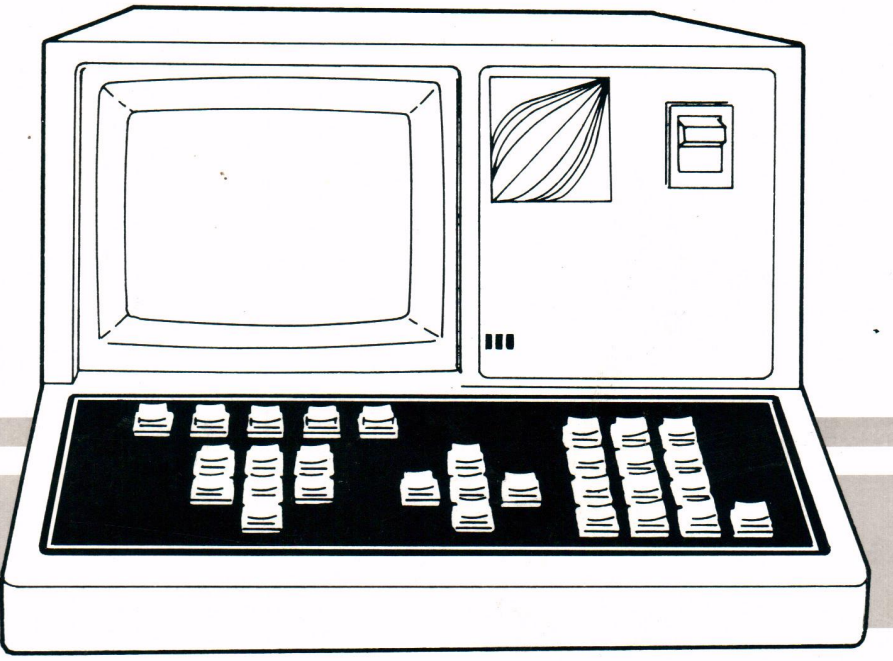
Model 680 Automated Gradient Controller

Operator's Manual



Waters
Division of MILLIPORE

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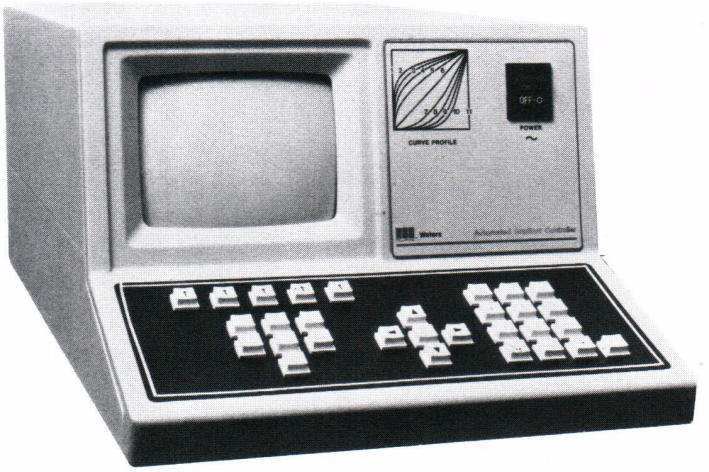


FIGURE 1-1 THE MODEL 680
AUTOMATED GRADIENT CONTROLLER
(Part Number 42000)

The Waters™ 680 Automated Gradient Controller (AGC) is an interactive, microprocessor-based pump controller that can operate and monitor up to three Waters solvent delivery pumps. The automated isocratic and binary or ternary gradient operations may be overridden manually at any time for complete operator control, or allowed to run unattended according to the operator's programmed instructions.

The AGC features a self-contained tutorial operator's guide for each step of the programming and operation process which may be accessed as the need arises. A system of non-obtrusive informational displays and messages ensure fault-free use — even for operators unfamiliar with the AGC.

The memory permits programming up to 10 gradient tables (with 10 operational steps for each) and 10 timed events tables (containing 20 operational steps) in permanent storage (even after a power failure) for repeated use and modification. Seven external switches are controlled through manual or timed events tables to run devices such as a solvent switching valve (four + 12 volt power terminals are also provided for devices requiring operating current).

A run start input is provided to allow an injector, either manual (Model U6K Universal Liquid Chromatograph Injector, referred to as the Model U6K) or automatic (WISP™ Model 710B intelligent sample processor, referred to as the Model 710B), to initiate the analysis. Another input accepts an abort signal from an external switch which stops the pumps. The HOLD output terminal connects to the Model 710B binary coded decimal (BCD) communications board to provide a signal, initiated from the AGC, to prevent further system operation when a failure necessitating a system shutdown has been detected.

2

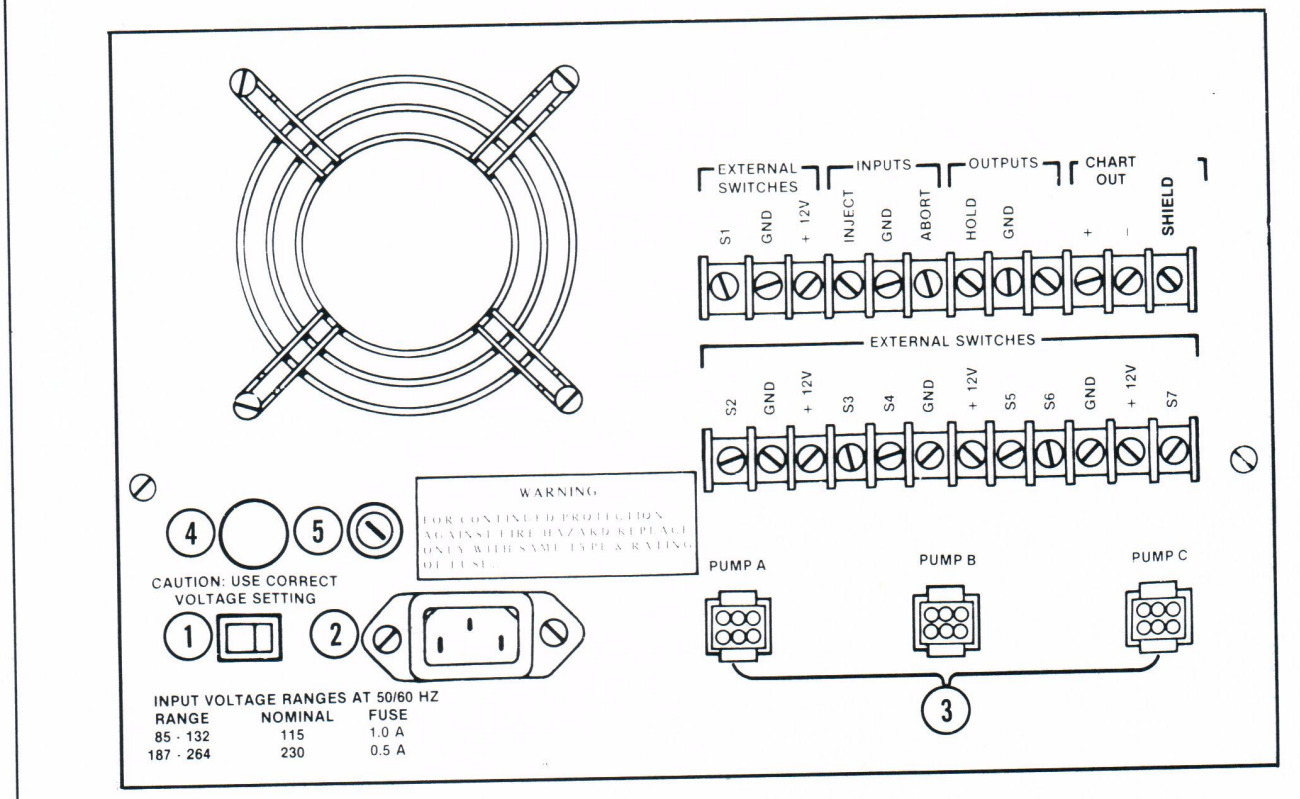
THE AUTOMATED GRADIENT CONTROLLER

2.1 UNPACKING

The AGC is packed in a specially designed container to prevent damage in shipment. Remove the packing material from the box, grasp the AGC from the bottom and lift the instrument straight up. The unit weighs 22.9 pounds (10.4 Kg).

After unpacking the instrument and associated hardware, check the contents against the packing list to ensure that the shipment is complete.

Inspect all items for damage. Report any damage immediately to both shipping carrier and Waters. Check the contents of the startup kit (Part Number 42012) against the list enclosed in the startup kit.



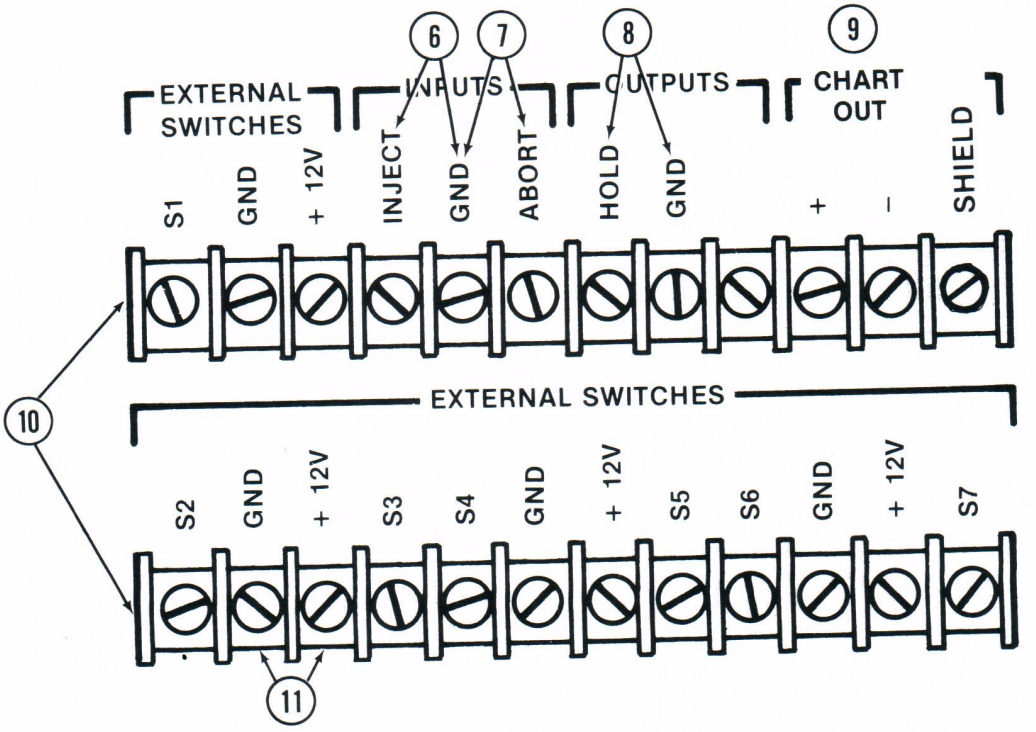


FIGURE 2-1 REAR PANEL FUNCTIONS

TABLE 2-1 REAR PANEL FUNCTIONS

Description	Use
1. Voltage Selection Switch	Match operating voltage (115 or 230 Vac)
2. Power Connection	Connector for ac power cord
3. Pump Connectors	Standard interface for all Waters pumps. Three inputs designated as A, B and C
4. Fuse	1 amp fuse (slow-blow)
5. Fuse	Punch out hole for installation of a second fuse and fuseholder for countries requiring fusing of both lines
DIGITAL (TTL) CONNECTIONS*	
6. Inject Terminal (and Ground)	Compatible input to accept a signal from the injector to begin a run
7. Pump Abort Terminal (and Ground)	Compatible input to accept a pump abort signal from an external switch (customer supplied) to stop all pumps
8. Hold (and Ground)	Compatible output for HOLD signal to the Model 710B BCD board to prevent further injections in case of power failure, pressure shutdown, abort signal, etc.

*Transistor-transistor logic (TTL) — +5 volts = Off, switch open
0 volts = On, switch closed

Description	Use
EXTERNAL OUTPUT CONNECTIONS	
9. Chart Out (+, −)	Plot output to a recorder which can represent pressure, %A, %B or %C changes during controller operations (10 mV represents full scale)
10. Output Terminals (and Grounds)	Seven external output switches (S1-S7) under time or operator control used to control external devices (maximum current capacity is 1 amp total)
11. +12 V Supply (and Grounds)	Power source for external relays to drive external devices (up to 1 amp total current)

2.2 ENVIRONMENTAL REQUIREMENTS

The preferred conditions for installation are:

Humidity — at least 20%
Ambient temperature — 41-104 °F (5-40 °C)
Relatively dust free

Refer to Appendix D, Environmental Specifications, for required conditions.

DO NOT install the AGC on a thickly carpeted surface. Vent holes on the base of the AGC require a one half inch space for proper ventilation. Six inches of clearance is recommended at the rear of the AGC to permit easy access to the cable connections and to allow additional ventilation space.

2.3 ELECTRICAL REQUIREMENTS

THE AGC requires a properly grounded ac power supply line that is relatively free of transients and fluctuations. The unit is shipped adapted for 60 Hz operation. Remove the AGC cover and change the position of the switch as indicated in (Figure 2-2) for 50 Hz use. Set the rear panel voltage selection switch as indicated in the following section.

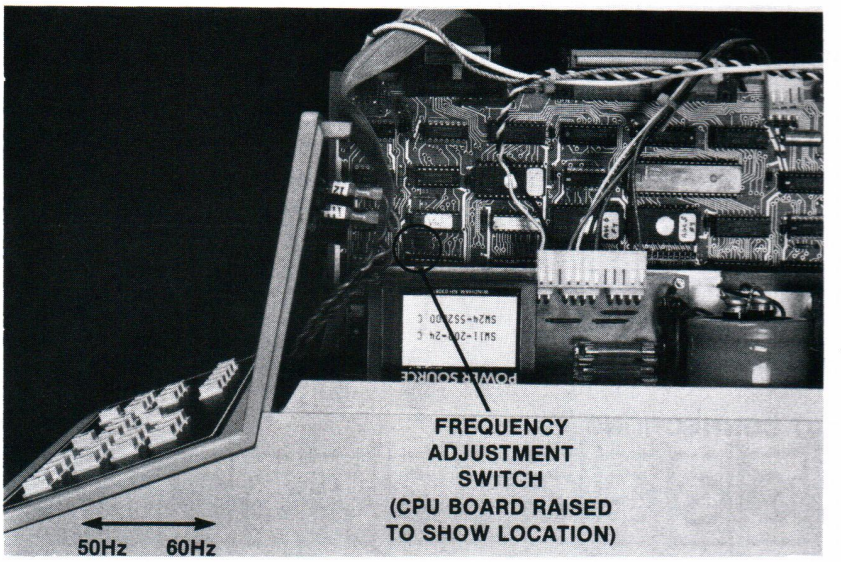


FIGURE 2-2 OPERATING FREQUENCY ADJUSTMENT

Voltage Selection

The AGC can be adapted to operate at the two nominal voltages listed below:

Nominal Voltage	Voltage Range	Fuse (amp)
115	85-132	1
230	187-264	0.5

The desired voltage may be selected by positioning the red slider switch on the rear panel Figure 2-1 to the RIGHT (facing the rear panel) for 115 Vac and LEFT for 230 Vac. Prior to powering up the instrument, be sure that the proper input voltage has been selected.

2.4 INSTALLATION

CAUTION

ALWAYS DISCONNECT THE POWER CORD FROM THE INSTRUMENT BEFORE MAKING ANY CONNECTIONS

NOTE

MODEL 6000A PUMPS WITH SERIAL NUMBERS BELOW 15,000 MUST BE MODIFIED WITH AN UPGRADE KIT, PART NO. 25808 (INCLUDED IN THE STARTUP KIT) TO DELIVER PUMP PRESSURE READINGS

Instructions are included with the kit to allow you to perform this procedure (which entails the addition of a single wire to the existing output cable of the pump) or contact your Waters Service Representative.

2.4.1 Electrical Connections

1. Make sure the voltage selection switch is set to the appropriate voltage.

2. Connect the injector (Model 710B or U6K) and a recorder as shown in Figure 2-3 (optional BCD connection is also shown).

   If a manually operated abort switch (or an environmental [solvent] monitor) is desired connect it as shown in Figure 2-4. A momentary, or contact closure, pushbutton switch is sufficient to activate this input and may be purchased locally at an electronics store.

3. Connect the devices requiring +5 volt (digital) level signals to the external switch terminals (S1-S7) and the associated grounds at the rear of the AGC (see Figure 2-4).

   If any of these devices require an external source of power four +12 V power output terminals (and adjacent grounds) are provided (Figure 2-4).

NOTE

THE MAXIMUM TOTAL CURRENT AVAILABLE (DIVIDED AMONG ALL DEVICES IN USE) IS 1 AMP

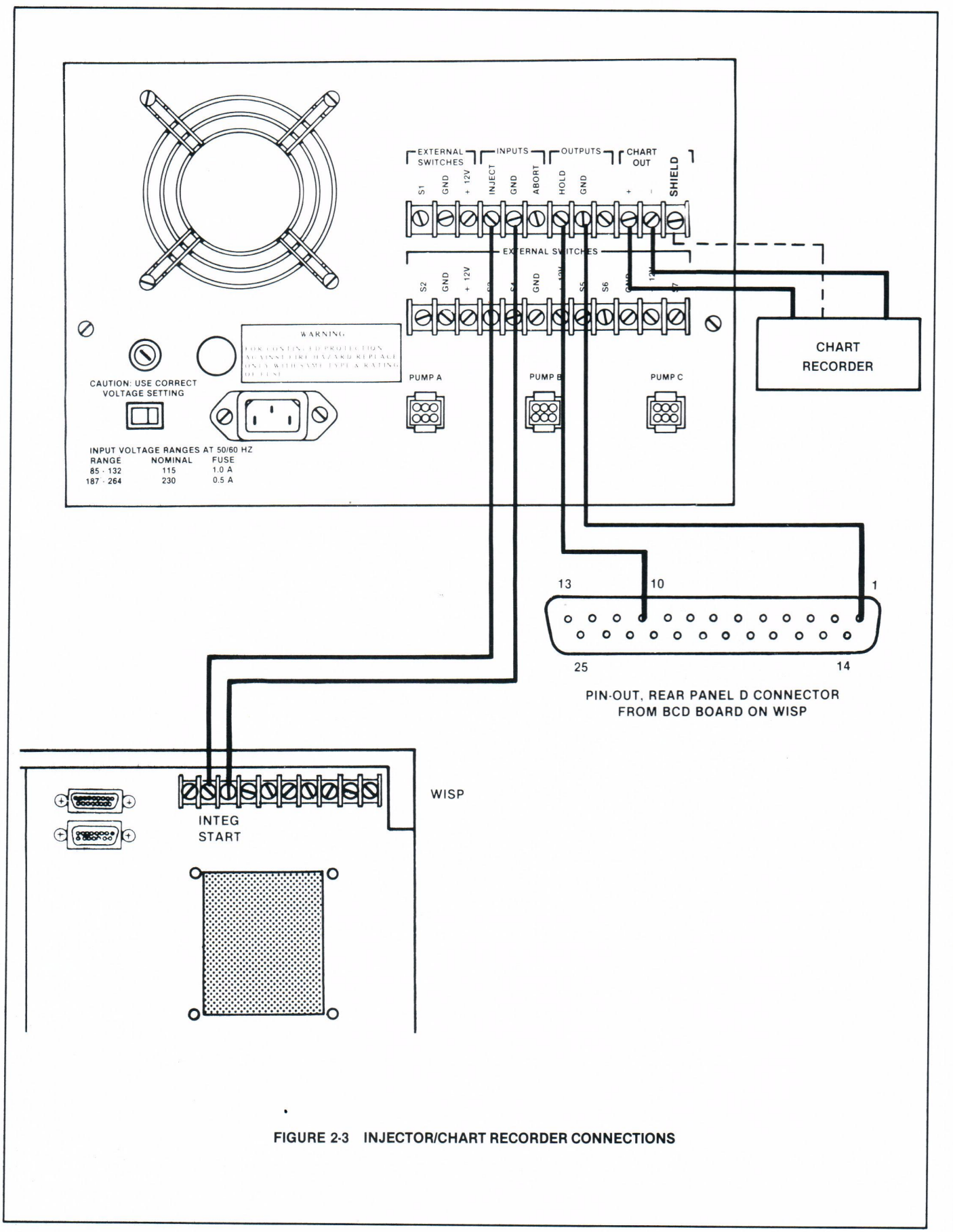
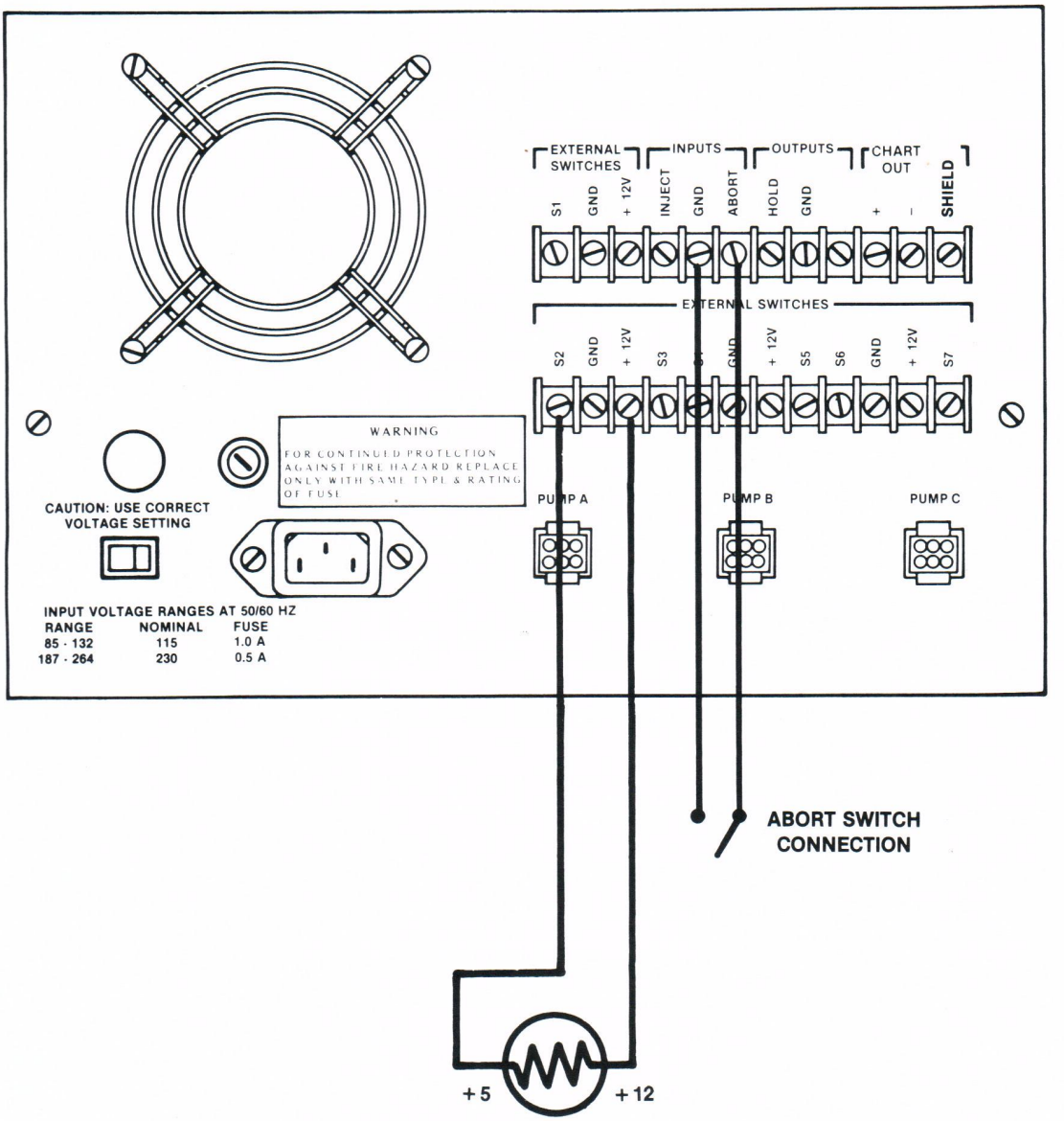


FIGURE 2-3 INJECTOR/CHART RECORDER CONNECTIONS



CONFIGURATION FOR A DEVICE REQUIRING +12 VOLTS (AND UP TO 1 AMP CURRENT)

SWITCHES 1 THROUGH 7:

HIGH OUTPUT (5V) — SWITCH OPEN DEVICE OFF

LOW OUTPUT (OV) — SWITCH CLOSED DEVICE ON

FIGURE 2-4 EXTERNAL SWITCH CONNECTION

2.4.2 Pump Connections

Plug the pumps into the pump outlets at the rear of the AGC and label them as A, B, and C. Connect the stainless steel tubing, Part No. 25592, (included in the Startup Kit) as described in Figure 2-5. The standard pump connection method will suffice in many solvent delivery applications.

An alternate method for connecting three pumps is shown in Figure 4-2 (Section 4, System Diagnostics) should some solvent mixing applications require it. The connection requires a second compression screw tee (Part No. 75215) and two compression screw unions (Part No. 97332) which are not included in the Start-up Kit.

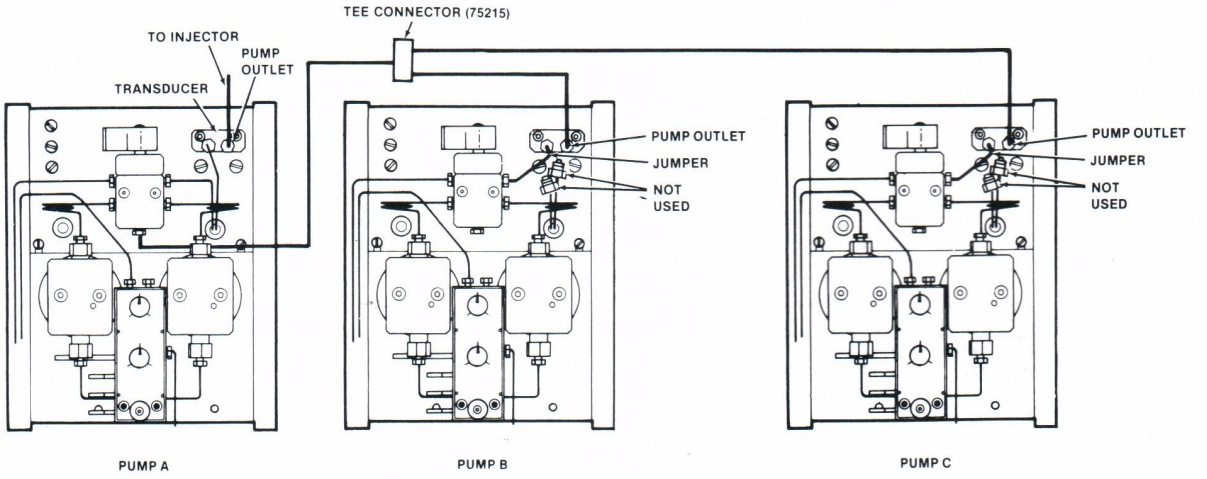


FIGURE 2-5 MULTIPLE PUMP TUBING CONNECTIONS

COMPRESSION SCREW AND FERRULE ASSEMBLY

Connecting the pump tubing requires a sharp edged file, one $^{5}/_{16}$ inch open end wrench (two are required if connecting the compression fitting to a union), and two pair of pliers.

1. Scribe the circumference of the tubing with the file edge and grip either side of the breakpoint with pliers wrapped in cloth to protect the surface of the tubing. Break the tubing by bending back and forth.

2. Slide the compression screw, nut-end first, over the tube followed by the ferrule (large end of taper first). Compression screws and ferrules (Part No. 25604) are included in the Startup Kit.

3. Properly fit the ferrule by bottoming the tube in the fitting seat for which its use is intended.

4. Tighten ¼ to ½ turn to set. Do not overtighten.

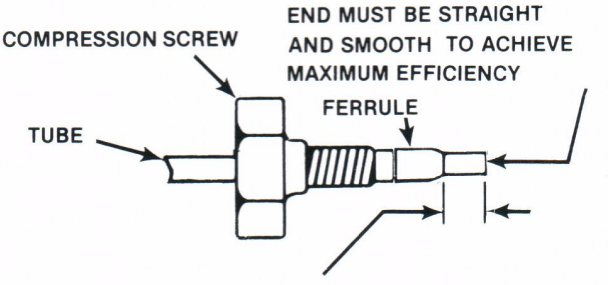


FIGURE 2-6 COMPRESSION SCREW AND FERRULE ASSEMBLY

2.4.3 Second Fuse Installation

Two fuses, one on each side of the line, are required in some countries. A diagram for this simple modification is shown in Figure 2-7. The second fuseholder and fuse are provided in the Startup Kit. The mounting hole has been punched in the rear panel.

WARNING

DISCONNECT AC POWER AT THE REAR PANEL TO PREVENT INJURY OR SHOCK. REMOVING THE POWER SUPPLY PROTECTIVE COVER ALLOWS POSSIBLE CONTACT WITH LINE VOLTAGE.

- Remove the top cover.
- Pop out the blank cover over the mounting hole on the rear panel.
- Insert the fuseholder (Part No. 97557) into the rear panel mounting hole from the outside. Position the fuseholder the same as the standard fuse so that the solder terminals are accessible.
- Fasten the retainer nut from the inside.

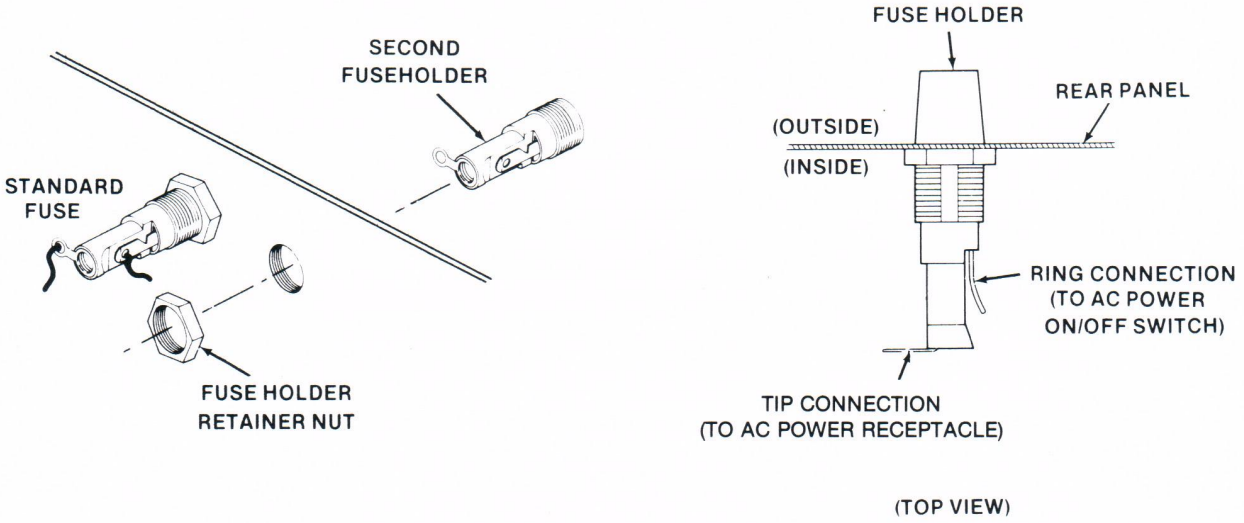


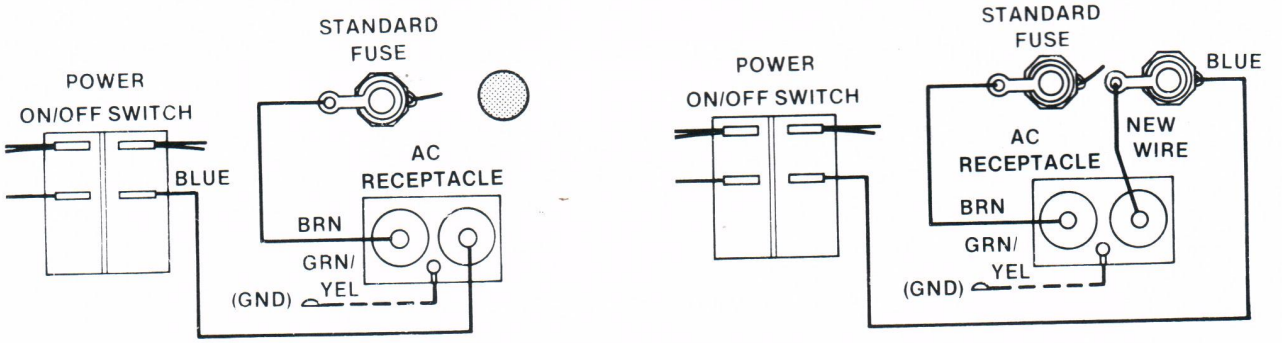
FIGURE 2-7 SECOND FUSEHOLDER ASSEMBLY INSTALLATION

A soldering gun or iron is required for the next step. Figure 2-8 illustrates before (A) and after (B) the modification.

- Unsolder and remove the blue wire from the ac power receptacle.
- Connect and resolder the blue wire to the ring connection of the second fuseholder.
- Install a new 6″ wire and solder it between the tip connection of the second fuseholder and the ac power receptacle.

Install the 0.5 amp fuse (Part No. 42091, if use will be 115 V install 1 amp, Part No. 42090) by mounting it in the fuse carrier (Part No. 97558) and the installation is complete.

Replace the AGC cover and reconnect the power cord.



(A) BEFORE MODIFICATION

(B) AFTER MODIFICATION

FIGURE 2-8 SECOND FUSE WIRING MODIFICATION

3

OPERATION

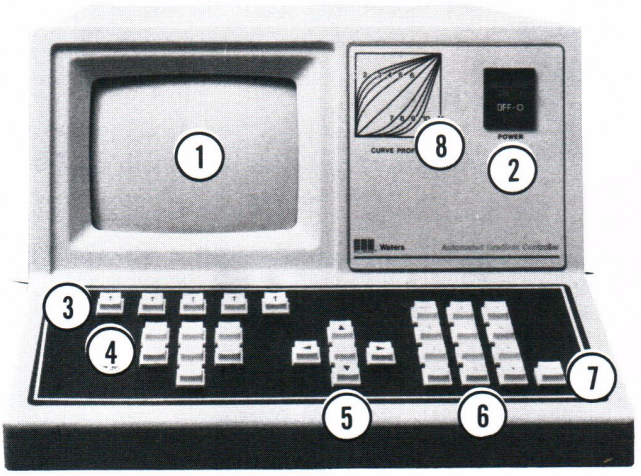


FIGURE 3-1 AGC FRONT PANEL

1. **CRT DISPLAY SCREEN —** White characters, on a dark background (the position indicator, or cursor, on the screen appears as a white box with dark characters), display operational parameters and pump status. The screen also displays the self-contained operator's guide, verifies entries and transmits diagnostic error messages.

2. **POWER SWITCH —** On/off rocker switch which indicates power applied to the instrument by its position.

3. **FUNCTION KEYS —** Five keys located directly below the CRT screen. The variety of functions available with these keys are displayed on the screen immediately above each key as the pages are selected.

   They allow, depending on the selected mode of operation:

   - Editing of currently running or stored programs
   - Permanent program storage
   - Manual gradient control (Run/Initial/Hold/Stop)
   - Stop and resume flow

4. **PAGE KEYS —** Six keys provide direct access to operations display pages. The key marked EXPLAIN provides operator's guidelines for the page on display when this key is struck.

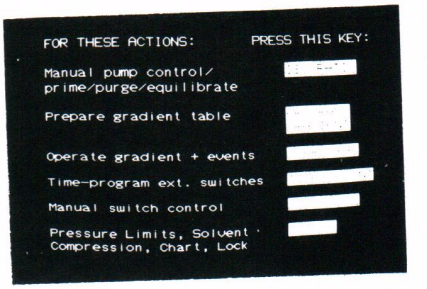
5. **CURSOR MOVEMENT KEYS —** Four keys move the CURSOR (the reverse video box on the screen) up, down, left or right. The key marked HOME moves the cursor directly to the home position from anywhere on the page (the home position for each page is identified in the following operations discussions).

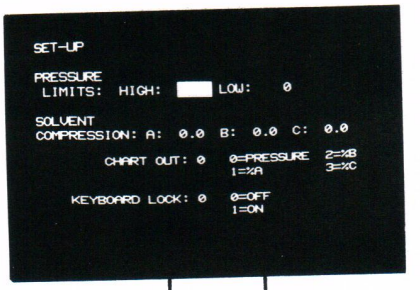
6. **KEY PAD —** Numeric data input keys (0 to 9, decimal point, CLEAR).

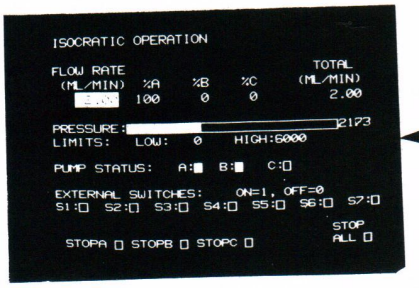
7. **ENTER KEY —** Required to enter numeric data. Places flashing values into temporary storage.

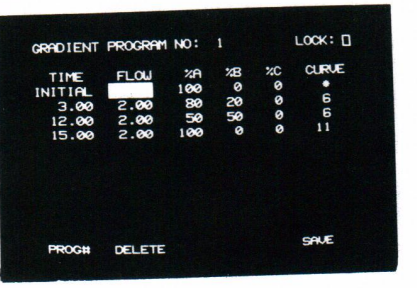
8. **CURVE PROFILES —** Convenient reference of all standard curves representing the rate of change during gradient operation.

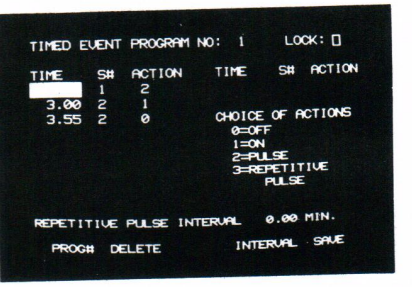
PAGE GUIDE











AN OPERATOR'S GUIDE IS AVAILABLE BY USING THE EXPLAIN KEY FOR EACH PAGE SHOWN WHEN IT IS DISPLAYED ON THE SCREEN. APPENDIX A CONTAINS REPRESENTATIONS OF EACH DISPLAY.

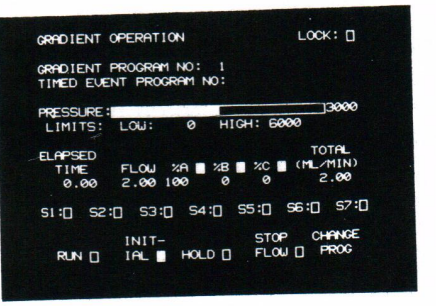


FIGURE 3-2 PAGE DISPLAYS

3.1 GENERAL OPERATING INFORMATION

Programming and monitoring of gradient and isocratic operation on the AGC is done through pages displayed on the CRT screen. Each page is also accompanied by an EXPLAIN page that may be called upon with the touch of a button to provide the operator with guidelines on how to use or program that page.

The AGC page displays are shown in Figure 3-2 (opposite) arranged in order of use. The following sections are arranged in similar fashion; Section 3.2, Isocratic Operation, and Section 3.3, Gradient Operation. In either case, both rely on initial preparation provided by the SET-UP page explained in this section and the manipulation of values, common to all displays, which are also explained here.

Turn the power on. A self-diagnostic routine is performed (see Section 4 for details) followed by this display:

WATERS ASSOCIATES, INC.
AUTOMATED GRADIENT CONTROLLER

The logo display is replaced by the PAGE GUIDE.

TO CALL UP A DESIRED PAGE

Select one of the operations listed on the PAGE GUIDE and press the appropriately marked page key.

TO CALL UP AN EXPLANATION PAGE (For the displayed page only)

Press the EXPLAIN key.

NOTE

THESE ARE INFORMATIONAL PAGES ONLY

Press the EXPLAIN key a second time to return to the operational page. Repeat this process with all pages to familiarize yourself with their content. A detailed list of the explanation pages for each page shown in this section is provided in the appendix.

THE CURSOR

The cursor location on each page is indicated by REVERSE VIDEO (dark characters, white background) and its movement is as indicated on the cursor keys.

The HOME key returns the cursor to a position designated for each page from anywhere on the page. These positions are indicated in the following discussions of Isocratic and Gradient operation.

NOTE

THERE IS NO CURSOR MOVEMENT CAPABILITY ON THE PAGE GUIDE AND GRADIENT OPERATION PAGES.

TABLE 3-1 CURSOR FUNCTIONS

Cursor Activity	Description
If the Cursor is not flashing (steady)	Value shown is either in volatile (scratch pad) or permanent memory
If the Cursor is flashing	Operator has keyed in the value but not pressed ENTER — entry is still pending (not in volatile or permanent memory).
	AGC has displayed a preset (or default) internal value — entry is still pending (not in volatile or permanent memory).
Cursor Location is blank	Gradient Program or Timed Events Tables — value appearing in the column immediately above is copied when ENTER is pressed.
	Other Pages — previous value will reappear when ENTER is pressed unless new value is entered.
Using the Cursor Movement Keys	Leaving a space before pressing ENTER — a flashing value will not be entered (previous value will return). If the display is steady the value remains as displayed.

HOW TO ENTER VALUES

Operational values are entered in one of two ways: individually, or by full lines.

Individual Entries

Entries that do not require other values before the AGC can translate the information for use during the operation may be entered singly. When these are entered the cursor does not automatically shift to the next required entry position. Entries may be made individually (instead of as full lines) on the:

- SET-UP page
- External switch line of the ISOCRATIC page

1. Move the cursor to the desired location.
2. Key in the desired value. For example, to enter 2500 (psi) for the high pressure limit on the SET-UP page, press 2-5-0-0. The cursor will flash to indicate the pending entry.

   If an error is made, press the CLEAR key and type in a new value.
3. Press the ENTER key. The display will become steady. If the value entered is outside of the limits allowed by the AGC, a "beep" will sound and a diagnostic message will flash at the bottom of the page. Press the CLEAR key and type in an acceptable value.
4. Move the cursor to the location of the next value to be entered and continue in the same fashion.

Full Line Entries

Entries requiring associated information before the AGC can use the parameter for operation are indicated by the cursor automatically shifting to the next position where an entry is called for. Cursor movement is restricted until the line is complete. Individual entries on the line may then be altered as described previously. All values on a line must be entered before the cursor may be moved up or down for the:

- GRADIENT PROGRAM page
- TIMED EVENTS page
- Flow line of the ISOCRATIC page

HOW TO DELETE LINES

Programs from the Gradient Program and Timed Events pages may also be modified by deleting whole lines.

1. Move the cursor to the TIME position on the line to be deleted.
2. Press the key under DELETE (see Sections 3.4.1, How to Prepare for a Gradient Run, and 3.5, Timed Events). This deletes the line the cursor is presently on and moves succeeding lines up.

NOTE

DELETIONS OF AN ENTIRE TABLE MUST BE PERFORMED LINE BY LINE (NO ONE STEP TABLE DELETIONS).

THE FIRST LINE OF A GRADIENT TABLE CAN NOT BE DELETED. ENTER NEW VALUES ON THE FIRST LINE OF THE GRADIENT TABLE INSTEAD OF DELETING THE LINE.

3. Press the key under SAVE. This action is necessary to store a new table or to commit the blank or deleted table to memory.

HOW TO SAVE PROGRAMS

Gradient and Timed Events programs are placed in a "scratch pad" memory as they are set up on the display screen. This temporary memory is not used for operation by the AGC. The programs must be SAVED, or stored in the permanent memory in order to be used.

To save a newly created program:

1. Enter all desired values.
2. Press the key under SAVE. A prompt, ENTER PROGRAM #: will appear with a flashing number.
3. If the program is to be stored under this number, press ENTER. Otherwise refer to Section 3.6.6, Duplicating Gradient and Timed Events Programs.

3.2 SET-UP PAGE (ISOCRATIC AND GRADIENT)

Values for Pressure Limits, Solvent Compression and Chart Out are entered on the SET-UP page and apply to all gradient and isocratic programs used with the AGC. If values other than those initially programmed are desired, clear the existing values and enter new values.

Press the SET-UP page key. The cursor will be at the Pressure Limits/High (Home) location.

OPERATOR ENTRIES

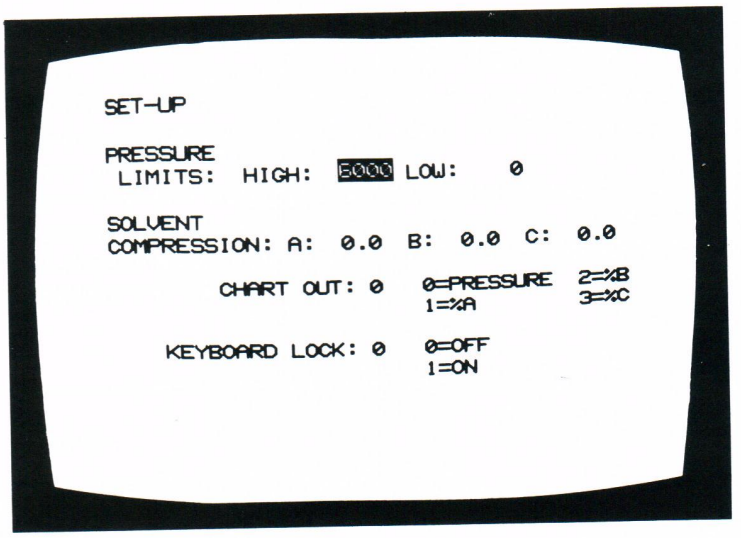
Pressure Limits — Maximum and minimum operating limits for the system.

OPTIONAL FEATURES

Solvent Compression — Compensation for flow rate changes due to pressure fluctuations, see Section 3.6.9.

Chart Out — Plot operating pressure or pump % to a recorder through 10 mV output,

Keyboard Lock — Protection against accidental entries during gradient operation, see Section 3.6.8.



There are no FUNCTION KEYS for this page.

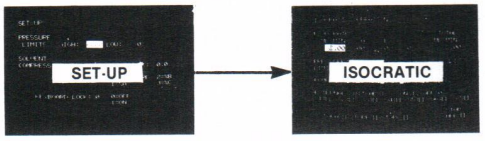
FIGURE 3-3 SET-UP PAGE

To enter Pressure Limits, position the cursor and enter high and low pressure limits (range: 51 - 6000 high, 0 - 5950 low, no decimal values).

These values will appear on the GRADIENT and ISOCRATIC pages under the bar graph. If the system exceeds these limits for more than two seconds (high pressure limit) or ninety seconds (low pressure limit) pump shutdown will occur and the controller will be forced to the ISOCRATIC page (and mode of operation) immediately. A HOLD signal will be sent to the Model 710B through the BCD link (if this option is installed) to prevent further injections.

Pump operation may be resumed by manually re-entering the flow rate and pump % values although another shutdown will occur if the cause has not been corrected.

3.3 ISOCRATIC OPERATION



Isocratic operation allows direct, immediate control of up to three Waters pumps and up to seven external devices. There are no time-dependent functions in this mode. The pumps respond immediately to the conditions entered for operation on the flow line when the last value on the line (%C) is entered.

Whenever a change is made in the flow line (Enter is pressed on Flow, %A, or %B), the TOTAL FLOW ml/min display will flash to indicate that:

Pending entries may alter total flow.
The current actual flow rate may be different from that displayed.

TOTAL FLOW will not equal FLOW RATE unless %A + %B + %C = 100. TOTAL FLOW will always indicate the actual flow. For example, if the Flow Rate is 2.00 and %A + %B + %C = 100, the total flow is 2.00 ml/min. If the Flow Rate is 2.00 and %A + %B + %C = 50, the total (or actual flow) is 1.00 ml/min

PUMP OPERATION

Turn the pumps on.

Press the **ISOCRATIC** page key. The cursor will be at the Flow Rate (Home) position and Pump Status lights will illuminate for each pump that is powered.

OPERATOR ENTRIES

Flow Line — Enter values for actual flow.

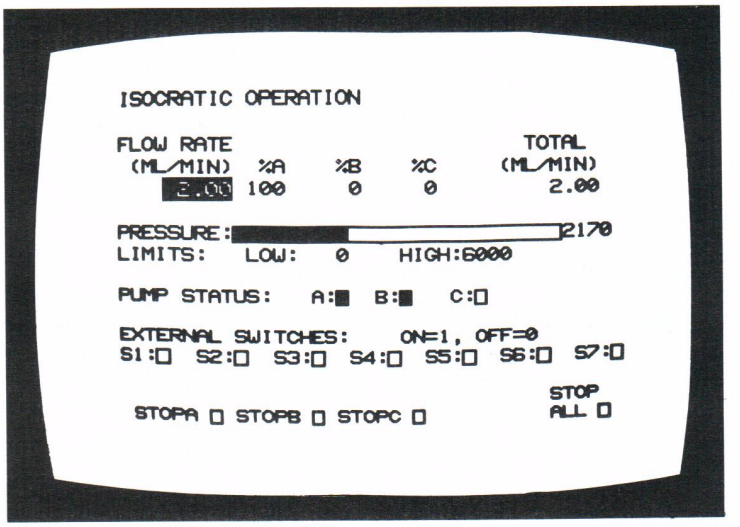
External Switches — (Not required for pump operation) manual on/off for devices connected to the rear panel. See Section 3.6.1.

MONITOR DISPLAYS

Pressure Limits — Active monitor of operating pressure.

Pump Status — Lights indicate pump power is ON.

Total ml/min — Reflects actual flow at all times.



FUNCTION KEYS

KEY 1	KEY 2	KEY 3	NOT	KEY 5
STOP A	STOP B	STOP C	USED	STOP ALL

Press keys to stop pumps (Function Indicator light on); repeat to resume flow (light off).

FIGURE 3-4 ISOCRATIC OPERATION

NOTE

THIS PAGE CAN BE LEFT ON DISPLAY TO ACTIVELY MONITOR OPERATION

Enter the desired Flow Rate value. For example, to enter a Flow Rate of 2.00 ml/min, press 2, ENTER. To enter 2.10, press 2.1, ENTER. The cursor will move to the %A position and will be flashing 100. The TOTAL ML/MIN value will be flashing to indicate the pump output may be different than the current display until committed to memory by pushing ENTER.

Enter pump percent compositions (0-100, no decimals). Press the desired percentage for pump A and ENTER. The cursor will move to %B and will be flashing the figure equal to 100% – %A – %C. For example, if %A = 40, %B will be flashing 60. If this is the desired value for %B, press ENTER. Otherwise, press the key pad for the desired %B value and ENTER. The cursor will move to %C (100 – %A – %B is shown) to allow you to perform the same operation for the third pump.

When %C is entered, the flow line is complete and the pumps will start running with the specified conditions. (Total ml/min display will be steady).

NOTE

IF %A + %B + %C IS GREATER THAN OR LESS THAN 100 A "BEEP" WILL SOUND AND A DIAGNOSTIC MESSAGE WILL APPEAR AT THE BOTTOM OF THE PAGE. THIS MESSAGE DOES NOT REQUIRE THAT VALUES BE CHANGED.

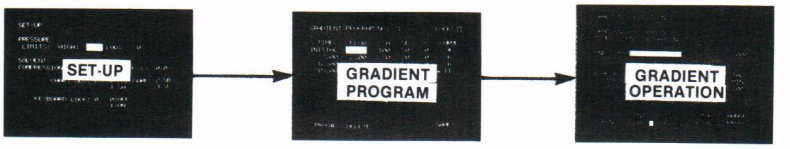
MANUAL PUMP STOP/START CONTROL

Any or all of the pumps can be stopped at any time by pressing the appropriate Function key (Stop A, Stop B, Stop C or Stop All) directly beneath the screen. To resume pump operation, press the key a second time or enter new flow conditions.

NOTE

WHEN ANY OR ALL PUMPS ARE STOPPED BY THE FUNCTION KEYS, THE INDICATOR LIGHT NEXT TO THE SCREEN ID IS ILLUMINATED INDICATING THE FUNCTION IS ENGAGED.

3.4 GRADIENT OPERATION



Gradient elution is dependent on time controlled flow rate changes of two or more pumps. Solvents are mixed in varying proportions throughout the separation and flow rate, analysis time and the proportion of the flow contributed from each pump (the gradient curve) are used to generate a chromatogram of well-defined peaks in a minimum of time.

Gradient operation requires the following three steps:

Develop the gradient program
Enter it into memory
Start the gradient

3.4.1 How To Prepare For A Gradient Run

Press the **PROGRAM GRADIENT** page key. Home position for the cursor is at FLOW on the INITIAL line.

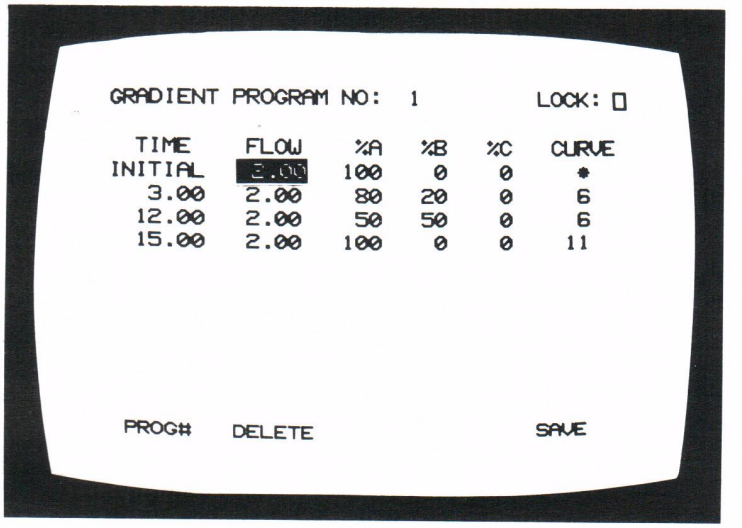
OPERATOR ENTRIES

Gradient Program No. — Enter number of the program to be run, edited or created.

Gradient Table (Time, Flow, %A, %B, %C, Curve) — Enter values to create gradient-programs for gradient operation (up to 10 programs; 10 lines per program).

OPTIONAL FEATURE

Lock — Indicates keyboard is locked.



FUNCTION KEYS

KEY 1 PROG#	KEY 2 DELETE	NOT USED	NOT USED	KEY 5 SAVE
Move cursor to display a program	To delete individual lines			To commit a table to memory for use indexed by number

FIGURE 3-5 GRADIENT PROGRAM

IF THE AGC IS IN THE ISOCRATIC MODE WHEN THIS PAGE IS ACCESSED: THE TABLE WILL BE BLANK. PROGRAM 1 (PRESET) WILL BE FLASHING

IF THE AGC IS IN THE GRADIENT MODE WHEN THIS PAGE IS ACCESSED: THE PROGRAM CURRENTLY RUNNING WILL BE DISPLAYED

1. Develop the Gradient Table

   a. Set up a grid having six columns and label each column as below.

   b. The number of rows in the grid (up to 10 rows plus initial conditions) will be determined by the number of steps to be executed in the gradient.

TIME*	FLOW*	%A	%B	%C	CURVE
Initial	2.0	100	0	0	*
3.00	2.00	80	20	0	6

*Time (min); Flow (ml/min).

   c. Enter each gradient segment time (the time refers to the time since injection) and respective conditions into the grid. (For information on how to determine gradient conditions — initial conditions, final conditions, gradient curve, etc. — see Appendix C, How to Develop a Gradient Program.)

2. Enter the Program

   a. Press the desired program number (if a program had previously been stored under this number, it would now be displayed on the screen) and ENTER. The cursor will move to the Flow position in the initial line.

   b. Enter the values from the grid into the gradient table. If an error is made, press the CLEAR key and type in a new value.

NOTE

A VALUE FROM THE PREVIOUS LINE CAN BE COPIED TO THE NEXT BY PRESSING ENTER WHEN THE CURSOR LOCATION IS BLANK

   The cursor will move to the next position on the line. After entering the last value on a line (curve) a beep will sound. A message "TO USE THESE NEW VALUES, PLEASE SAVE" will appear at the bottom of the page and the cursor will move to the TIME position on the next line.

3. Save the Program

   When the complete gradient table has been entered, press the key under SAVE. The cursor will move to ENTER PROGRAM NO.

NOTE

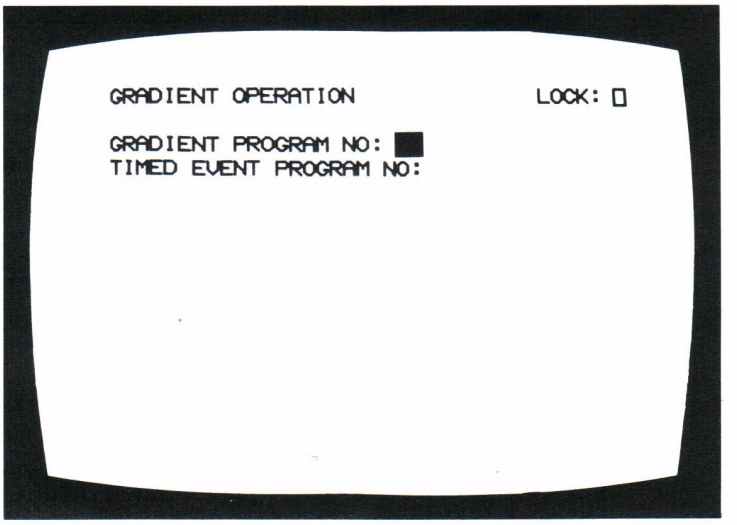
VALUES ENTERED ON THE GRADIENT PROGRAM PAGE DO NOT BECOME OPERATIONAL UNTIL SAVED. PROGRAMS THAT ARE NOT SAVED ARE LOST AND CANNOT BE ACCESSED FOR FUTURE USE.

Type in the number under which this program is to be saved, press ENTER and the screen blinks momentarily confirming the program is ready for use.

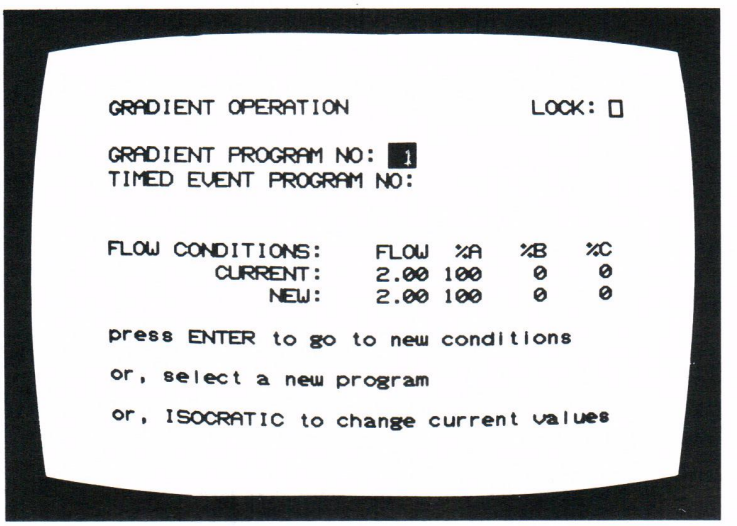
3.4.2 Transition To Gradient Operation

Press the **OPERATE GRADIENT** page key.

If you are not currently operating in the gradient mode, such as when making a transition from isocratic to gradient or at startup, the following display will appear:



Press ENTER and this interim display appears as a prompt and a warning:



Press ENTER again to place the AGC in the gradient or time controlled mode of operation.

FIGURE 3-6 TRANSITION TO GRADIENT OPERATION

3.4.3 How To Perform a Gradient Run

The gradient program can be started automatically by a signal from an injector to the INJECT terminal at the rear of the AGC, or may be started manually by pressing the unmarked Function key under RUN. The program may then be run to completion or may be adjusted manually to hold gradient conditions or stop flow. (The HOLD adjustment may be particularly useful in methods development. STOP FLOW is generally used upon completion of a run but also serves as a manual control for temporary or emergency shutdown.)

If the AGC is currently operating in the gradient mode when the **GRADIENT** key is pressed, this display will appear:

OPERATOR ENTRIES

Gradient/Timed Event Program No. — Enter number of gradient and timed events programs to be run.

MONITOR DISPLAYS

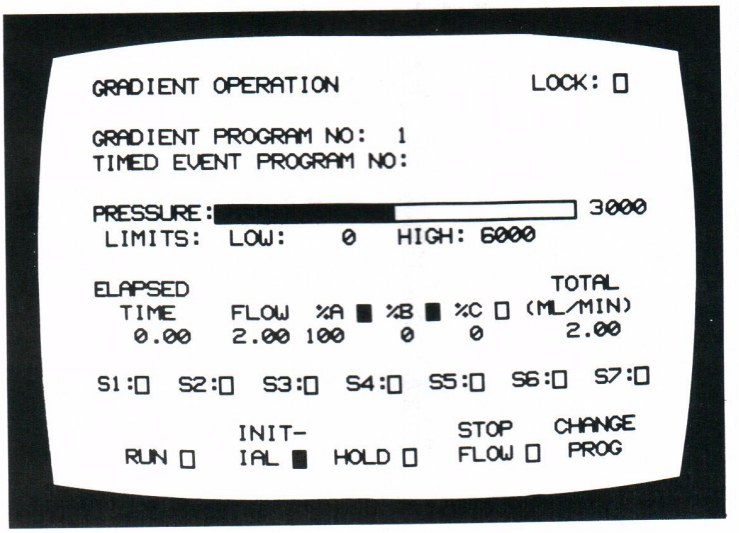
Pressure Limits — Actively monitors operating pressure.

Flow Line — Actively monitors current gradient parameters during a run including pump on/off status.

External Events Line — Boxes light when devices are activated.

OPTIONAL FEATURE

Lock — Indicates keyboard is locked.



FUNCTION KEYS

Refer to Section 3.4.4, How to Control A Gradient Run During Operation.

FIGURE 3-7 GRADIENT OPERATION

NOTE

THIS PAGE CAN BE LEFT ON DISPLAY TO ACTIVELY MONITOR OPERATION

The cursor is now at the Timed Events Program Number location. If no timed events program is to be run, press ENTER.

If a timed events program is to be run, see Section 3.5.

INJECT THE SAMPLE AND INITIATE THE GRADIENT

Press the function key under RUN or initiate the gradient from the injector. A "beep" will sound and gradient operation will begin.

Gradient values during operation are monitored on the GRADIENT page. Operating pressure is indicated by a bar graph and digital readout. External device operation is indicated by lit indicator positions. Gradient conditions flow, elapsed time and pump percentages are continually monitored on the flow line.

Repetitive runs may be initiated by a signal from the injector. At each injection, the AGC returns momentarily to initial conditions and repeats the gradient program. Refer to Section 3.6.2 for programmed returns to initial conditions.

3.4.4 How To Control A Gradient During Operation

To HOLD a gradient from change during a run, press the key under HOLD (the HOLD Function indicator light is on).

Flow rate and pump percentages will now run at unchanging rates. Elapsed time will freeze.

To continue the gradient from where it was held, press the key under RUN (the RUN Function indicator light is on; Hold light is off).

To STOP FLOW and HOLD the gradient, press the key under STOP FLOW (the STOP FLOW Function indicator light is on).

Pumps will stop and flow will equal zero.

To continue the gradient from where it was stopped and restart the flow, press the key under RUN (the RUN indicator light is on; the STOP FLOW light is off).

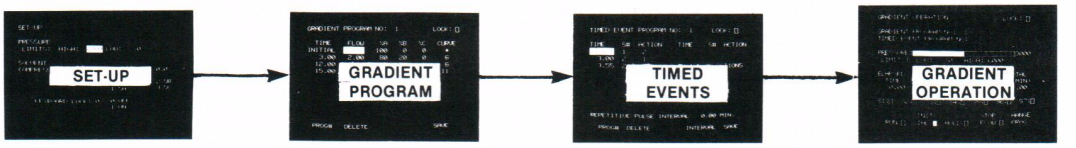
TO RESET CONDITIONS FOR SUBSEQUENT GRADIENT RUNS

The last step in any gradient program should return the system to initial conditions automatically. This step will ensure, without operator intervention, that before each succeeding injection the system returns to equilibrium at initial conditions, refer to Section 3.6.2, Programmed Return to Initial Conditions.

To manually repeat a gradient and return immediately to the initial conditions of the gradient program, press the unmarked key under INITIAL. Flow will continue and Gradient conditions will return to those at Initial Conditions (Time 0.00) and remain there unchanging.

To change the program to be run, press the key under CHANGE PROG. Type in the number of the gradient program to be run, press ENTER. If another Timed Event program is to be run, type in that number and press ENTER. If not, just press ENTER.

3.5 TIMED EVENTS



Output to external devices (such as fraction collectors or solvent switching valves) can be incorporated into gradient operation by programming actions for up to seven devices on the TIMED EVENTS page. (There is also an internal, timed, audible beeper accessed as S8 which can be used to serve as a reminder for the operator such as to signal the end of a run.)

Devices may be time controlled to turn on, off, pulse or repeatedly pulse at regular operator specified intervals. Pulse duration is ON for 100 ms, then OFF. If a device was ON and a pulse specified, the switch would turn off momentarily before pulsing. If the interval is 1.0 min, the device would pulse (ON for 100 ms, then OFF) and would remain off for 1 min then pulse, then wait 1 min, then pulse again, etc. This would continue until some other action is specified for that switch at which time the repetitive pulsing would cease.

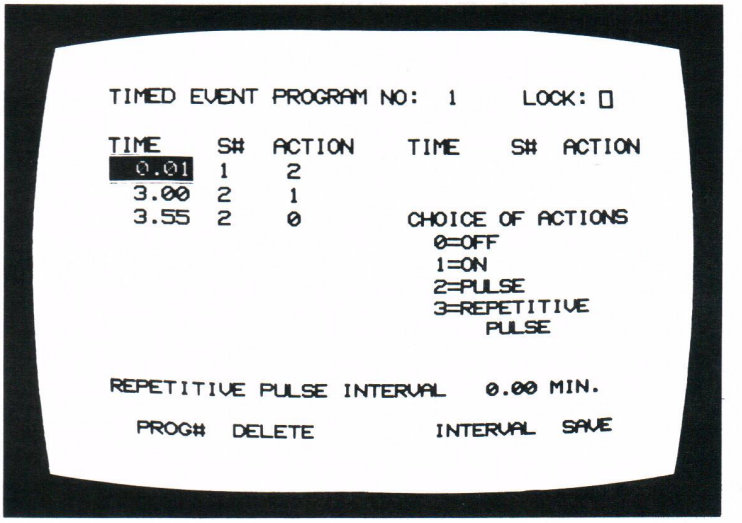
NOTE

THIS TABLE CAN ONLY BE USED IN THE GRADIENT MODE

OPERATOR ENTRIES

Timed Event Program No. — Enter number for program to be run, edited or created.

Timed Events Table (Time, S#, Action) — Enter values to create timed switch operation program (up to 10 programs; 20 lines per program).



OPTIONAL FEATURES

Repetitive Pulse Interval — Time interval between pulses — settable from 0.01 — 655.35 min.

Lock — Indicates keyboard is locked.

FUNCTION KEYS

KEY 1 PROG#	KEY 2 DELETE	NOT USED	KEY 4 INTERVAL	KEY 5 SAVE
Move cursor to display a program	To delete individual lines		To enter time interval for pulses	To commit program to memory for use

FIGURE 3-8 TIMED EVENTS OPERATION

3.5.1 How to Prepare a Timed Events Program

1. Develop the Timed Events table.

   a. Set up a grid with three columns labelled as below.

TIME	SWITCH NO.	ACTION
0.00	1	2
3.00	2	1
3.55	2	0

   The number of rows in the grid (up to 20) will be determined by the number of actuations of the device.

   b. Enter the time of each actuation during the run. Associated with each time is the number of the switch the device is connected to and the type of actuation (on, off, pulse, repetitive pulse). Place these values in their respective locations in the grid.

2. Press the **TIMED EVENTS** page key. The cursor will be at the TIMED EVENTS PROGRAM NO. location.

   a. Press the desired new program number (if a program had previously been stored under this program number, it would now be displayed on the screen) and ENTER. The cursor will move to the time position on line one of the table.

   b. Enter the values from the timed events table onto the TIMED EVENTS page in full lines before progressing to the next line. The "Choice of Actions" display will disappear from the screen when necessary to allow the full 20 lines to be entered.

NOTE

THE AGC WILL COPY VALUES FROM THE PREVIOUS LINE IN EACH COLUMN (DOWN) BY PRESSING ENTER WHEN THE CURSOR IS ON A BLANK SPACE

   c. Enter a time for Repetitive Pulse Interval if a switch action has been designated as Action 3. (Press the Interval Function key. Enter the time value between pulses.)

Press the Function key under SAVE. The cursor will move to ENTER PROGRAM #.

NOTE

VALUES ENTERED ON THE TIMED EVENTS PAGE DO NOT BECOME OPERATIONAL UNTIL SAVED. PROGRAMS THAT ARE NOT SAVED ARE LOST AND CANNOT BE ACCESSED FOR FUTURE USE.

Press ENTER and the screen blinks momentarily confirming the program may be used (to duplicate a timed events program see Section 3.6.6).

3.5.2 How to Run a Timed Events Program

Press the OPERATE GRADIENT page key.

IF THE AGC IS OPERATING IN THE GRADIENT MODE:

Press the CHANGE PROG Function key.
Press ENTER with the cursor at the GRADIENT PROGRAM NO. location.
ENTER the number of the Timed Event Program.

IF THE AGC IS OPERATING IN THE ISOCRATIC MODE:

ENTER the gradient program number (the cursor moves to the Timed Event location).
ENTER the number of the Timed Event Program.

Inject the sample.

3.5.3 Effects of Operational Changes on Switch Status

TABLE 3-2 TIMED EVENTS OPERATION

Condition	External Switch Status
Isocratic Mode	Executed as set (manually) on the ISOCRATIC page. Timed Event Program does not run.
Gradient Mode	
Initial	Timed Events programs can be changed without affecting switch status. Whenever Initial Function key is pressed, switches freeze in their current state.
Hold or Stop Flow	If Timed Events Program changed by operator all switches reset to off. If Timed Events Program currently running — switches freeze in their current state.
Run (also signal from injector)	If Timed Events Program changed by operator — all switches reset to off. Initiates Timed Events Program (if Run signifies inject) or continues running Timed Events Program if resuming operation after Hold or Stop Flow. Does not reset switches to off at each new injection (any on at end of run will remain on when a new injection is made until program times them off). Repetitive Pulse DOES reset at Injection.

3.5.4 How to Stop a Timed Events Program

To stop a program without resetting the switches:

1. Press CHANGE PROG.
2. Press ENTER on GRADIENT PROGRAM NO. (the cursor moves to the Timed Event Program Number location).
3. Press CLEAR and ENTER a blank for the TIMED EVENT PROGRAM NO.

Timed Event program execution will stop and switch status remain constant. Subsequent injections with a blank Timed Events program number will not run any timed events program.

If the Gradient mode is aborted (the AGC goes to the Isocratic page), Timed Events program execution will cease and switches freeze in their current state. The switches can then be manually changed on the Isocratic page.

3.6 HOW TO USE THE OPTIONAL FEATURES

3.6.1 Manual Control of External Devices

Go to the ISOCRATIC page, move the cursor to beneath the desired external switch and:

Press 1, ENTER to turn the device on.
Press 0, ENTER to turn the device off.

3.6.2 Programmed Return to Initial Conditions

The time required to equilibrate a system to initial conditions varies according to the type of column, mobile phase, etc. A standard rule of thumb such as three column volumes may be sufficient in many instances but must be substantiated through experience with your particular system.

A return to initial conditions allowing for sufficient equilibration time can be accomplished in two ways depending on whether a WISP Model 710B intelligent sample processor is included in your system.

Set the RUN TIME to allow the initial flow conditions sufficient time to equilibrate, or program the desired length of time after the run into the WISP automatic injector as EQUILIBRATION TIME. The next injection (initiated by the injector) will not be undertaken until this time has elapsed.

The return to initial conditions can be programmed into the gradient table which is especially useful with columns requiring greater care when making the transition.

1. Program the line following the actual analysis for a time interval and reverse the pump percents and flow rate (if necessary) back toward the conditions at the start of the run.

2. As indicated above, the number of steps (lines) used to return to initial conditions and the length of time required depends on your actual application requirements.

3. Enter all information and SAVE the table for use.

3.6.3 Programmed Flow Stop (or Reduction)

Programming the pumps to stop flow (gradient mode) following a completed program provides a number of benefits for ensuring fault-free unattended operation. Solvent waste is minimized and low pressure protection is guaranteed by stopping the pumps (should the operator inadvertently omit programming a low pressure shutoff setting).

Columns and solvent systems that require constant flowing solvent can be programmed to reduce the flow rate in the same manner.

Enter an additional line on the Gradient program containing the following information (see Section 3.4.1, How to Prepare for a Gradient Run, for inserting new program information):

TIME — far enough past the last program line to allow proper analysis.
FLOW — either 0 or a reduced rate.
%A,%B,%C — copy the previous line.
CURVE — This value should always specify CURVE #11. The flow rate will therefore decrease only when the time programmed for the line is reached, not gradually during analysis as with other curve profiles.

3.6.4 Editing a Previous Program

Currently running or future gradient or timed event programs may be edited at any time. As changes are made, the AGC will automatically sort an addition (or deletion) to either the Gradient Program or Timed Event tables based on the time entered for the line. This constitutes a new program and must be saved before attempting to use it for operation.

If a change is made in the FIRST gradient segment, the INITIAL function key must be pressed before starting the run MANUALLY. Otherwise the change made to the first segment will not be effective until the following run. This procedure is only required for restarting a run from the AGC itself, not from an external source (such as the WISP Model 710B injector) through the inject input terminals.

NOTE

IT IS NOT POSSIBLE TO MODIFY A GRADIENT SEGMENT CURRENTLY BEING EXECUTED. ANY SUBSEQUENT SEGMENT CAN BE MODIFIED DURING A GRADIENT RUN AS LONG AS ANY CHANGES ARE LOADED IN THE MEMORY USING THE SAVE AND ENTER KEYS.

3.6.5 Programming a Binary Gradient With a Third Pump Controlled Isocratically

In some cases you may wish to run a binary gradient along with one pump run at a relatively constant rate. It may be to your advantage to enter the program as a binary gradient with a third pump controlled isocratically, rather than calculating total flow rate plus pump percentages for each pump in each gradient segment.

1. Develop the gradient table.
   a. Set up a grid having 6 columns as below and enter each gradient segment time and respective binary gradient conditions into the grid (%A + %B = 100). For example:

TIME	FLOW	%A	%B	%C	CURVE
0.00	2.0	100	0		*
3.00	2.0	80	20		6

b. Calculate the third pump's % value based on desired flow output

$$\%C \text{ Value} = \frac{\text{Desired Output (ml/min)}}{\text{Flow Rate Value}} \times 100$$

For example, the desired output is 1.0 ml/min.

$$\%C \text{ Value} = \frac{1.0}{2.0} \times 100 = 50$$

Enter values for %C on the grid.

2. Enter the values from the grid onto the gradient table on the GRADIENT PROGRAM page.

After entering a gradient segment line, a message (%A + %B + %C > 100) will appear at the bottom of the page. This message is a warning to indicate that Total Flow will exceed the entered Flow Rate. For example, with a flow rate of 2.0 and pump percentages of 50, 50 and 50, total flow will equal 3.0 (2.0 X 150%).

3. Press the key under SAVE
   a. Type in the number under which this program is to be saved.
   b. Press ENTER.

3.6.6 Duplicating Gradient and Timed Event Programs

Duplicating Gradient and Timed Events programs (also referred to as the clone feature) is a quick way to enter values for new programs. Duplication provides a model from which values for new programs can be entered by modifying or deleting existing values. This eliminates the need to enter all new values.

1. Display the program to be duplicated.
   a. Press the key under PROG #.
   b. Type in the number of the program and press ENTER.
2. Modify the displayed program as desired.
   a. Delete full lines to be eliminated.
   b. Change individual values.
   c. ENTER new values.
3. Press the key under SAVE.
   a. Type in the new number under which this program is to be saved.
   b. Press ENTER.

NOTE

IF A PROGRAM IS CURRENTLY STORED UNDER THE CHOSEN PROGRAM NUMBER, IT WILL NOW BE DISPLAYED ON THE SCREEN

If you wish to eliminate this program to save the new program, press 1, ENTER. If you do not wish to save the program displayed on the screen, press 0, ENTER. Enter a new program number when prompted and press ENTER.

When the SAVE is performed (values stored in memory) the parameter values will disappear momentarily.

3.6.7 How To Erase an Old Program

When 10 programs have already been stored on the AGC, it will be necessary to replace one of the existing programs in storage in order to enter and use a new program.

1. Press the Gradient or Timed Events page and press the number of the program to be replaced. Press ENTER.

2. Move the cursor (to the second line if a Gradient Program) and press the DELETE key until all lines have been deleted. Remember on a Gradient Program the first line values must be cleared and new values entered.

3. Press SAVE, then ENTER.

3.6.8 Locking the Keyboard

Locking the keyboard prevents accidental alteration of gradient flow conditions. Activating the keyboard lock locks the SET-UP page, the GRADIENT PROGRAM page, the GRADIENT page, and the TIMES EVENTS page and prevents call-up of the ISOCRATIC page. It disables the following keys:

- The Numerical Key Pad
- The Cursor Movement Keys
- The Blank Keys Under the Screen
- The Isocratic Page Key

NOTE

MAKE SURE THAT THE PROGRAM (GRADIENT AND TIMED EVENTS) ARE COMPLETE AND CORRECT BEFORE LOCKING THE KEYBOARD

NOTE

MULTIPLE INJECTIONS USING THE KEY UNDER RUN TO INITIATE GRADIENT EXECUTION ARE NOT POSSIBLE WHILE THE KEYBOARD IS LOCKED

To Lock the Keyboard:

1. Press SET-UP key.
2. Move the cursor to the KEYBOARD LOCK location.
3. Press 1, ENTER. The keyboard is now locked and the box in upper right corner of all locked pages will now be lit.

To Unlock the Keyboard:

1. Press the SET-UP key.
2. Press 0, ENTER.

3.6.9 Solvent Compression Factors — Maintaining Constant Flow Rates

Solvent compression values are used by the AGC in calculating pump output and reflect the percent increase in pump speed for accurate delivery at varying pressures.

To enter solvent compression values:

1. Calculate solvent compression factors for each pump with a particular solvent. (See Appendix B for information on how to calculate the solvent compression factors.)

2. Press the SET-UP page key.

3. Position the cursor and ENTER solvent compression values (0-40% in 0.1% increments).

   Entered values will now apply to **ALL** gradient and isocratic runs. To change values position the cursor, press CLEAR and ENTER new values.

4

SYSTEM DIAGNOSTICS

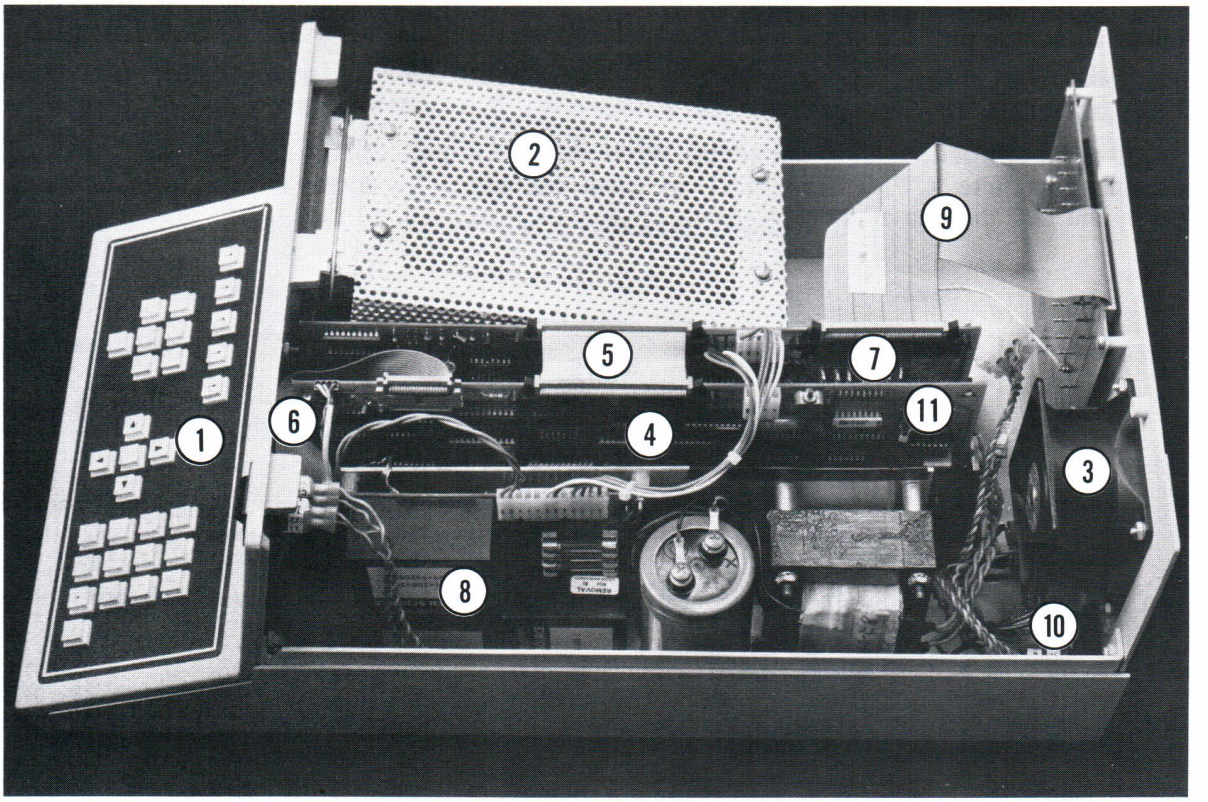


FIGURE 4-1 AGC MAJOR ASSEMBLIES

1. KEYBOARD (4200
2. CRT DISPLAY (74846)
3. FAN (98056)
4. CPU BOARD (42007)
5. CABLE, CPU-I/O (74076)
6. CABLE, KEYBOARD-CPU (42161)
7. I/O BOARD (42008)
8. POWER SUPPLY (42132)
9. CABLE, I/O-OUTPUT (42131)
10. POWER RECEPTACLE (97656)
11. SELF-DIAGNOSTIC LEDS

4.1 SELF-DIAGNOSTIC ROUTINES

The diagnostic LEDs (shown in Figure 4-1) indicate the following tests are being performed (the LED order below relates to Figure 4-1, left to right).

1. ROM Test (check sum test on CPU)
2. Not Used
3. Simple RAM Test (data check)
4. Simple RAM Test (address check)
5. Full RAM Test (indicator light in the upper right corner of the screen flashes while the test is in progress.)
6. Pump Test (pumps will be audibly activated)
7. Not Used
8. (blinks during Full RAM Test)

On power up, all LEDs are flashing. When the self-diagnostic test is passed, the logo appears on the screen and the LEDs will be off.

Diagnostic tests are performed in sequence. When each LED goes on, the test indicated is being performed. If the test halts, a "FAIL SELF TEST" message appears on the screen and the test that failed is indicated by the LED that remains on.

NOTE

VARIOUS SELF-DIAGNOSTIC TESTS CAN BE PERFORMED INITIATED BY KEYSTROKES AT POWER-UP AS EXPLAINED BELOW

NO KEYSTROKES — AGC IS SIMPLY SWITCHED ON:

The following tests are performed in order:

1. ROM Check
2. Simple RAM Test
3. Pump Check

TO PERFORM A FULL RAM TEST

Hold down the HOME and the 1 keys simultaneously while switching the power on and the following tests will be performed:

1. ROM Check
2. Simple RAM Test
3. Full RAM Test
4. Pump Check

TO INITIALIZE THE EEPROM

When new EEPROMs are installed or if the need should arise to fully clear the memory, hold down the HOME and the 7 keys simultaneously while switching the power on and the following tests will be performed:

1. ROM Check
2. Simple RAM Test
3. EEPROM Initialization
4. Pump Check

4.2 SYSTEM MESSAGES

The following messages appear on the AGC pages indicated in the subheadings to inform the operator of various operating/programming conditions. The appearance of a message may be for informational purposes and does not always require action on the operator's part.

TABLE 4-1 SYSTEM MESSAGES

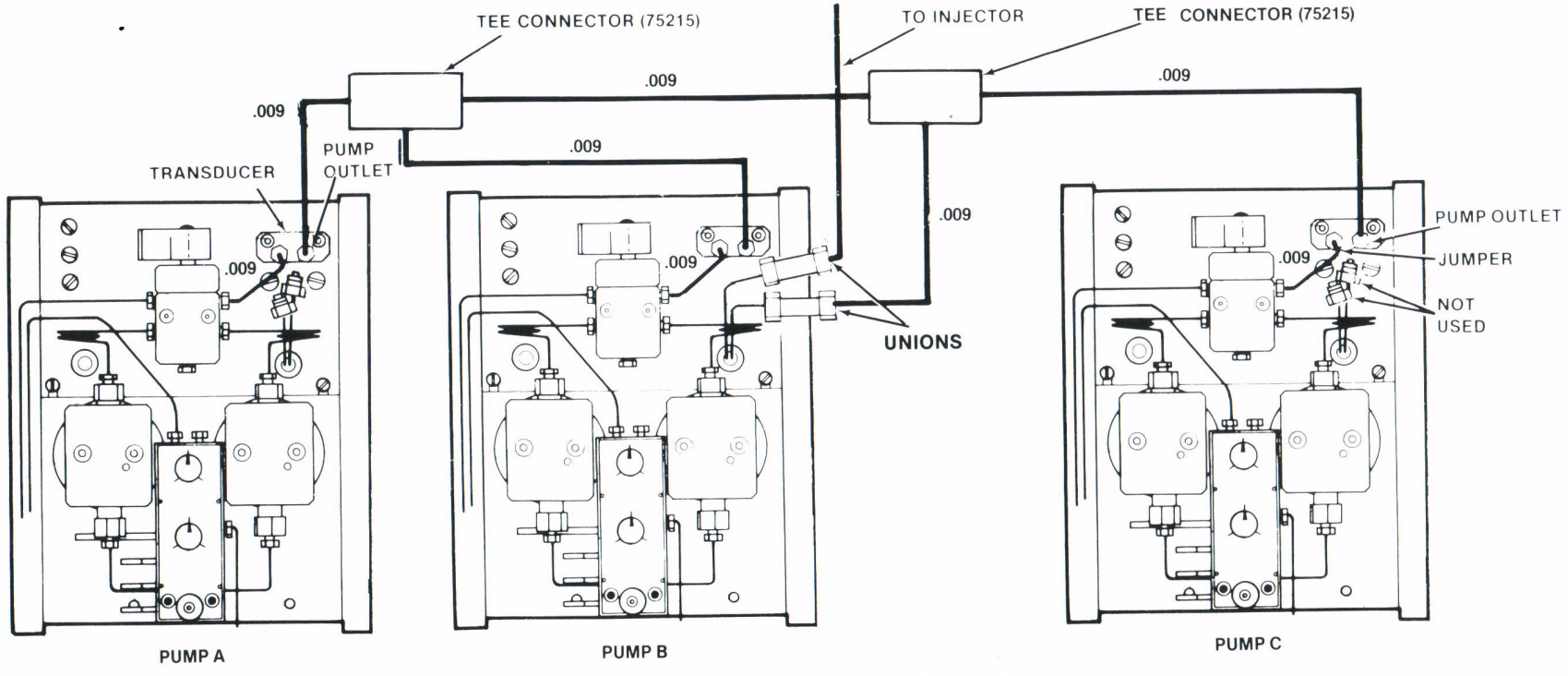
Diagnostic Message	Cause
Set Up Page	
Maximum high pressure limit is 6000 psi	Value entered >6000 Input range is 51-6000
Maximum low pressure limit is 5950 psi	Value entered>5950 Input range is 0-5950
High limit less than low limit	High limit value entered less than current low limit value. High limit must be greater than low limit to be valid.
Low limit greater than high limit	Low limit value entered greater than current high value. Low limit must be less than high limit to be valid.
Maximum compression factor is 40.0	Value entered >40.0 Input range is 0.0-40.0
Maximum chart output is 3	Value entered >3 Input range is 0, 1, 2 or 3
Minimum high pressure limit is 51 psi	Value entered <51 Input range is 51-6000
Isocratic Page	
Maximum flow rate value is 10 ml/min	Value entered >10.00 Input range is 0.00-10.00

TABLE 4-1 SYSTEM MESSAGES (CONT)

Diagnostic Message	Cause
Isocratic Page (Cont)	
Maximum %A, %B, %C is 100%	Value entered for %A, %B, or %C > 100. Input range for each % value is 0-100 (integers only).
%A + %B + %C > 100	%A, %B, %C values total more than 100%. Allowable condition. Acts as warning that the actual flow rate delivered will be less than the specified flow rate.
%A + %B + %C < 100	%A, %B, %C values total less than 100%. Allowable condition. Acts as warning that the actual flow rate delivered will be less than the specified flow rate.
High pressure shutdown	System pressure detected to exceed high limit for 2 seconds. To recover: re-enter flow conditions on isocratic page, or place AGC in gradient mode by selecting a gradient program on gradient page.
Low pressure shutdown	System pressure detected below low limit for 90 seconds. To recover: re-enter flow conditions on isocratic page or place AGC in gradient mode by selecting a gradient program on gradient page.
Pump abort	Contact closure signal detector from external device connected to abort terminal on rear panel. To recover: re-enter flow conditions on isocratic page, or place AGC in gradient mode by selecting a gradient program on gradient page.
Gradient Program Page	
Maximum program number is 10	Gradient program number > 10 entered. Input range is 1-10. Integers only.
Minimum program number is 1	0 was entered for gradient program number. Input range is 1-10. Integers only.
Maximum flow rate value is 10 ml/min	Value entered > 10.00. Input range is 0.00-10.00.
Maximum %A, %B, %C is 100%	%A, %B, or %C value entered > 100. Input range for % values is 0-100. Integers only.
Maximum time is 655.35 minutes	Value entered > 655.35. Input range is 0.01-655.35 minutes.
Minimum time value is 0.01 minutes	0 minutes entered for time value. Input range is 0.01-655.35 minutes.
Maximum curve # is 11	Value entered > 11. Input range is 1-11. Integers only.
%A + %B + %C < 100	%A, %B, %C values total more than 100. Allowable condition. See isocratic page.

TABLE 4-1 SYSTEM MESSAGES (CONT)

Diagnostic Message	Cause
Gradient Program Page (Cont)	
%A + %B + %C > 100	%A, %B, %C values total more than 100. Allowable condition. See isocratic page.
This line is incomplete	Up or down cursor movement attempted on incomplete line. All entries on line must be made before cursor can be moved to another line.
To use these new values, please save	Appears whenever the cursor leaves a line. AGC cannot use values entered until saved.
Deletion of initial line not allowed	Delete key pressed when on first line. Change initial conditions manually.
Enter program #	Save key was pressed. Prompts with program number under which displayed table is to be saved.
Replace with new program (0 = no, 1 = yes)	Clone procedure. Program under that number displayed. To save over it, answer yes or no to try again.
Timed Events Page	
Maximum program number is 10	Timed event program number entered > 10. Input range is 1-10. Integers only.
Minimum program number is 1	0 entered for timed event program number. Input range is 1-10. Integers only.
Maximum time is 655.35 minutes	Value entered > 655.35. Input range is 0.00-655.35.
Maximum interval is 655.35 minutes	Value entered > 655.35. Input range is 0.00-655.35.
Maximum switch is 8	Switch # > 8 entered. Input range is 1-8. Integers only.
Maximum action is 3	Value entered greater than 3. Input range is 0, 1, 2, or 3 only.
This line is incomplete	Up or down cursor movement attempted while present line is incomplete. Current line must be complete before it can be left.
To use these new values, please save	Appears whenever a line is left. See gradient program page.
Enter program #	Save key pressed. See gradient program page.
Replace with new program? (0 = no, 1 = yes)	See gradient program page.



NOTE: ALL TUBING SHOULD BE .009 IN, AND LESS THAN 15″ IN LENGTH OR AS SHORT AS POSSIBLE.

FIGURE 4-2 ALTERNATE PUMP CONNECTION METHOD

EXPLANATION PAGES
(GUIDE FOR THE OPERATIONS PAGES)

Page Guide Page

Set-Up Page

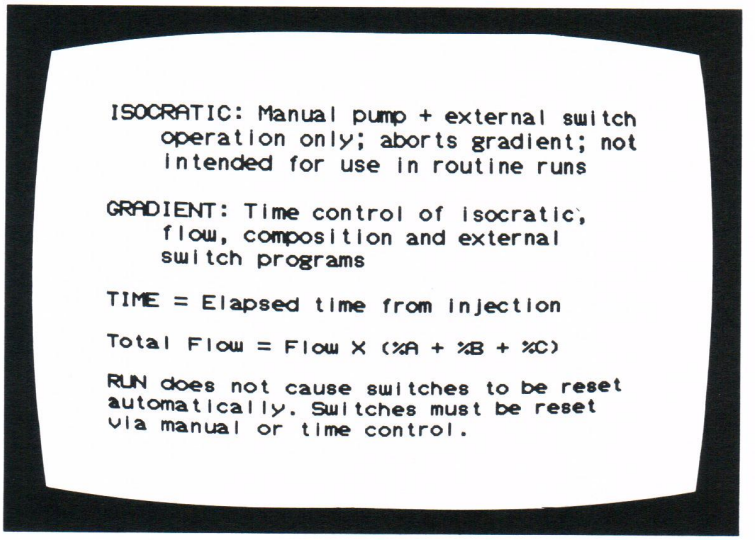
Isocratic Page

Gradient Program Page

Timed Events Page

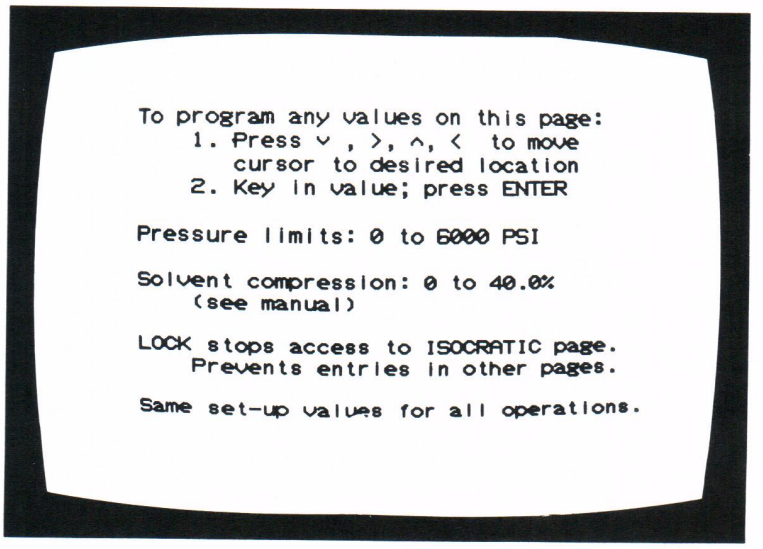
Gradient Operation Page (in gradient mode)

APPENDIX A



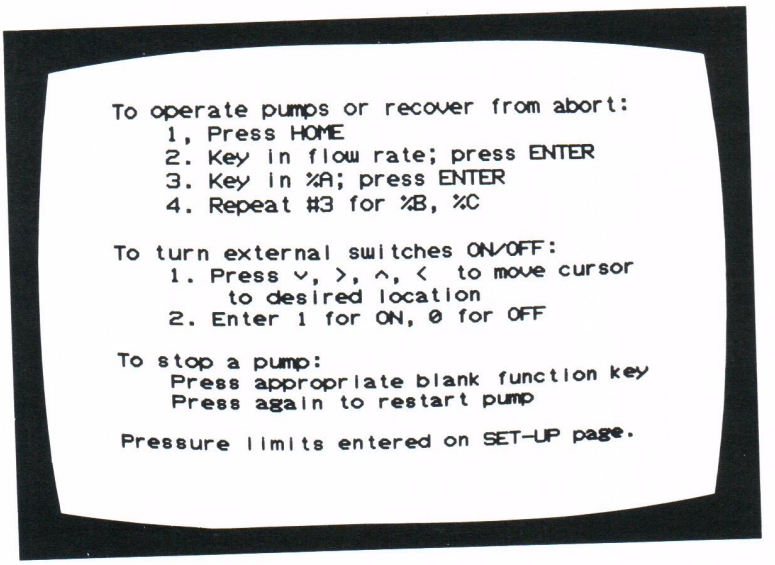
PAGE GUIDE

Refer to Section 3.1 for Page Guide and General Use Information.



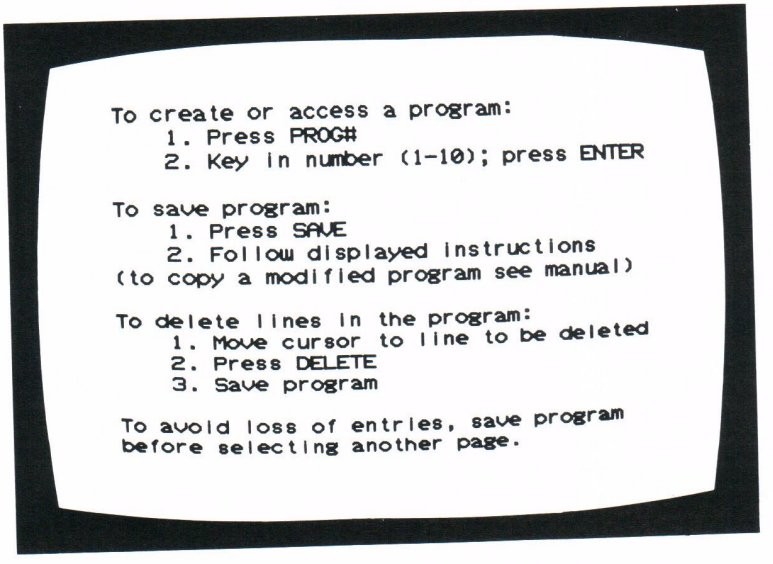
SET-UP PAGE

Refer to Section 3.2 for details on programming the Set- up page.



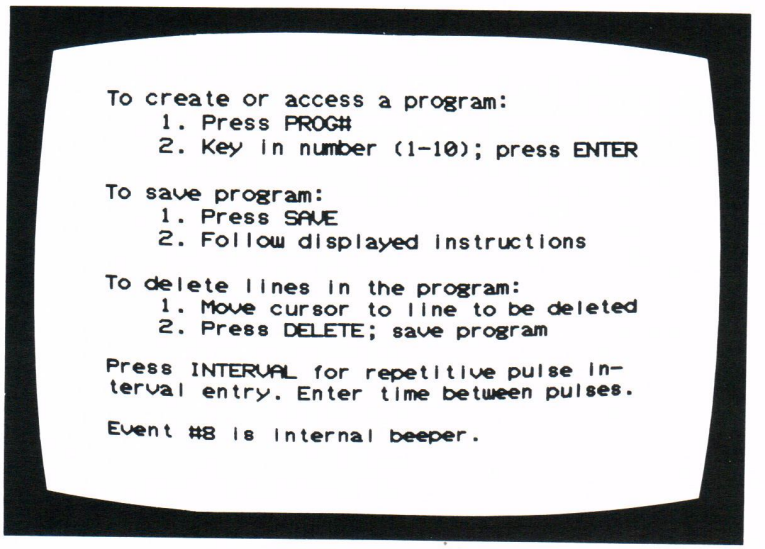
ISOCRATIC PAGE

Refer to Section 3.3 for details on Isocratic Operation.



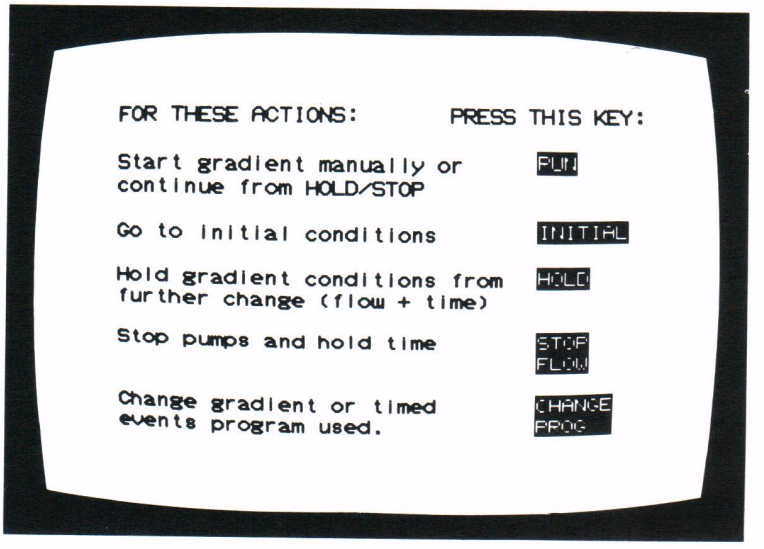
GRADIENT PROGRAM PAGE

Refer to Section 3.4.1 for details on programming a gradient.



TIMED EVENTS PAGE

Refer to Section 3.5 for details on programming the timed events.



GRADIENT OPERATION PAGE

Refer to Section 3.4.4 for details on each of these gradient control functions.

HOW TO CALCULATE SOLVENT COMPRESSION FACTORS

APPENDIX B

SOLVENT COMPRESSION VALUES FORCE THE PUMPS TO DELIVER A CONSTANT FLOW REGARDLESS OF PRESSURE CHANGES. THE SOLVENT COMPRESSION FACTOR IS DETERMINED FOR THE SOLVENT USED WITH EACH PUMP.

1. Press the ISOCRATIC key.
2. Turn on pump A.
3. Set flow rate at 2.0 ml/min. Set %A at 100.
4. Measure the column pressure.
5. Collect the eluent at the column outlet in a 10 ml graduated cylinder.
6. Measure the time (in minutes) it takes to collect 10 ml.
7. Determine the solvent compression factor for the solvent.

$$F = \left(\frac{\text{Time}}{\text{Volume/Flow Rate}} - 1\right) \times 100 \times \frac{6000}{\text{Pressure}}$$

For example, the compressibility factor is 15 for a solvent that takes 5.25 minutes to fill the flask at 2000 psi.

$$F = \left(\frac{5.25 \text{ min}}{10 \text{ ml}/2 \text{ ml/min}} - 1\right) \times 100 \times \frac{6000}{2000} = \left(\frac{5.25}{5.00} - 1\right) \times 300 = \frac{1}{20} \times 300 = 15$$

Repeat Steps 3 through 7 for each pump with its respective solvent.

Once the compressibility compensation factor is entered into the solvent table, the AGC determines the solvent delivery rate by the following calculation:

$$F = F_o\left(100 + f \times \frac{\text{Pressure}}{6000}\right)$$

Where:

F is the delivery rate.
F_0 is the delivery rate with no compressibility compensation.
f is the compressibility factor.
Pressure is the system pressure in psi.

For example, the delivery rate for the same solvent (compressibility factor 15) in a system operating at 3000 psi is:

$$F = F_o\left(100 + 15 \times \frac{3000}{6000}\right)$$

$$F = F_o(107.5)$$

Pump delivery is increased by 7.5 percent to compensate for solvent compressibility and to enhance flow rate accuracy.

HOW TO DEVELOP A GRADIENT PROGRAM

APPENDIX G

Gradient elution is a useful technique for improving resolution while shortening chromatographic analysis time. Solvents are mixed in varying proportions throughout the separation, and flow rate, analysis time and gradient curve are optimized to generate a chromatogram with well-defined peaks in a minimum period of time.

The most important variable is the composition of the solvent mixture. For example in a reverse phase separation, weak solvents (such as water) allow resolution of early eluting peaks and therefore, comprise the major percentage of the mixture early in the gradient run. Strong (less polar) solvents such as methanol have a greater ability to elute the sample from the column and are delivered in percentages that will hasten elution of later peaks but will not prevent resolution of early peaks.

Solvent selection is also important, and strong and weak solvents should be selected which will complement each other to initiate peak resolution immediately after void volume has been reached and to space peak elution over the course of a run.

Common problems in solvent selection are shown in Figure C-1.

Gradient programming methods development begins with establishing final gradient conditions for the solvent mixture. One hundred percent of strong solvent will generally elute the sample unresolved in the void volume. Establish final conditions by reducing (by approximation or trial and error) the strong solvent percentage until one or two of the final peaks resolve. The percentage of which this occurs will be the final condition.

Initial conditions should be determined to allow resolution of all significant early-eluting peaks. This can be approximated or determined isocratically.

The gradient curve is generally determined by beginning with the linear curve, No. 6, and altering it to improve resolution. Concave curves, Nos. 7-11, will improve the resolution of earlier eluting peaks while causing later eluting peaks to move in closer together. Convex curves, Nos. 1-5, improve later resolution and bring earlier eluting peaks in closer (see Figures C-4 and C-5).

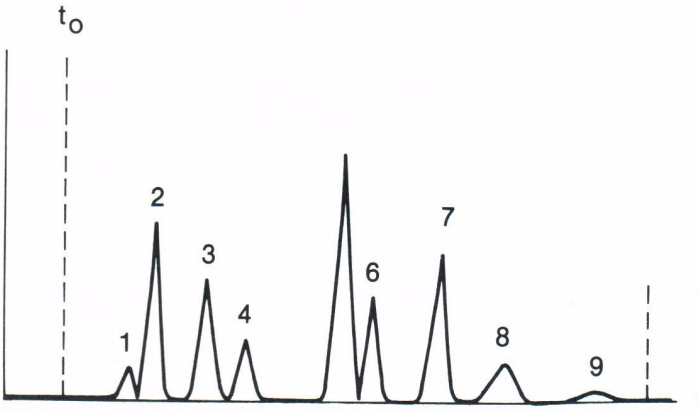
Flow rate may be adjusted to improve resolution. Many times, unlike isocratic separations, higher flow rates improve resolution in gradient operation and should, therefore, be set as high as possible without exceeding column backpressure limitations.

The duration of a run should generally be planned to allow about one minute per component in the sample.

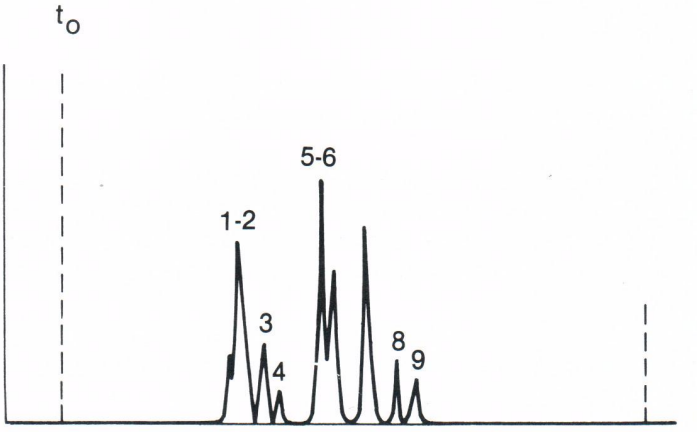
Three solvent gradients may often be desirable to obtain better separations using an intermediate polarity solvent between the strong and weak solvents used in binary gradient separations.

Typical problems in gradient programming and their solutions are summarized in Figure C-3.

A. Desired Results



B. Solvent strength is too great. Sample peaks are crowded together in the center (or some part) of the chromatogram, with resulting loss in resolution.



C. Solvent strengths are too close together. Gradient separation resembles isocratic elution. Initial bands are poorly resolved while later bands widen excessively and require a long separation time.

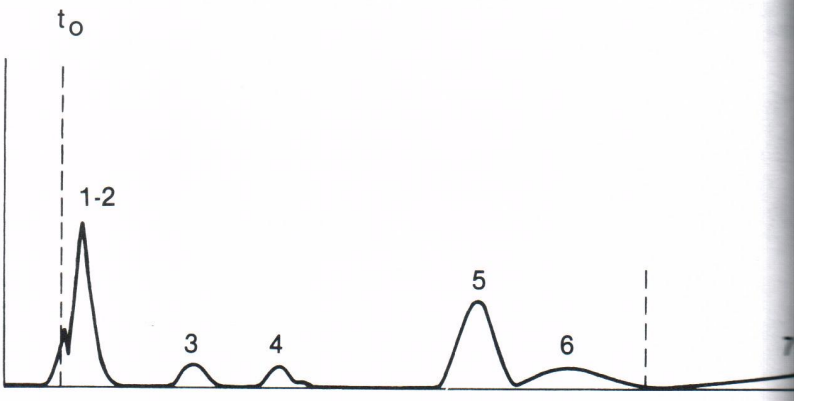
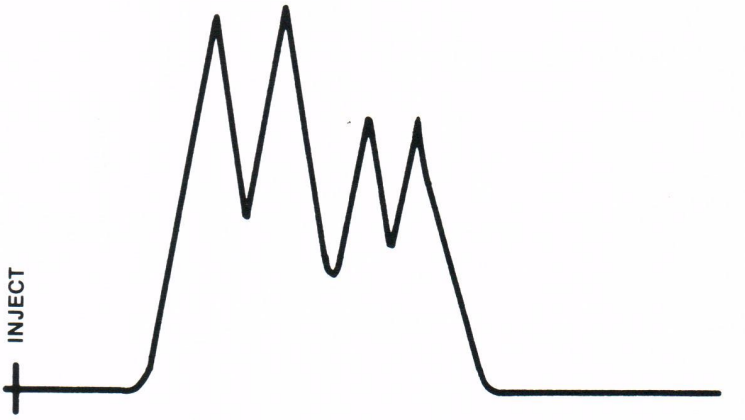


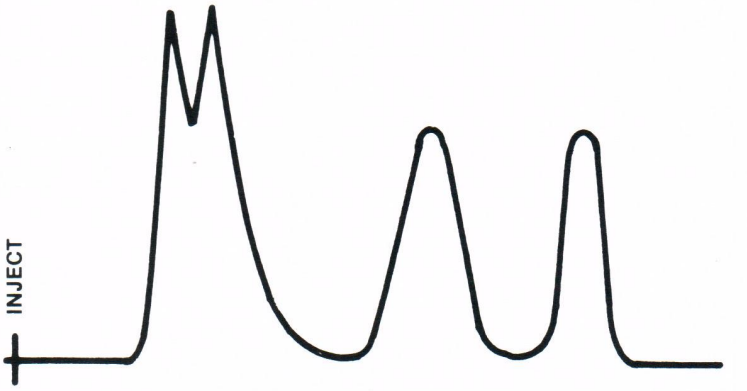
FIGURE C-1 SOLVENT SELECTION IN GRADIENT OPERATION

Every sample must first be run on a linear gradient. There are three and only three conditions of inadequate resolution in gradient elution.

1. Inadequate resolution over all.



2. Inadequate resolution at the beginning.



3. Inadequate resolution at the end.

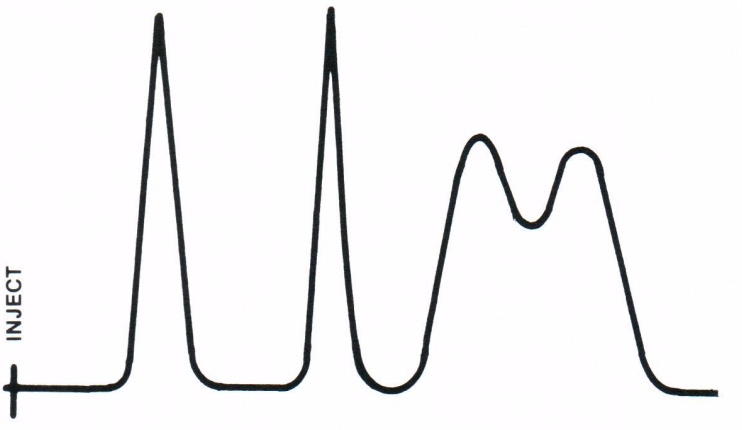
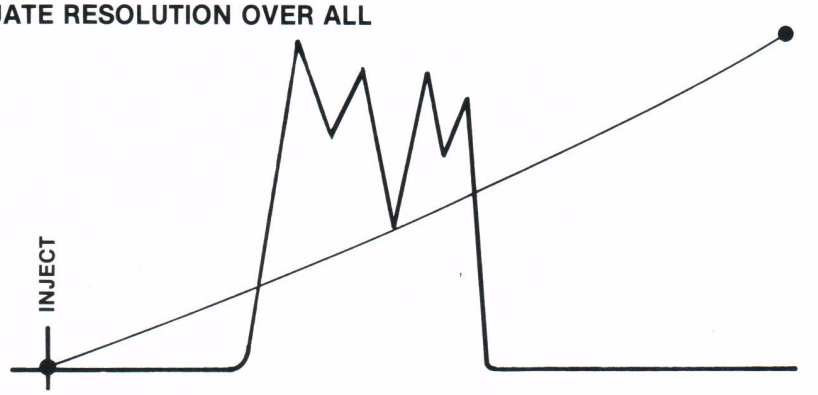


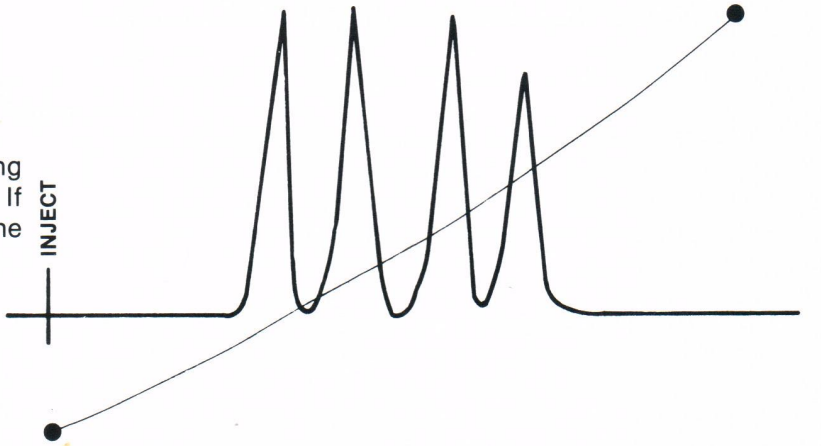
FIGURE C-2 RESOLUTION INADEQUACIES

INADEQUATE RESOLUTION OVER ALL

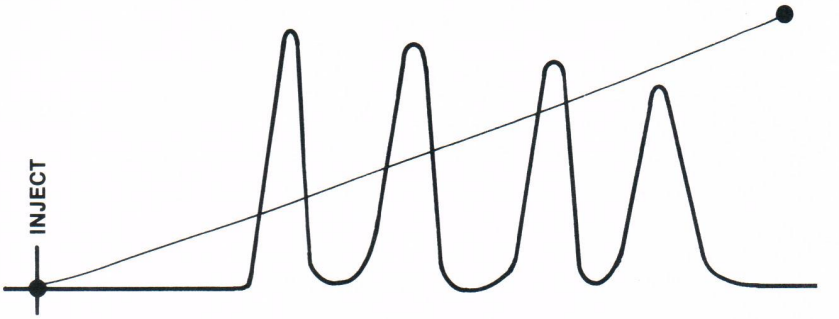
The choices are logical.



1. Decrease the initial conditions. If working in normal phase, decrease the polarity. If working in reverse phase, increase the polarity.



2. Increase the run time of the gradient.



3. Double the flow rate — use original time.

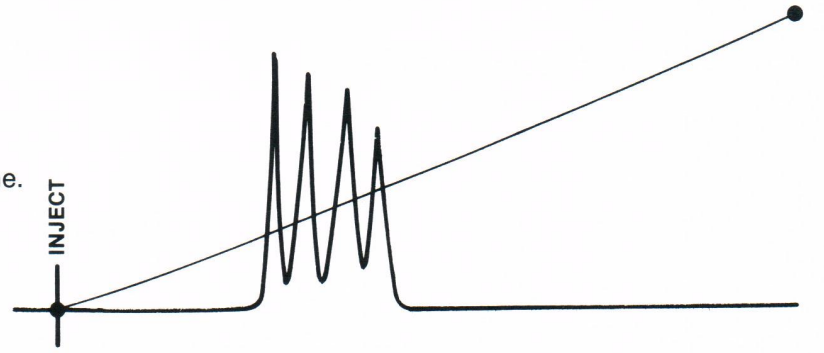


FIGURE C-3

INADEQUATE RESOLUTION AT THE BEGINNING

1. Decrease the initial solvent strength.

2. Change to concave curve. Delay the rate of change at the start and increase the rate toward the end.

3. As above and vary degree.

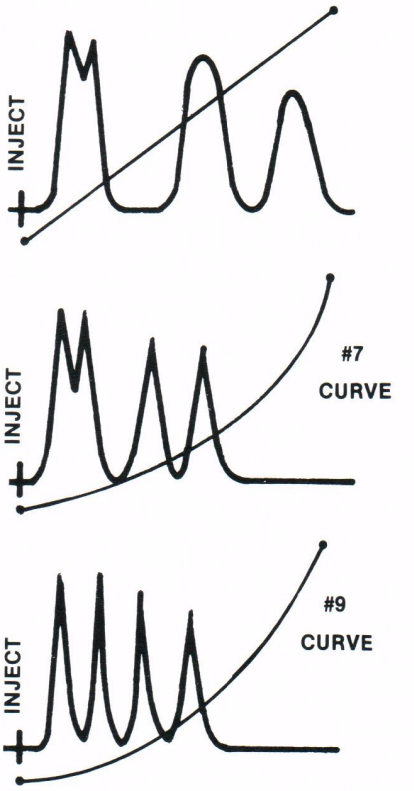


FIGURE C-4

INADEQUATE RESOLUTION AT THE END OF THE CHROMATOGRAM

1. Decrease the final strength of the solvent composition.

2. Change to convex curve. Increase the rate of change at the beginning and decrease the rate toward the end.

3. As above and vary degree.

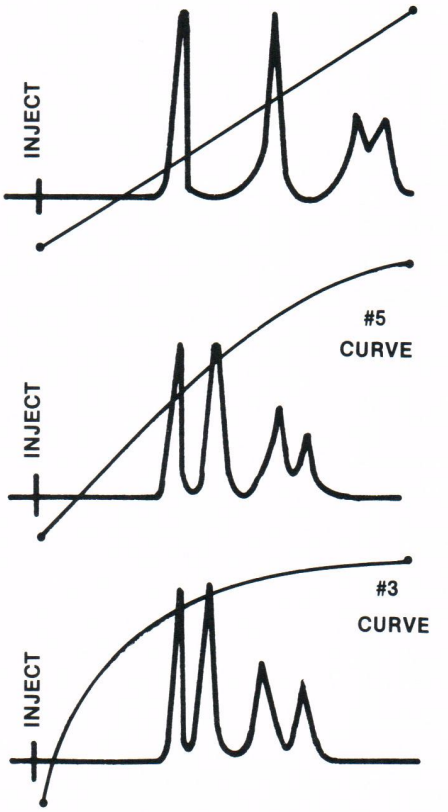


FIGURE C-5

SPECIFICATIONS

APPENDIX D

Operational

Flow	0 to 10 ml/min in 0.01 ml/min increments for each operational pump (0-30 ml/min total for three pumps)
Composition	0 to 100% in 1% increments for each of three components — A, B and C
Gradient Programs	10 storable programs, each with 10 segments. Each segment allows simultaneous changes in total flow and composition of A, B or C in accordance with any of 11 different curves.
Timed Event Programs	10 storable programs, each with 20 timed events. Capable of controlling 7 external devices and an audible beeper.
Program Memory Protection	Programs stored in memory retained indefinitely without line power or batteries. Stored programs may be modified or deleted by operator.
Low Pressure Limit	Settable from 0 to 5950 psi in 1 psi increments. Low pressure for more than 90 sec will cause pumps to stop.
High Pressure Limit	Settable from 51-6000 psi in 1 psi increments. High pressure for more than 2 sec will cause pumps to stop.
Compressibility Compensation	Settable from 0 to 40% in 0.1% increments for each of three components.

Electrical

Power Requirements	Maximum power — 80 VA
Voltage Select Switch	Nominal: 115 V, 230 V Allowable Voltage Range: 85 to 132 V, 187 to 264 V
Frequency	50 or 60 Hz (switch selectable on CPU board)
Fuse	Single side fused except in those countries requiring both sides fused. End caps provided for both 3 AG or 5 X 20 mm fuses. (115 V 1 A; 230 V 0.5 A)

Time or Operator Controllable Switch Closures	7 controllable switch closures to ground. TTL compatible terminals pulled with 10 K ohms to 5 V. May be pulled up to 12 V. Maximum allowable current — 1.0 A. Maximum allowable voltage — 12 V. Outputs may be controlled from keyboard directly or from timed event program. Two modes available from keyboard: 1-ON (switch closed to ground); 2-OFF (switch open, +5 V to ground). Two additional modes available from timed events program: 3-Pulse (single contact closure to ground for 100 millisec). 4-Repetitive Pulse (series of contact closures to ground) occurring at settable intervals from 0.01 to 655 minutes in 0.01 minute increments.
Power Supply	12 V to ground buffered supply, common to 4 terminals. 1A max current.
Hold Output	1 TTL compatible switch closure to ground. Maximum current sink: 100 mA. Low output indicates pumps stopped due to: a. exceeding high pressure limit b. below low pressure limit c. abort input d. "power up" condition Used to communicate with HOLD input on WISP BCD board to prevent further injections.
Chart Output	2 terminals (+,−) operator selectable to represent %A, %B, %C or pressure. 0 to 10 mV full scale = 0 to 100% or 0 to 6000 psi. Resolution = 0.1% of full scale.
Ground Terminals	Four terminals connected to signal ground used as reference for outputs, inputs or 12 V power supply (other than 10 mV output).

Environmental

Temperature	
Operating Range	5° to 40°C
Storage Range	−45° to 65°C
Humidity	
Operating Range	20% to 95% RH
Storage Range	0% to 100% RH (non-condensing)

Standards

Accepted for FDA Class L
Complies with FCC 80-148 emission regulations

Physical Dimensions

Height: 8 inches (20.3 cm)
Depth: 21 inches (53.3 cm)
Width: 11¼ inches (28.6 cm)

Weight: 22.9 pounds (10.4 kg)

WARRANTY/SERVICE INFORMATION

Waters Service Department Message Center
1-800-252-HPLC

APPENDIX E

_____ Serial No.

_____ Startup Date

LIMITED PRODUCT WARRANTY

Millipore Corporation, including its Waters Chromatography Division (Waters), provides this limited warranty (the Warranty) to protect customers from nonconformity in the product workmanship or materials. Except for WISP and ALC/GPC 150C, the Warranty covers all new products manufactured by Waters and its subsidiaries.

The Warranty is as follows:

Waters warrants that all products sold by them will be of good quality and workmanship. The products will be fit for their intended purpose(s) when used strictly in accordance with Waters instructions for use during the applicable warranty period.

The foregoing warranty is exclusive and in lieu of all other express and implied warranties, including but not limited to fitness for any other purpose(s). In no event will Waters be liable for consequential, economic or incidental damages of any nature. Waters reserves the right not to honor this warranty if the products are abused by the customer. The Warranty will not be deemed to have failed of its essential purpose so long as Waters is able and willing to repair or replace any non-conforming part or product.

Warranty Service

Warranty service will be performed at no charge and at Waters option in one of three ways: (1) a service representative will be dispatched to the customer's facility; (2) the product will be repaired at a Waters repair facility; or (3) replacement parts will be sent to the customer. If installation procedures are appropriate, they will be included with the replacement part.

Non-conforming parts or products will be repaired, replaced with new or like-new parts, or refunded in the amount of the purchase price, when the product is returned. Warranty service will be performed only if the customer notifies Waters during the applicable warranty period.

Unless otherwise agreed at the time of sale, warranty service will not be provided by dispatching a service representative when the equipment has been removed from the initial installation location to a new location outside the home country of the selling company.

Warranty service will not be performed upon:

a. Any product or part which has been repaired by others, improperly installed, altered, or damaged in any way.

b. Product or parts identified prior to sale as not manufactured by Waters. In such cases, the warranty of the original manufacturer will apply.

c. Products that malfunction because the customer has failed to perform maintenance, calibration checks, or observe good operating procedures.

Repair or replacement will not be made:

a. For expendable items such as filament devices, panel lights, fuses, batteries, and seals, if such items were operable at the time of initial use.

b. Because of decomposition due to chemical action.

c. For used equipment.

d. Because of poor facility, operating conditions or utilities.

Warranty Period

The Warranty Period begins when the product is installed or, in the case of a customer installation, fifteen days after shipment from Waters. In no case will the warranty period extend beyond 15 months from date of shipment. If an item is replaced during its warranty period, the replacement part will be warranted for the balance of the original warranty period.

The warranty period for the **MODEL 680 AGC** (free-standing and system components) is 12 months.

ORDERING INFORMATION

WHERE TO PLACE ORDERS

Mail Orders — Millipore Corporation, Waters Chromatography Division, 34 Maple St., Milford, MA 01757. Attn: Order Processing Dept.

Telephone Orders* — 1-800-252-HPLC Customer Sales Dept.

Telex Orders — 94-8413

Or contact your nearest Waters subsidiary or representative.

*Confirming orders mailed after a telephone order has been placed must be clearly marked "CONFIRMING" to avoid duplication.

HOW TO PLACE ORDERS

Normally, delays or errors in processing orders are caused by incorrect or incomplete information. To minimize delays and errors in processing your orders please provide all of the information requested below. Please list part numbers in ascending numerical order.

1. Catalog numbers and descriptions as given.
2. Quantity desired.
3. Complete purchase order number — orders cannot be processed without it. Requisition numbers are insufficient.
4. Complete "Ship To" address and marking if applicable.
5. Complete "Bill To" address if other than "Ship To."
6. Required delivery date.
7. Method of transportation desired.
8. Name and telephone number of person to contact if clarification is required.

PRICING

Prices listed are FOB Milford, MA, unless otherwise agreed. Prices and product information contained in any catalog or price list were current at the time of printing. In a continuing effort to provide the finest products available, Waters reserves the right to change specifications, models, or prices without notice and without liability for such changes. Where price changes have occurred, prices prevailing at time of receipt of your order will apply.

TERMS OF PAYMENT

Our terms are net 30 days from invoice date with approved credit. If your credit has not been previously established with Waters, our terms are payment in advance or COD.

SHIPMENTS-DAMAGES CLAIMS-RETURNS

SHIPMENTS

As all shipments are made FOB Milford, MA, we suggest insurance be authorized on all shipments. Instruments and major components will be packed and shipped via surface, unless otherwise requested. Supplies and/or replacement parts are packed and shipped via UPS, UPS Blue, air parcel post, or parcel post unless otherwise requested.

DAMAGED SHIPMENTS

The interstate Commerce Commission has held that carriers are as responsible for concealed damage as for visible damage in transit. Unpack shipment promptly after receipt as there may be concealed damage even though no evidence of it is apparent. When concealed damage is discovered, cease further unpacking of the unit involved and request immediate inspection by local agent or carrier and secure written report of his findings to support claim. This request must be made within 15 days of receipt. Otherwise, claim will not be honored by the carrier. Do not return damaged goods to factory without first securing an inspection report and contacting Waters for a return authorization number.

FILING OF CLAIMS

After a damage inspection report has been secured, Waters will cooperate fully in supplying replacements and handling of a claim which may be initiated by either party.

RETURNS

No returns may be made without prior notification and authorization. If for any reason it is necessary to return material to us, please contact our customer service department or your nearest Waters subsidiary/representative for a return authorization number and forwarding instructions.