solvent C

Add Add the next timed event
Mixt Enter the composition
1/ENTER At 1.00 minutes
30/ENTER 30 % solvent B

30/ENTER 30 % solvent B
0/ENTER 0 % solvent C

Add Add the next timed event.

Mixt Enter the composition

2/ENTER At 2.00 minutes
30/ENTER 30 % solvent B
0/ENTER 0 % solvent C
Add Add the next timed event

Mixt Enter the composition
2.5/ENTER At 2.50 minutes
5/ENTER 5 % solvent B
0/ENTER 0 % solvent C

Add Add the text timed event
Mixt Enter the composition
4.0/ENTER At 4.00 minutes

5/ENTER 5 % calculates

5/ENTER 5 % solvent B 0/ENTER 0 % solvent C

End Finish writing the method

program

Quit

### **Program List 5**

Key to press	Notes
Menu	
File	
4/ENTER	File # 4
New/Edit	
1/ENTER	1 Loop
None	No link at end of file
Mixt	Enter the composition
0/ENTER	At 0.00 minutes
0/ENTER	0 % solvent B
0/ENTER	0% solvent C
Add	Add the next timed event
Flow	Enter the flowrate
0/ENTER	At 0.00 minutes
0/ENTER	0mL/min
End	Finish writing the method
Quit	Go back to the Ready-to-Run screen

Operation

4

# Maintenance and Troubleshooting 5

The 305 pump has been designed to require a minimum level of care and maintenance. In practice, maintenance is limited to cleaning and replacing parts of the pump head.

### **Pump Head Maintenance**

The check valves and filters can be cleaned. Piston seals, check valves, piston assemblies, anti-extrusion gaskets and return springs should be replaced on a regular basis. A maintenance kit is available for each model of pump head. For details about maintenance kits and procedures, see the User's Guide for your pump head.

The use of equipment for continuous, unattended operation is becoming more and more important. For this reason, the following table gives an indication of replacement periods of maintenance parts according to the type of use, intensive, regular or occasional. The data in the table below assumes that the pump is working at half of its maximum flow rate and pressure. The nature of the liquid and the pump head model have only a small influence on these figures.

The time between each maintenance operation can be viewed by using the **Info** soft key in the **Pump** menu.

Time table for checking replacement parts according to the type of use.

Parts/Use	Intensive (168 h/week)	Regular (40 h/week)	Occasional (10h/week)
Piston seal	2 - 3 months	6 - 9 months	1 year
Set of check valves	3 - 6 months	1 year	2 years
Piston assembly	6 - 12 months	2 - 3 years	5 years
Anti-extrusion gasket	6 - 12 months	2 - 3 years	5 years
Return spring	1 year	2 - 3 years	5 years

# **Troubleshooting**

# **Electrical Problems**

Problem	Possible cause	Solution
Pump does not operate and power indicator does not light.	Power cord unplugged. Fuse blown. Incorrect voltage setting.	Check for power. See 'Electrical installation' in Chapter 3.
Slave pump does not operate.	GSIOC cable not connected or incorrectly connected. Incorrect GSIOC identity number.	Check GSIOC cable is connected correctly to the socket on the 305.  Check GSIOC identity number is set correctly.
Message 'Pump X missing'	Incorrect GSIOC identity number set in pump X.	Set the GSIOC identity correctly. Refer to Chapter 3.
Pressure reading does not appear on display, or	Output signal from manometric module or adapter not connected.	Check the pressure signal output is properly connected.
High pressure limit menu does not appear.		The pressure signal must be connected directly to the Master pump, not to a slave.
Invalid settings flashing	Refill time is too long for the flow rate programmed.	Lower the Refill time and flow rate.
Pump does not stop at end of program.	Not programmed.	To stop the flow at the end of a method program, you must program a flow rate of 0 mL/min. With a gradient system, you must program a composition point and then a flow rate of 0 mL/min.

# **Hydraulic problems**

Problem	Possible cause	Solution
Leaks from the hole at the bottom of the pump head.	Defective piston seal.	Replace piston seal. Refer to User's Guide for the pump head.
Low flow rate.	Leaks.	Check for leaks.
	Plugged inlet filter.	Clean or replace the inlet filter. Refer to User's Guide for the pump head.
	Defective check valve.	Clean or replace the check valve. Refer to User's Guide for the pump head.
	Pump head not mounted properly.	Check that the pump head is properly mounted.
Air bubbles appear in both and outlet tubing.	Loose connection of inlet tubing.	Tighten the connection (but do inlet not overtighten).
	Worn flange of inlet tubing.	Replace the inlet tubing.
	Inlet filter partly clogged.	Clean or replace the inlet filter.
	Refill time is too long for the solvent.	Decrease the refill time.
Air bubbles appear only in outlet tubing.	Loose connection of outlet tubing.	Tighten the connection (but do not overtighten).

# Accessory Parts List Appendix A

Parts lists for the 305 Pump, consisting of Standard Accessories and Additional Accessories.

# **Standard Accessory Parts List**

Reference	Qty	Description
3645388	1	SC type pump head clamp
638314512	1	Terminal block connector, 14 pin
36610101	1	Double-ended wrench, 1/4"- 5/16"
6730204007	4	Fuses 2.0 Amp type "T" slow blow (5 x 20 mm) for 100-120 V
7080316105	1	Power cord for 100-120 V
6730104006	4	Fuses 1.0 Amp type "T" slow blow (5 x 20 mm) for 220-240 V
7080316106	1	Power cord for 220-240 V
LT80152	1	Model 305 User's Guide

Only one power cord and one set of fuses are supplied. The standard accessory package that you receive will contain suitable parts for your voltage.

# **Additional Accessory Parts List**

Reference	Description
36078143	GSIOC cable
709910406	Four-wire electrical cable, 1.7 m length, for I/O connections.*
03434939	Mast clamp
2105703	Hex Mast, 3/4" x 16 mm

<sup>\*</sup> Several units may be required according to the system configuration.

# **GSIOC Control**

# **Appendix B**

This chapter explains how to control the 305 from a computer using Gilson HPLC system controller software.

#### **GSIOC Features**

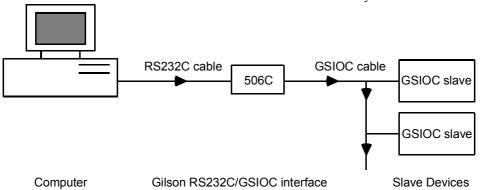
GSIOC stands for Gilson Serial Input Output Channel. This communications channel links all of the Gilson modules in a system together. The system controller controls all of the modules in a system by sending GSIOC commands to the slave modules, for example pumps or detectors. Each device connected to the GSIOC channel is distinguished by a GSIOC identity number between 0 and 63. The GSIOC identity number is set by switches inside each module or by the module's software. The controller communicates with one slave device at a time. The hardware and software requirements to control a module from a computer using the GSIOC are as follows:

- A PC running under Windows® 95, 98, NT, or higher.
- A Gilson interface module, 506C.
- A Gilson HPLC system controller software package.

To get started you must:

- Install the Gilson HPLC system controller software on the computer.
- Connect the computer to the Gilson interface using the cable provided with the interface.
- Connect the output from the interface to the Gilson module using a GSIOC cable.
- Start the Gilson HPLC system controller software.

For more details consult the documentation that accompanies the Gilson software.



#### **GSIOC Commands**

The GSIOC commands can be used to control Gilson modules directly from a computer or from a Gilson HPLC system controller software package. The use of the GSIOC commands is completely detailed in the 305 technical manual.

#### 305 Commands

Specific 305 GSIOC commands consider the pump as a set of peripheral devices which can be either processed by the internal software, or by the GSIOC bus. There are two device types: input devices and output devices.

- 305 input devices are: the Keypad, Contact Inputs, and the Pressure Input.
- 305 output devices are: the Upper Line of Display, the Lower Line of Display, and Contact Outputs.

All commands are detailed below with their type, mode and function. Immediate commands have their response format described. Buffered command parameters are documented. Comments are added where necessary.

Command	Туре	Function
%	- 1	Request Pump Identification
\$	- 1	Master Reset
I	В	Write Contact Inputs
I	1	Read Contact Inputs
i	В	Read Contact Input Buffers
J	В	Write Contact Outputs
J	1	Read Contact Outputs
j	1	Read Contact Output Buffers
K	В	Remote Keystrokes
K	- 1	Read Key Entry
L	1	Request Manometric Module
		Identification
Р	В	Pulse Contact Outputs
Q	В	Enter Pressure Value
Q	- 1	Read Pressure Value
W	В	Write Display
W	- 1	Read Display
W	I	Read Display Buffer

Immediate %	Request Module Identification Response format: "305 Va.bc" where Va.bc is the software version.
Immediate \$	Master Reset Response format: \$ is echoed.
Buffered I	Write Contact Inputs Syntax: labcd where a is the START/STOP input, b the PAUSE input, c the IN#1 input, and d the IN#2 input.  Parameters: "C" for closed "D" for open, "X" for state unchanged, "-" for reconnected to internal software Command example: "I-XCD" to reconnect START/STOP to the internal software, leave PAUSE as it is, close IN#1, and open IN#2.

#### Immediate I Read Contact Inputs Response format: "abcd" where a is the START/STOP input, b is the PAUSE input, c is the IN#1 input and d is the IN#2 input. For each input: "C" if closed and disconnected from software, "c" if closed and disconnect from software, "D" if open and connected to software, "d" if open and disconnected from software. Response example: "DDCD" for IN#1 shorted to ground and all inputs connected to the internal software. Immediate i Read Contact Inputs Buffers Response format: "abcd" where a is the START/STOP input, b is the PAUSE input, c is the IN#1 input and d is the IN#2 input. For each input: "C" if closed and disconnected from software, "c" if closed and disconnected from software, "D" if open and connected to software, "d" if open and disconnected from software. Response example: "DcCD" for PAUSE activated and disconnected from the software, and other inputs connected to the software with IN#1 shorted to ground. **Buffered J** Write Contact Outputs Syntax: Jabcde where a is the OUT #1 relay, b is the OUT #2 relay, c is the OUT #3 relay, d is the HIGH pressure limit relay, e is the LOW pressure limit relay. Parameters: "C" for connected to ground "D" for open "P" for pulse (output state reversed, see comment) "X" for state unchanged "-" for reconnected to internal software. Command example: "JPDXXX" to pulse OUT #1 and open OUT #2. Comment: the "P" parameter is valid only for programmable outputs abc. The pulse duration is defined by the P command. At the power ON, the default pulse duration is 600 milliseconds. Immediate J Read Contact Output Response format: "abcde" where a is the OUT #1 relay, b is the OUT #2 relay, c is the OUT #3 relay. d is the HIGH pressure limit output, e is the LOW pressure limit output. For each input: "C" if closed and connected to software, "c" if closed and disconnected from software, "D" if open and connected to software,

"d" if open and disconnected from software.

the internal software.

Response example: "DDDDD" for all relays open and outputs connected to

#### Immediate j

Read Contact Output Buffers

Response format: "abcde" where

a is the OUT #1 buffer,

b is the OUT #2 buffer,

c is the OUT #3 buffer,

d is the HIGH pressure limit buffer,

e is the LOW pressure limit buffer.

#### For each input:

"C" if closed and connected to software,

"c" if closed and disconnected from software,

"D" if open and connected to software.

"d" if open and disconnected from software.

Response example: "DDDDD" for all relays open and outputs connected to the internal software.

#### **Buffered K**

Input Remote Keystrokes

Syntax: Kcodes, up to 30 codes per command.

Parameters: ASCII codes, as follows:

Command example: "Kdea 1 Ee" to start

flowing 1 mL/min.

Key Name	Dec	Hex	Code
SOFT#1	97	61	"a"
SOFT#2	98	62	"b"
SOFT#3	99	63	"c"
SOFT #4	100	64	"d"
SOFT #5	101	65	"e"
PRIME	80	50	"P"
HELP	72	48	"H"
CANCEL	67	43	"C"
DEC. POINT	46	2E	""
ENTER	69	45	"E"
ZERO	48	30	"0"
ONE	49	31	"1"
TWO	0	32	"2"
THREE	51	33	"3"
FOUR	52	34	"4"
FIVE	53	35	"5"
SIX	54	36	"6"
SEVEN	55	37	"7"
EIGHT	56	38	"8"
NINE	57	39	"9"

#### Immediate K

Read Key Entry

Response format: "x..xx", where

x..xx is an ASCII string, 1 up to 7 characters long, encoding the keys

pressed. See table above. Response example: "1Ee".

Comment: if no key pressed, or if the keyboard has not been locked by

buffered K or L commands, the null ASCII character is returned.

Reading clears the buffer.

#### Immediate L

Request Manometric Module Identification

Response format: 4-character alphanumeric string.

Response example: "M805".

Comment: if no manometric module present, "None" is returned.

#### **Buffered P**

Pulse Contact Outputs

Syntax: Pnt

Parameter: n is the output number (1-3) and t is the pulse duration in tenths of a second (0-32767). If t is omitted, the last entered time is used. If t equals zero, the pulse is ignored. The previous pulse will also be ignored if t = zero.

Command example: "P210" to pulse OUT #2 for 1 second.

Comment: the pulse consists of reversing the output state for the time specified. The default duration after switching the power ON or after a master reset is 0.6 second. This pulse duration is used by the bufferd J command.

Buffered Q	Enter Pressure Value Syntax: "QBxx.x" in bars, "QPx.xx" in MPa, or "QKxx.x" in kpsi. Parameter: if xxx is omitted, the pressure value is read from the manometric module and the pressure unit is selected for the immediate Q command. Command example: "QP1.23" for 1.23 MPa. Comment: this command does not affect the pressure unit on the display.
Immediate Q	Read Pressure Value Response format: as above, in the pressure unit used by the last Q buffered command. The default selection is in bars. Response example: "B321" for 321 bars. Comment: if no manometric module present, "N" is returned.
Buffered W	Write Display Syntax: "WO = xxx" for upper line, "W1 = xxx" for lower line. Parameter: xxx is a 24-character long alphanumeric string. All 7-bit ASCII characters are valid. Note that characters shown on the Japanese-made display unit may differ from characters sent by a micro-computer (if used as a master controller).
Immediate W	Read Display Response format: "W0 =" (upper line) or "W1 =" (lower line) plus 24- character alphanumeric string. Response example: "W0 = Flow rate 1.000 mL/min". Comment: the first line to be returned is the last entered by the buffered W write command. If the write command has not been used, the upper line of display is read at the first occurence of the read command. The other line is read at the second one. And so on.
Immediate w	Read Display Buffers Response format: as above. Comment: reading mechanism same as above. The response is the image of the related display line from the buffer. This is also the message which will be restored to the display after a partial (W0/W1) or total (W) command.

#### **Identification Number**

Slave Pump	ID Number
Pump B	2
Pump C	3
Injection Pump (	D) 4

The Model 305 identification number is factory set to 1. In a multi-pump configuration, it is necessary to change the addresses of the slave pumps according to the opposite table.

To change the GSIOC ID number, turn the power ON. Press <Menu>, <I/O>, then <Prev>. The display comes "GSIOC Unit ID: 1". Key in the desired number (from 2 to 4) and confirm by hitting <Enter>, then <Quit>.

If a Model 302 or 303 pump is used as a slave pump, its ID number must also be changed by setting DIP switches on the Thumbwheel PC Board. See the related User's Guide on how to set ID number. Identification of the master pump (pump A) is not critical. However, if a Model 305 is used with a computer-based Gilson system, its ID number must be cleaned in the Setup menu of the software package (Model 704, 712, 714 or 715).

#### **Command Examples**

# **Example 1: GSIOC Command - Zeroing the Manometric Module**

In order to zero the Manometric Module when controlling the 305 from a computer-based system controller, stop all pumps, or pause without flow. This ensures no actual pressure inside the Manometric Module. The following GSIOC commands must be entered:

Send the zeroing K buffered command string. From the ready-to-run menu, this command string is made up of: Kdbbce.

This mimics the following key entry sequence: <Menu>, <I/O>, <Prev>, <Zero>, and <Quit>.

This command can be sent before the run by using the "GSIOC Control" option of the "Manual" menu. It can also be added to the method as a GSIOC event ("Edit" menu, "GSIOC Events" or "Gilson Instruments" option).

#### Example 2:

To write "HELLO" on the upper line of the display and "My name is Model 305" on the lower line.

Command	Comment
(B) W0 = HELLO	Write on upper line of display
(B) W1 = My name is Model 305	Write on lower line of display

#### **Example 3:**

To remote read the display.

The use of the lowercase w immediate command allows you to read messages displayed before the use of the W buffered write command.

Command	Response	Comment
(I) W	W1 = My name is Model 305	Read last entered line
(I) W	W0 = HELLO	Read the other line
Command	Response	Comment
(I) w	W1 =	Menu Run
(I) w	W0 =	Flow rate 1.000 mL/min

#### **Example 4: to restore display messages**

Command	Comment
(B) W0	Reconnect upper line to software.
(B) W1	Reconnect lower line to software.

This sequence sends "reconnect" commands to the internal software for the two related output devices. Original messages are restored. In the case where a separate access to the two lines of the display is not required, the same action is performed by a single command as above.

# Example 5: to select the flow mode, and start pumping at 1 mL/min.

Command	Comment
(B) W	Reconnect both lines to software
(B) W1	Reconnect lower line to software.
Command	Comment
(B) Kdea 1Ee	Mimic key entries
(B) K	Reconnect keyboard to software.

Codes sent correspond to the following key sequence: <Menu> <Mode> <Flow> <1> <Enter> <Run>. The reconnect command is necessary if a direct access to keyboard keys is required.

# Twin-pump Systems Appendix C

In each of the systems outlined in this manual there is a maximum of four pumps. The signals from the controller to the pumps are carried on a communications channel called the Gilson Serial Input Output Channel, or GSIOC for short. For a single pumping system, all of the pumps are connected in parallel and the controller distinguishes between any two pumps by a GSIOC identity number which you enter in the software. Each pump must have a different GSIOC identity number.

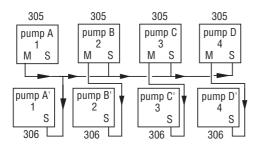
If we connect the pumps as shown in the following drawings, we have in effect two or more parallel pumping systems. The same commands will go to each pump associated with the same solvent, i.e. pumps A, and A' will receive the same commands. Pump B and B' will receive the same commands etc. Each associated pump will operate in exactly the same way, one being a mirror image of the other. In this way, we can create two identical, parallel liquid streams with one controller. The hydraulic outlet tubing can then be connected together to increase the overall flow rate. Theoretically there is no limit to the number of pumps that can be connected in parallel, but effectively a twin system with two liquid streams is the most practical. The advantage of this arrangement is to be able to use a smaller head and hence work at a higher pressure. For example, two 25SC heads could be used in parallel to obtain a flow rate of 50 mL/min. The maximum operating pressure would then be 28 MPa instead of a maximum of 14 MPa with a single 50SC head.

The actual flow rate of the combined pumps is the value programmed in the controller multiplied by the number of pumps in parallel.

#### **Two Possible Cases:**

- A system of mirror pumps controlled by a 305 Master pump.
- A system of mirror pumps controlled by a computer and a Gilson System Controller software.

When using a computer with a HPLC System Controller software, the identity numbers for the pumps can be chosen between 0 and 63.



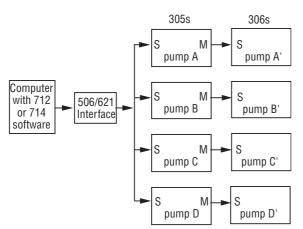
When using a 305 as a master, the following identity numbers must be used.

- Pumps A 1
- Pumps B 2
- Pumps C 3
- Pumps Inj 4

The requirements for a parallel system are:

- All pumps must be Model 305 or 306.
- Each pump in parallel, (A + A', B + B', etc...), must have the same headsize.
- Each pump in parallel must have the same GSIOC identity number, e.g. A and A' pumps must be 1.

Diagrams of the electrical connections for the GSIOC cables are given in the opposite figures.



M represents the GSIOC to SLAVE socket.

S represents the GSIOC FROM CONTROLLER socket.

# Reference Informations Appendix D

This chapter contains Table of Solvent Miscibility, Liquid Compressibility Values and Flow Rate Accuracy Principle.

# **Solvent Miscibility Table**

Miscibility means that solvents should mix with each other in all proportions. That solvents should be miscible is important both during elution and when switching from one solvent to another. You are advised to refer to the table, below, when selecting solvents. For some solvents, lower toxicity alternatives are indicated [(1), (2), (3)], as follows:

ACETIC ACID																			•	,				met		
ACETONE																								rofu	ura	n
ACETONITRILE																			(;	3) E	Ξth	an	ol			
BUTYL ALCOHOL																										
CHLOROFORM (1)																										
CYCLOHEXANE																										
DICHLOROETHANE (1)																										
DICHLOROMETHANE																										
DIMETHYLFORMAMIDE																										
DIMETHYL SULFOXIDE																										
DIOXAN (2)																										
ETHYLACETATE																										
ETHANOL																										
DI-ETHYLETHER																										
HEPTANE																										
HEXANE																										
METHANOL (3)																										
METHYLETHYL KETONE																										
I-OCTANE																										
PENTANE																										
ISOPROPANOL																										
DI-PROPYLETHER																										
TETRAHYDROFURAN																										
TOLUENE																										
WATER																										
XYLENE																										
		П			П	П	$\Box$	П	,,,		П	П	П	$\Box$	П	П	П	Щ	П	П			П	П		П
							€	븻	MIDE	ê								KETONE					AN			
			l	Ы	Ξ		IAN	ΓHΑ	MAI	9		Ш		œ							١.	ER	띪			
	اوا		ACETONITRILE	BUTYL ALCOHOL	CHLOROFORM (1)	CYCLOHEXANE	DICHLOROETHANE (1)	DICHLOROMETHANE	DIMETHYLFORMAMIDE	DIMETHYL SULFOXIDE		THYLACETATE		<b>DI-ETHYLETHER</b>			(3)	METHYLETHYL			NOL	<b>DI-PROPYLETHER</b>	TETRAHYDROFURAN			
	ACETIC ACID	Ä	Į	ALC	(OF	밀	SRO	)RO	1	¥	V (2)	ACE	о П	YLE	ŊĘ	Ш	METHANOL	LET	믣	밁	SOPROPANO	ΙΔ	H H	岁		اسا
		ACETONE	ET0	$\mathbb{Z}$	LOR.	5	뇘	됬	1ETI		DIOXAN (2)	Ϋ́	ETHANOL	팂	HEPTANE	HEXANE	THA	直	-OCTANE	PENTANE	PR(	PRC	IRA	TOLUENE	WATER	XYLENE
MISCIBLE	ACI	ACI	ACI	BN.	R	Š						山	Ē	금	里	出	ME	ME	Š	PEI	SC	1-10	Ξ	2	W	X
IMMISCIBLE																										

# **Liquid Compressibility Values**

## **Bibliography Data**

The values of isothermal compressibility given below can be used for the Compressibility value in the pump setup menu. These values are given under athmospheric pressure (X<sub>0</sub>) and are expressed in Mbar<sup>-1</sup>.

The opposite table refers to *Handbook of Chemistry and Physics, CRC Press, 60th Ed.* (1979).

Liquid	Temperature (°C)	Compressibility (Mbar-1)
Water	20 25 30 40 40	46 46 45 45 44
Benzene	20 25 30 40	94-95 96-97 101-103 110
Chloroform	20 25 30 40	97-101 97 108-110 118-119
Methylene chloride	25	97
Carbon tetrachloride	20 25 30 40	103-105 106-108 112-113 120-122
Ethanol	20 25 30 40	110-112 114-116 118-119 126-127
Acetone	20 25 30 40	123-127 124 133 144-156
Methanol	20 25 30 40	121-123 125-127 129-130 138
n-Heptane	20 25 30 40	140-145 142-149 150-155 160
n-Hexane	20 25 30 40	150-165 161-171 165-180 183
Diethyl ether	20 25 30	184-187 195-200 208-209

#### **Other Data**

Liquid		Compressibility (Mbar-1)				
Acetonitrile			99			
Tetrahydrofuran			93			
Water-methanol,	10-90	(v-v)	117			
u	20-80	"	86			
u	40-60	"	56			
u	50-60	"	52			
u	60-40	"	46			
u	80-20	"	40			

For other liquids currently used at ambient temperatures (20-25 °C), the following data is given. This data is a result of experiments done using the 305 pump, the figures are not presented as physical constants of scientific value.

If no data is available in this chapter for the liquid you use, and if you wish an accuracy error within the specifications, you can experimentally determine a value to reach this goal.

To do so, use a trial-and-error empirical method. Select the initial value for Liquid Compressibility according the following guidelines:

- For an organic solvent, take a value given for the same, or similar, chemical family.
- For a mixture, including salt aqueous solutions, take the value of the dominant solvent.

Pump your liquid under high pressure to obtain a significant error, preferably use a gravimetric method if you know the density of the liquid.

Then, with a few successive approximations, adjust the Liquid Compressibility value by assuming a linear relationship between this parameter and the resulting error.

## Flow Rate Accuracy Principle

To generate the selected flow rate with high accuracy, maintained under high pressure and for a variety of liquids, the 305 software adds to complementary corrections to the basic "piston flow rate". Defined from the piston stroke volume only, the piston flow rate is theoretically accurate at athmospheric pressure only.

The objective flow rate, F, is considered as the sum of three components:

$$F = F_0 + F_1 + F_2$$

F<sub>0</sub>, the piston flow rate decreases when pressure increases;

F<sub>1</sub>, the compensation flow rate for the liquid compressibility, increases with pressure;

 $F_{2'}$  the compensation flow rate for all others factors, also increases with pressure.

The piston flow rate,  $F_{0}$ , is defined by:

$$F_0 = N_0 V_S$$

with  $N_0$ , number of piston cycles per unit time; and  $V_{s'}$  piston stroke volume.

The compensation flow rate for the liquid compressibility,  $F_1$ , is calculated as a function of five variables:

$$F_1 = f_1 (F_{0'}, V_{S'}, V_{D'}, P, X)$$

where  $V_{\scriptscriptstyle D}$  is the volume of the dead space inside the compression chamber; P, the operating pressure; and X, the compressibility of the liquid under the pressure P.

In the 305 software, X is calculated using the simplified Tait equation:

$$X = c$$
 $P + d$ 

Coeeficient c varies only slightly with the nature of the liquid. It is a constant included in the software.

Coefficient d is calculated from the Liquid Compressibility at athmospheric pressure,  $X_0$  (for P = 0), entered by the user as a "set-up" parameter. Values of  $X_0$  for some common solvents are tabulated in the previous page.

Operating pressure, P, is continuously transmitted to the pump by the Manometric Module (pressure feedback).

The complementary compensation flow rate for all other factors,  $F_{\gamma}$ , is defined as the difference:

$$F_2 = F (F_0 + F_1)$$

It was measured and the experimental results were expressed using a simple function of operating pressure P:

$$F_2 = f_2 (a, b, P)$$

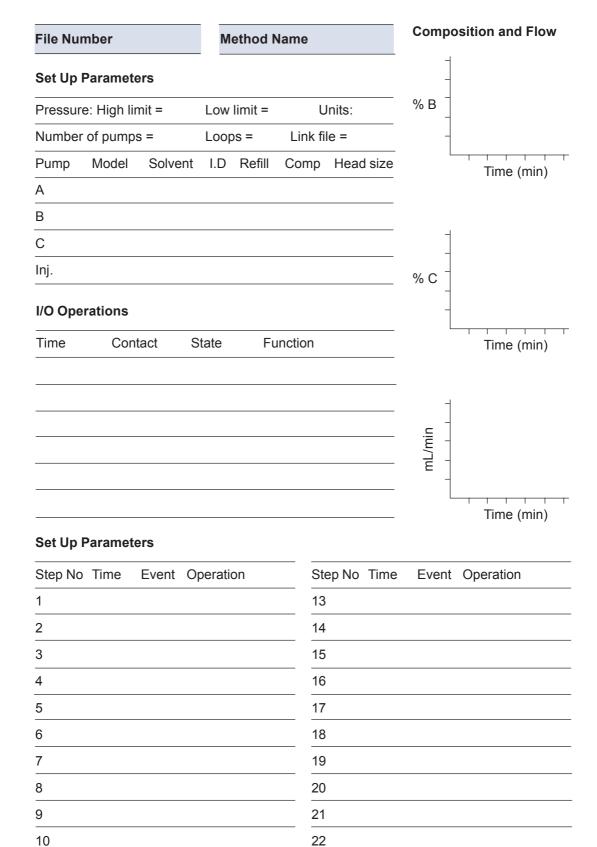
Coefficients a and b were determined for each pump head. They are manufacturing constants attached to the parameter Head Size, a second 'Setup' parameter entered by the user.

All of these factors taken together allow the 305 to deliver a highly accurate flow rate, independent of the pump head size, type of liquid and pressure.

# 305 Programming Sheet Appendix E

The programming sheets provided on the next pages should be properly filled out prior to programming the pump.

This wil ensure that the parameters are entered correctly, and will enable you to quickly cross check them when required.



23

24

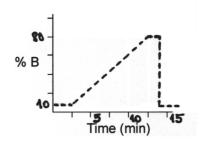
11 12

#### File Number 1

### Method Name SAPONIN

#### **Set Up Parameters**

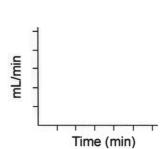
Pressu	re: High I	Low limit = 20 Units: bas							
Number of pumps = 3				Loops = 20 Link file = _					
Pump	Model	Solvent	I.D	Refill	Comp	Head size			
A	305	ACN	1	250	99	25			
В	306	watea	21	125	46	25			
С									
Inj.	306	Sample	4			5			



**Composition and Flow** 

#### **I/O Operations**

Time	Contact	State	Function
1.2	1	Pulse	Event on Recorder
6	2		Starto FC 202



Time (min)

#### **Set Up Parameters**

Step No	Time	Event	Operation
1	0	FLow	14 mL/min
2	0	MIXT.	10%
3	0.5	INT.	4 mL injected
4	2.5	MIXT.	10%
5	12	MIXT.	80%
6	13	MIXT.	80%
7	13.1	MIXT.	10%
8	15	MIXT.	10%
9			
10	o-0		
11		)	
12		2	

Step No Ti	me	Event	Operation
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23		-	
24			

# **Technical Data**

# **Appendix F**

The following information presents construction and operational characteristics for Gilson 305 Pumps.

# **Type of Pump**

Programmable reciprocating pump with singlepiston interchangeable head, constant stroke, and special fast-refill motion.

## **Working Range & Performance Data**

# Working Range, Pump Heads and associated Manometric Modules

A complete Gilson liquid delivery system includes up to four pumping modules with appropriate heads, a manometric module and a mixer for gradient elution.

Pump head (model)	Flow rate range* (mL/min)	Pressure range* (MPa)	Manometric module (model)		
5SC	0.010 - 5	0.1 - 60	805		
10SC	0.050 - 10	0.1 - 60	805		
10WSC	0.050 - 10	0.1 - 60	805		
10WTi	0.050 - 10	0.1 - 60	805		
25WTi	0.125 - 25	0.1 - 28	806		
25SC	0.125 - 25	0.1 - 28	806		
50SC	0.250 - 50	0.5 - 14	806		
100SC	0.500 - 100	0.5 - 7	807		
200WTi	1.000 - 200	0.5 - 3.5	807		

SC:Standard self-centering piston.

- Ti: Titanium liquid contact-parts.
- W: Washing compartment for salt-concentrated solutions (> 0.1M).
- \*: Minimum values for the flow rate and pressure are not absolute limits, they are indicated to obtain specifified precision and accuracy. Flow rate is adjustable down to 0.01 % of the maximum flow rate.

#### **Adjustable Parameters**

Liquid compressibility, refill time and inlet pressure. Compressibility from 0 to 2000 Mbar <sup>-1</sup>, refill time from 125 to 1000 ms and inlet pressure from 0 to 10 MPa.

#### **Residual Pulsations**

Typically less than 1 %.

# Flow Rate Precision and Accuracy at 20°C over full working range

#### **Coefficient of Variation**

0.1 to 0.6% with aqueous solutions or hydro-organic polar solvent mixtures and 0.3 to 1% with hydrocarbons or chlorinated volatile solvents.

#### **Maximum Accuracy Error**

±1% with water over the full flow rate and pressure ranges.

#### **Liquid-contact Materials**

316L stainless steel, titanium, sapphire/ceramic, ruby, PCTFE, PEEK, PTFE/HDPE.

# Binary Gradient Systems (two-solvent composition programming)

#### **Mean Accuracy Error**

 $< \pm 3$  % determinated in the range 0 - 10 % by 1 % steps and in the range 0 - 100 % by 10 % steps.

#### **Deviation from Linearity**

 $< \pm 1 \%$ .

#### Repeatability

< 1 % maximum imprecision for any composition.

Repeatability of gradient HPLC analysis (ICI test F for liquid chromatographs):

< 0.2 % for retention times, and < 0.7 % for peaks areas.

Composition (% of solvent B)	Total flow rate (% of maximum)
1 - 99	100 - 50
2 - 98	50 - 25
5 - 95	25 - 10
10 - 90	10 - 5

These specifications are valid for any model of the 305 technology in the following conditions:

liquids of known compressibility, pumped at ambient temperatures (20-25 °C), in the entire pressure range, with any pair of identical pump heads operating within the ranges indicated hereafter.

#### **Total Delay Volume (ASTM E-19.09.07)**

1.2~mL dynamic mixing chamber, in-line filter (0.5  $\mu$ m, 0.4 mL) and 0.5 x 500 mm tubing.

#### **Effective Mixing Volume (ASTM E-19.09.07)**

1.1 mL in the same conditions as above.

#### **Control and Interfaces**

#### **Operation Modes**

Constant flow rate (Flow), constant volume (Dispense), and timed-based sequence (Program) for up to four Gilson pumps controlled either by one 305 acting as a Master 305, or by an external computer.

#### **Programmable Parameters**

Solvent composition points for high pressure mixing of 3 solvents from 3 pumps.

Flow rate points that can be superimposed over gradient composition profile.

Timed events for programming 3 output contact closures, 1 input and 1 injection pump.

Time, adjustable from 10<sup>-2</sup> to 10<sup>4</sup> min, with increments from 0.001 to 1 min depending on the range used.

Flow control, adjustable from 0.01 % to 100 % of the maximum flow rate of the pump head being used.

Possibility to program 999 loops with unlimited linking of files.

Storage for 10 user programs and 4 error files with a maximum of 25 points and timed events in each program.

## **Dispense Mode**

The dispensed volume is fully adjustable, in mL, between 10-4 and 100 mL multiplied by the model number of the pump head. The same range is available for the injection pump in the program mode.

#### **User Interface**

2-line, 48-character LCD display.

Front panel keypad.

Built-in help messages.

## **Electrical Interface**

4 inputs, 3 programmable outputs.

## **Digital Interface**

Gilson Serial Input/Output Channel.

The pump can act as master or slave.

### **Environmental Conditions**

### **Storage**

Indoor use only.

Installation : Category II.

Altitude: Up to 2000 m.

Temperature range: 4 - 40 °C.

Pollution degree 2.

Humidity: Up to 80 %.

### **Power Requirements**

Frequency: 50 to 60 Hz.

Voltage: 100 to 240 Vac.

Power rating: 110 VA.

## **Size and Weight**

Size (W x D x H): 330 x 330 x 150 mm.

Weight: 10 kg (22 lb).

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