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Teledyne Tekmar 7000

HEADSPACE Autosampler

User Manual

TELEDYNE
INSTRUMENTS
Tekmar
A Teledyne Technologies Company

14-4333-000_ Rev. B

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Tekmar
A Teledyne Technologies Company

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Contents

1 *Introduction*

1.1 Overview of the Section 1-1
1.2 Scope of the Manual 1-1
1.3 How the Manual is Organized 1-1
1.4 Assumptions 1-2
1.5 Conventions Used in the Manual 1-2
1.6 Figures Used in the Manual 1-3
1.7 Product Description 1-5
1.8 Product Applications 1-7

2 *Safety and Specifications*

2.1 Overview of the Section 2-1
2.2 Warnings 2-1
2.3 Specifications 2-4

3 *Description of Components*

3.1 Overview of the Instrument Components 3-1
Program Panel/Keypad 3-1
Rear Panel Area 3-4
Plumbing Assembly/Pneumatic Controls 3-6
7050 Carrousel 3-8
Platen Area 3-9
Electronics Card Cage 3-10

4 *Installing the 7000/7050*

4.1 Overview of the Section 4-1
4.2 Preliminary Procedures 4-1
Materials Needed for Installation 4-1
Power Requirements 4-3
Unpacking the Instrument 4-3
Inspecting the Instrument 4-3
Removing the Shipping Inserts 4-4
Placing the 7000 at Your Workstation 4-4
4.3 Pneumatic Connections 4-5
Venting the 7000 4-5
Choosing a Gas Supply 4-6
Connecting the Pressurization Gas Supply 4-7

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	Connecting the Carrier Gas Supply	4-8
	Connecting the Transfer Line	4-15
4.4	Electronic Connections	4-36
	Setting the DIP Switches	4-38
	Replacing the ROM	4-40
	Installing Accessory Boards	4-42
	TekLink™ Option	4-43
	RS232 Interface Option	4-43
4.5	Installing the 7050 Carrousel	4-44
4.6	Changing the Sample Loop and Sample Needle	4-46
4.7	Leak Checking the System	4-48
4.8	Checking the Vent	4-52

5 *Setting Pneumatics*

5.1	Overview of the Section	5-1
5.2	Pneumatic Controls and Gauges on the 7000	5-3
5.3	Principles of Operation - Column Carrier Flow	5-5
5.4	Principles of Operation - Vial Needle Flow	5-6
5.5	Principles of Operation - Static Vial Pressure	5-8
5.6	Setting Vial Pressurization	5-10
5.7	Setting Standby Vial Needle Flow Rate	5-11
5.8	Setting the Variable Injection Pressure Regulator	5-11

6 *Loading Vials and Inserts*

6.1	Overview of the Section	6-1
6.2	Preparing the 7000 for 9 ml and 12 ml Vials (with 7050)	6-1
6.3	Preparing the 7000 for 22 ml Vials (with 7050)	6-4
6.4	Preparing the 7000 for 9 ml and 12 ml Vials (without 7050)	6-6
6.5	Preparing the 7000 for 22 ml Vials (without 7050)	6-7
6.6	Preparing Vials for Sampling	6-8
6.7	Loading Vials into the 7000 (without 7050)	6-11
6.8	Loading Vials into the 7000 (with 7050)	6-13
6.9	Unloading Vials from the 7000 (without 7050)	6-14
6.10	Unloading Vials from the 7000 (with 7050)	6-15

7 *The Microprocessor*

7.1	The Microprocessor	7-1
7.2	Program Panel	7-2

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8 *Setting Parameters*

8.1	Overview of the Section	8-1
8.2	Constant Heat Time	8-1
8.3	Varying GC Cycle Times and GC Cycle Time Conflict Errors	8-4
8.4	Constant Heat Time and Parallel Sample Introduction - Two 7000s and One GC	8-5
8.5	Reviewing Method Parameter Values	8-6
8.6	Setting New Method Parameter Values	8-8
	Multiple Headspace Extraction	8-10
	Programming Vial Sizes	8-20
8.7	Copying Method Parameters	8-21
8.8	Method Optimization Mode™ (MOM)	8-22
	Tracking	8-27

9 *Developing Methods*

9.1	Overview of the Section	9-1
9.2	Fundamentals of Headspace	9-2
9.3	Injection Techniques	9-4
9.4	Quantitation of Headspace	9-8
9.5	Optimization of Headspace Sensitivity	9-10
9.6	Example Application Parameters	9-24

10 *Scheduling Methods/Aborting Runs*

10.1	Overview of the Section	10-1
10.2	Scheduling Methods (7050 only)	10-1
10.3	Aborting Runs/Priority Sampling	10-5
10.4	Running an Unscheduled Automated Method	10-6

11 *7000 Sequence of Operation*

11.1	Overview of the Section	11-1
11.2	Sequence of Operation - Summary	11-2
11.3	Sequence of Operation - Flowchart	11-19
11.4	Valve Output Chart	11-20

12 *Operating the 7000*

12.1	Overview of the Section	12-1
12.2	Steps Before Running a Method	12-2
12.3	Running Self Tests	12-5
12.4	Aborting Runs/Priority Sampling	12-8
12.5	Running an Unscheduled Automated Method	12-9

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13 *Failure and Error Screens*

13.1 Overview of the Section 13-1
13.2 Error: Vials in Platen 13-1
13.3 Power Fail During Cycle - Kept Memory 13-3
13.4 Power Fail - Memory Lost - Reset Clock 13-4
13.5 Error - GC Cycle Time Conflict 13-6
13.6 Failure - Thermocouple Open 13-7
13.7 Failure - Setpoint Not Reached 13-8
13.8 Fatal Failure - Platen Motor 13-9
13.9 Fatal Failure - Carrousel Motor 13-11
13.10 Fatal Failure - Vial Loader 13-12
13.11 Fatal Failure - Sample Loader 13-13
13.12 Fatal Failure - Vial Loader 13-14
13.13 Fatal Failure - Mixer Sensor 13-15
13.14 Fatal Failure - Carrousel Index 13-16
13.15 Fatal Failure - Platen (or Carrousel) Motor 13-17

14 *Routine Procedures and Maintenance*

14.1 Overview of the Section 14-1
14.2 Reviewing and Changing Instrument and Accessory Configurations 14-1
14.3 Changing the Viewing Angle of the Screen 14-2
14.4 Reviewing and Resetting the Clock 14-3
14.5 Locking the Keypad 14-4
14.6 Cleaning the 7000 14-6
14.7 Cleaning the 7050 14-6

15 *Troubleshooting*

15.1 Overview of the Section 15-1
15.2 Viewing the Sensor Screen 15-2
15.3 Using the Motor Test Mode Screen 15-3
15.4 Verifying Outputs 15-4
15.5 Troubleshooting Flow Charts 15-4
15.6 Expediting the Troubleshooting Process 15-18

16 *Service and Replacement Parts*

16.1 Contacting Tekmar Service 16-1
16.2 Replacement Parts and Accessories 16-2

Diagrams

Index

Glossary

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Essential Instructions

Please read this page before proceeding!

Tekmar designs, manufactures, and tests its products to meet many national and international standards. Because the 7000 Headspace Autosampler/7050 Carrousel is a sophisticated technical product, you must properly install, use, and maintain the instrument to ensure that they continue to operate within their normal specifications. Also, you must adhere to and integrate the following instructions into your safety program when installing, using, and maintaining these Tekmar products. Failure to follow the proper instructions may invalidate the warranty.

- Read all instructions prior to installing, operating, and servicing the product. Follow all warnings, cautions, and instructions marked on and supplied with the product and in this manual. If you do not understand any of the instructions, contact your Tekmar representative for clarification.

- Educate your personnel in the proper installation, operation, and maintenance of the product -- only qualified personnel should install, operate, update, program, and maintain the product.

- Install your equipment as specified in the installation section of this manual and according to applicable local and national codes. Connect all products to the proper electrical and pressure sources.

- When replacement parts are required, ensure that qualified individuals use replacement parts specified by Tekmar. Unauthorized parts and procedures can affect the product's performance and jeopardize safety. Using look-alike substitutions may result in fire, electrical hazards, or improper operation.

- Ensure that all equipment doors are closed and protective covers are in place -- except when maintenance is being performed by qualified persons -- to prevent electrical shock and personal injury.

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1.1 Overview of the Section

This section of the 7000/7050 User Manual explains the manual and its organization. It also lists the figures used throughout the document and includes a description of the 7000/7050 and a summary of its applications.

1.2 Scope of the Manual

This manual describes the Tekmar™ 7000 Headspace Autosampler and 7050 Carrousel and tells you how to:

- Install the instrument
- Set optimum pneumatics
- Develop methods
- Schedule methods
- Load vials
- Operate the instrument from startup to shutdown
- Perform routine maintenance and troubleshooting procedures
- Order replacement parts and service support

1.3 How the Manual is Organized

The manual is organized into 19 main sections.

Section	*Topics*
Section 1 -	**Introduction** is an overview of the manual organization. It describes the autosampler, its operation and applications.
Section 2 -	**Safety Information and Specifications** warns of possible safety hazards and lists product specifications.
Section 3 -	**Description of Components** briefly describes major components of the instrument.
Section 4 -	**Installing the 7000/7050** includes such procedures as unpacking, pneumatic/electronic connections, leak checking.
Section 5 -	**Setting Pneumatics** explains how to determine static vial pressure, set flow rates, and other related topics.
Section 6 -	**Loading Vials and Inserts** tells you how to prepare and load vials into the 7050 carrousel or the 7000 platen.
Section 7 -	**The Microprocessor** explains the program panel and keypad.
Section 8 -	**Setting Parameters** tells you how to run self tests, review, change, and copy parameter values, and perform other tasks.
Section 9 -	**Developing Methods** explains the steps -- and your options -- in developing methods for the 7000/7050.

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Section	Topics
Section 10 -	**Scheduling Methods/Aborting Runs** discusses scheduling methods, and unscheduled automated runs.
Section 11 -	**Sequence of Operation** describes the 7000 program steps.
Section 12 -	**Operating the 7000** has step-by-step instructions for running the instrument.
Section 13 -	**Failure and Error Screens** describes the errors you may encounter when operating the instrument.
Section 14 -	**Routine Procedures and Maintenance** covers cleaning, instrument adjustments, and leak checking.
Section 15 -	**Troubleshooting** lists potential problems and solutions.
Section 16 -	**Service and Replacement Parts** tells you how to contact Tekmar for service support and replacement parts.
Section 17 -	**Diagrams** includes wiring and flow diagrams.
Section 18 -	**Index** is a listing of key topics and their location.
Section 19 -	**Glossary** provides a definition of technical terms.

1.4 Assumptions

The manual assumes that you are familiar with gas chromatography technology and somewhat familiar with static headspace technology.

1.5 Conventions Used in the Manual

To help you locate and interpret information more easily, the manual uses certain typefaces and symbols with specific meanings, including:

DANGER

This symbol alerts you to a situation where incorrect operation of the 7000 could result in electrical shock or other serious hazard and cause severe and permanent personal injury as well as damage to your equipment.

WARNING

This symbol points out a situation where incorrect operation could result in personal injury and equipment damage.

CAUTION

This symbol indicates that incorrect operation could lead to equipment damage.

Note:

The "notes" throughout the text point out very useful information.

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1.6 Figures Used in the Manual

The following figures are included in the manual:

Figure #	*Description*	*Page #*
1-1	7000 Headspace Autosampler w/ 7050 Carrousel	1-6
3-1	7000/7050 Components	3-1
3-2	7000 Keypad	3-3
3-3	7000/7050 Rear View	3-5
3-4	Valve Oven and Plumbing Assembly	3-6
3-5	Valve Oven	3-7
3-6	Pneumatic Controls	3-7
3-7	7050 Carrousel	3-8
3-8	Platen Area	3-9
3-9	Electronics Card Cage	3-11
4-1	Location of Platen Shipping Inserts	4-4
4-2	Typical Pressurization Gas Connection	4-7
4-3	Carrier Gas Connection	4-9
4-4	Flow Element Location	4-10
4-5	Carrier Gas Connection	4-12
4-6	GC-Supplied Carrier Gas Connections	4-14
4-7	Septum Needle Adapter	4-22
4-8	Low Volume Injector Assembly	4-28
4-9	Cryofocusing Module	4-31
4-10	VIPR Connections	4-34
4-11	VIPR Connections	4-35
4-12	VIPR Back View	4-35
4-13	VIPR Front View	4-35
4-14	7000 Interface Board	4-37
4-15	DIP Switch Settings	4-39
4-16	ZIF Socket/ROM Chip Orientation	4-41
4-17	7050 Carrousel Installation	4-45
4-18	Valve Oven Area	4-46
5-1	7000 Flow Diagram	5-2
5-2	Pneumatic Controls and Gauges	5-3
5-3	7000 Pneumatic Controls	5-4
5-4	Valve Configuration of the 7000 in Standby Mode	5-6
5-5	Vial Needle Flow Rate and Vial Pressure	5-7
5-6	7000 Flow in Vial Pressurization Mode	5-9
6-1	Carrousel Collar Location	6-1, 6-4
6-2	Platen Chambers	6-2
6-3	Properly Sealed Vial	6-8
6-4	Undercrimped Vial	6-8

continued

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Figure #	*Description*	*Page #*
6-5	Overcrimped Vial	6-9
6-6	Hand Crimper and Vial	6-10
7-1	7000 Keypad	7-2
7-2	7000 Numbered Keypad	7-3
9-1	Full Evaporation Technique	9-5
9-2	Parameter Optimization Kit	9-7
9-3	Equilibration Curve	9-12
9-4	Non-Equilibrating Sample	9-12
9-5	Parameter Worksheet	9-21
11-1	7000 Flow Diagram	11-2
11-2	7000 Screen and Flow in Standby Mode	11-4
11-3	7000 Screen and Flow in Sample Equilibrate Mode	11-7
11-4	Equilibration Curve	11-8
11-5	Non-Equilibrating Sample	11-8
11-6	Screens and Flow in Mixing Mode	11-9
11-7	Screen and Flow in Stabilize Mode	11-10
11-8	Screen and Flow in Pressurize Mode	11-11
11-9	Screen and Flow in Pressure Equilibrate Time Mode	11-12
11-10	Screen and Flow in Loop Fill Mode	11-13
11-11	Screen and Flow in Loop Equilibrate Time Mode	11-14
11-12	Flow in Inject Mode	11-17
11-13	Screen and Flow in Re-Equil Mode	11-18
17-1	7000 Sampling Sequence	17-1
17-2	7000 Wiring Diagram Page 1	17-2
17-3	7000 Wiring Diagram Page 2	17-3
17-4	7000 Flow Diagram	17-4
17-5	7000 Valve Plumbing Assembly Page 1	17-5
17-6	7000 Valve Plumbing Assembly Page 2	17-6

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1.7 Product Description

The Tekmar™ 7000 Headspace Autosampler is a microprocessor-controlled automatic sampler that is designed to improve sensitivity/ precision in gas chromatographic or GC/spectrometry analysis of volatile compounds in a wide range of samples.

The 7000 is built upon the "static" headspace (as compared to the "dynamic" purge and trap) sampling technique. The 7000 removes volatile compounds from a non-volatile matrix and injects them into the gas chromatograph in vapor form.

In general terms, the instrument works like this:

- The sealed vial, containing the sample, is lowered into the electrically-heated sample platen. The platen can hold and heat up to 12 vials simultaneously.
- During the heating time, the volatile compounds in the sample partition into the vial's atmosphere, known as the "headspace."
- When the platen has reached the temperature setpoint -- and the rate of analytes going into the gas phase is equal to the rate of analytes returning -- the system reaches a state of equilibrium.
- The vial is pressurized with gas to a set value.
- The gas in the sample vial exits through the sample loop to the atmosphere, as the loop is filled with sample vapor.
- At equilibrium, the 7000 extracts the compounds (in vapor form) from the headspace of the vial, then injects them through the sample transfer line into the gas chromatograph for analysis. During injection, there is precise control of sampling pressure, volume, and temperature, which greatly increases quantitation -- and therefore reliability -- of the sampling run.

With the Tekmar™ 7050 Carrousel installed, the 7000 can automatically control the sample sequencing of up to 50 vials. These samples can be programmed to run under "Constant Heat Time™", a feature that provides equal thermal exposure to each sample. Constant Heat Time reduces overall analysis time and increases the number of samples that can be run, improving lab productivity.

The 7000's screen displays menus that allow you to choose from various options for scheduling and editing methods, setting parameters, testing internal components, etc.

continued

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The optional TekLink™ software/hardware package connects the 7000 to a PC and allows for complete remote control of the GC, data collection system, and the 7000. TekLink operates with Microsoft® Windows™.

Additional standard features such as Method Optimization Mode™ allow you to easily increment the optimal time, temperature, and mix power settings for a given sampling run.

The 7000's "valve and loop" sampling design offers increased precision and reproducibility of injection volume. The exclusive PV/T Injector™ in the 7000 valve oven ensures precise control of injection **pressure**, **volume** and **temperature**.

Carryover is kept to a minimum because the 7000 automatically performs a carrier-gas sweep to clean all sample lines between injections. Sample loops are interchangeable; the 7000 can accommodate stainless steel, E-Form, or Polar option sample loops in various sizes.

Flow elements are also interchangeable, giving you four different ranges from one flow controller and the ability to set the optimal flow rate best suited to your specific column type and size.

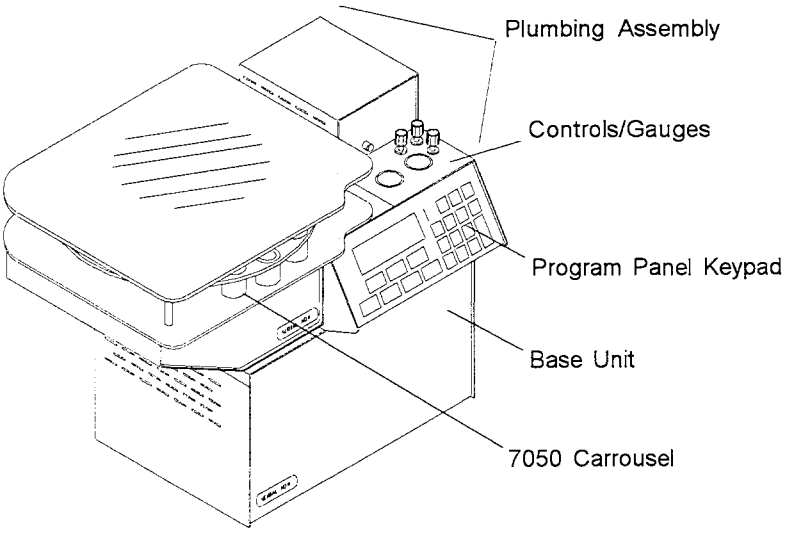


Figure 1-1 Tekmar™ 7000 Headspace Autosampler with 7050 Carrousel

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1.8 Product Applications

The Tekmar™ 7000 Headspace Autosampler/7050 Carrousel system retains sensitivity and precision in the analysis of volatile components.

The 7000's primary applications are:

- **Environmental screening of soils, water, and solid wastes**
  The 7000 is ideal for screening and direct analysis of priority pollutants and BTEX samples.

- **Blood alcohol and forensic medicine**
  The 7000 ensures accurate identification and quantitation of levels of ethanol, acetone, and other toxins in blood and other biological samples.

- **Monomers in polymers and residuals in food packaging materials**
  Residuals in packing materials such as ethane, propane, and propylene can be easily and accurately quantitated.

- **Residual solvents in pharamaceutical tablets and capsules**
  The 7000/GC process allows for routine monitoring of volatile organic impurities in bulk pharmaceuticals and excipients.

- **Flavors in foods and beverages**
  Flavor profiling determines the composition and taste of fruit, wine, spices, coffee, soda, cheese, beer, and other foods and beverages.

The static headspace technique around which the 7000 is designed improves sensitivity of the lighter compounds including HCN, SO_2, butadiene, as compared to analysis of these compounds by liquid injection. Static headspace technology is also ideal for semi-volatile organics such as pyridine, di- and tri-chlorobenzenes, etc.

Through precise positioning and control of the heating process (as well as through proper insulation), the 7000 maintains accuracy in sample temperature within 0.1°C between samples.

Several of the features built into the 7000, including Method Optimization Mode™, Optimix™ Equilibration, Multiple Headspace Extraction, Re-Equilibration, Constant Heat Time, and Full Evaporation Technique, also help ensure optimal analytical results. All of these features are discussed in more detail in later sections of this instruction manual.

If you have questions about the 7000/7050 as it relates to your particular application, please call Tekmar at (800) 543-4461 to speak with an Applications Chemist.

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2.1 Overview of the Section

2.2 Warnings

The first three pages of this section describe safety and operation issues to be aware of before operating the 7000/7050. The last part of Section 2 covers instrument specifications.

⚠ DANGER



Never connect a flammable gas (such as hydrogen) to the pressurize bulkhead fitting on the rear of the 7000. Venting of this gas in the Standby mode creates an explosion or fire hazard.

⚠ WARNING



Electrical shock hazard inside this instrument. Unplug power cord before servicing.

⚠ WARNING



It is recommended that safety glasses be worn at all times when operating the 7000. Heated sample vials are under pressure during normal operations. An inadvertent over-pressurization could cause a vial to burst.

⚠ WARNING



This instrument contains heating elements. Touching any heated zone during operation could cause a burn. (The system's heaters will come on anytime their setpoints are above ambient temperature.) When operating the 7000, keep all instrument panels fastened.

⚠ WARNING



The dust cover shipped with the 7050 (50-Position Carrousel) must be installed before operating the system. The cover protects the carrousel and also protects the operator by providing a safety shield in the event that an overpressurized vial would burst.

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WARNING

Be alert for environmental, shock, or other hazards in the event that a vial would break inside the instrument. Before cleaning up, unplug the instrument and determine the nature of the sample that has spilled. Use extreme caution and apply the appropriate clean-up measures.

WARNING



Always use the insert removal tool (Tekmar p/n 14-4365-027) when removing inserts from the platen. The inserts are at the same temperature as the platen heater and may cause a burn if handled improperly.

WARNING



Use extreme care when handling hot vials, particularly when unloading vials from the 7000 without the 7050. Improper handling of the hot vials may cause a burn.

WARNING



When the 7050 carrousel is indexing, do not place hands between the carrousel and the valve oven area.

WARNING



Do not over-pressurize or over-heat samples during operation. Extreme static pressure build-up in a vial due to excess pressure levels or heat settings could cause a vial to burst.

WARNING



When accessing the valve oven area, be certain that the valve is cool before opening the cover.

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⚠ CAUTION

The 6-port valve will fail at elevated temperatures. When using the E-slider option, do not exceed a valve temperature of 200°C with E-Form or Polar option 7000s.

⚠ CAUTION

When changing from 9 or 12 to 22 ml vials, remove the sleeves from the 12 platen chambers. Operating the instrument with a sleeve in any chamber may damage the unit.

⚠ CAUTION

Use the appropriate mixer setting. A setting that is too high can cause sample to splash onto the septum, which may lead to system contamination. Always start with the lowest mixer setting when developing your method.

⚠ CAUTION

Turn the 7000 power off before connecting or disconnecting the 7000/7050 cable to prevent instrument damage.

⚠ CAUTION

Modifications to this unit not expressly approved by the party responsible for compliance could void the manufacturer's warranty.

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to Part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy, and if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference, in which case users will be required to correct the interference at their own expense.

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2.3 Specifications

7000 Headspace Autosampler

Capacity and Vial Size:	12 vials, simultaneously heated 9, 12, or 22 ml (removable sleeve inserts required to use 9 and 12 ml vials)
Sample Loops:	Interchangeable 0.1, 0.25, 0.5, 1, 1.25, 2, 3, and 5 ml stainless steel, E-Form or Polar option loops
Sample Platen:	Oil-free, resistance heated, jacketed, variable temperature (ambient +15°C to +200°C), programmable in 1°C increments, sample uniformity ±0.1°C position to position
Valving:	Motor-actuated 6-port valve (ambient +15°C to +250°C); two solenoid-operated 2-port valves for sample pressurize and vent
Sample Path:	Teflon-free 1/16" nickel, heated transfer line, variable temperature (ambient +15°C to +250°C), stainless steel sample loops or optional E-Form or Polar option path
Sample Mixing:	Optimix™ Equilibration System, programmable 0.1 to 999.9 minutes, power programmable from 1 to 10
Modes of Operation:	**Manual:** Single sample **Standard:** Multiple samples, minimum time for equilibrium **CHT:** Constant Heat Time, identical thermal exposure time for each sample (requires 7050) **MHE:** Multiple Headspace Extraction, up to nine extractions per sample (with or without multiple septum puncture); standard or concentrate mode **MOM:** Method Optimization Mode™ (MOM) Successive samples receive incremental changes in method parameter setpoints for time, temperature or mixing power (requires 7050)
Electronic Control:	Parameter entry via a tactile response panel Baud Rate: 1200
Display:	240 x 64 pixel graphics LCD screen
Size:	20" W x 22" H x 13" D
Weight:	79 lbs.
Utilities:	115V, 50/60 Hz, 8A; 230V, 100V optional

continued

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7050 Carrousel

Capacity:	50 Vials
Vial Size:	9, 12, or 22 ml (removable inserts and carrousel collar required to run 9 and 12 ml vials)
Not heated:	All vials held at ambient temperature
Electronic Control:	25-pin I/O cable, parameter input through the 7000 microprocessor
Size:	20" W x 9" H x 18" D
Weight:	30 lbs. (shipped with protective cover)

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3.1 Overview of the Instrument Components

The following major components of the 7000/7050 are described and, in some cases, illustrated in this section of the manual:

- Program panel/keypad
- Rear panel
- Plumbing assembly area
- 7050 Carrousel
- Platen area
- Electronics card cage

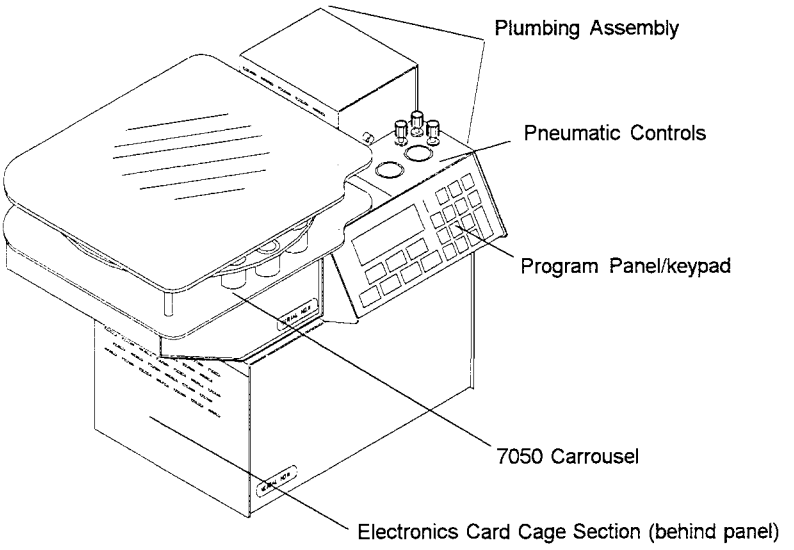


Figure 3-1 7000/7050 Components

Program Panel/ Keypad

The 7000 program panel area on the front of the 7000 has:

- An 8-bit microprocessor with 64K of program ROM, 2K of RAM
- A membrane keypad to modify program parameter values
- A 240 x 64 dot matrix LCD that displays the modes of operation

The controller uses a 6303 eight-bit CMOS-type microprocessor to manage operation of the various functions of the 7000/7050. Instructions for the microprocessor are stored in ROM. Program parameters can be reviewed and changed. Battery back-up saves changed parameters in the event of a power failure.

The program panel controls and/or displays the following:

- Method parameters
- Time/date
- Accessory configurations
- Special function screens
- Timing of valves
- Electronic outputs
- Viewing angle of the screen
- Error signals
- Failure signals

continued

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Here is a description of the function keys:

- Keys **F1**, **F2**, **F3**, **F4** correspond directly to commands displayed on the bottom line of the **LCD** screen. These commands differ for each program step (for example, **F1** may mean "Help", "Lock", "On", etc., depending upon where you are in the program).

- **AUTO** causes the 7000 to proceed through its run automatically.

- **HOLD** pauses the 7000 in a particular mode of operation. When **HOLD** is pressed, the 7000 remains in its present mode of operation, and stays there until you press either the **STEP** key or the **AUTO** key.

- **STEP** moves the system to the next mode of operation. It also loads vials into and out of the 7000 platen.

- **LOAD/UNLOAD** is used to manually load and unload vials into the platen.

- **START** signals the 7000 to proceed from the "Ready" mode to begin a run.

The numbered keypad serves two functions:

1) To let you enter time, temperature, and mix power values when developing methods. Keys **Y/7** and **N/9** are used to enter "Yes" and "No", respectively, for parameter **ON/OFF** commands.

2) To let you access diagnostic screens. These include troubleshooting, pneumatic, and electronic output setting screens.

- **Copy (C)** enables you to copy parameters from one method to another.

- **<- (BACKSPACE)** removes a parameter value that appears in a high-lighted box on the screen. You can move the highlighted box to a value that you want to change by pressing **F2 (<-)** or **F3 (->)**.

- The **PAGE UP** and **PAGE DOWN** keys serve two separate functions:

  1) To change the viewing angle of the screen.

  2) To view parameter listings (e.g., Method Parameters screen) when the screen prompt "**<PAGE UP/PAGE DOWN for more>**" appears.

continued

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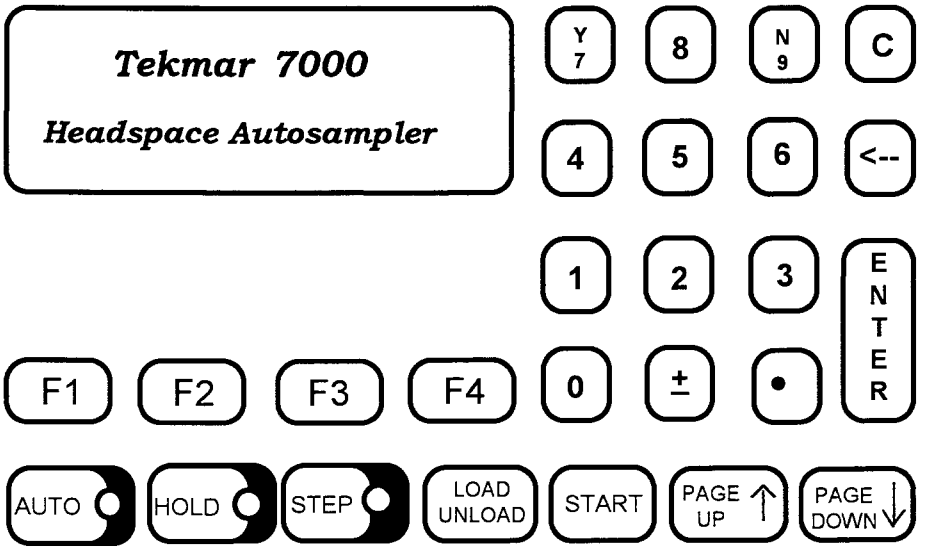


Figure 3-2 7000 Keypad

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Rear Panel Area

The 7000/7050 rear panel is where all external connections are made to the instrument. The rear panel area has the following components:

- **Carrier gas inlet**
- **Pressurize gas inlet**
- **Vent outlet**
- **Heated transfer line** to the GC
- **Power entry module** containing two fuses: 8 amp fuses for the 110V unit and 4 amp fuses for the 220V unit
- **Cooling fans**: one for pulling ambient air into the instrument, one for pulling warmer air out of the instrument, and one for circulating ambient air around the platen assembly
- **GC I/O port** for communication (electrical handshaking) between the 7000 and the GC with cable
- **Carrousel (7050) port** for connecting the 7000 to the 7050 with cable
- **RS232 Serial Connection** for software cable control

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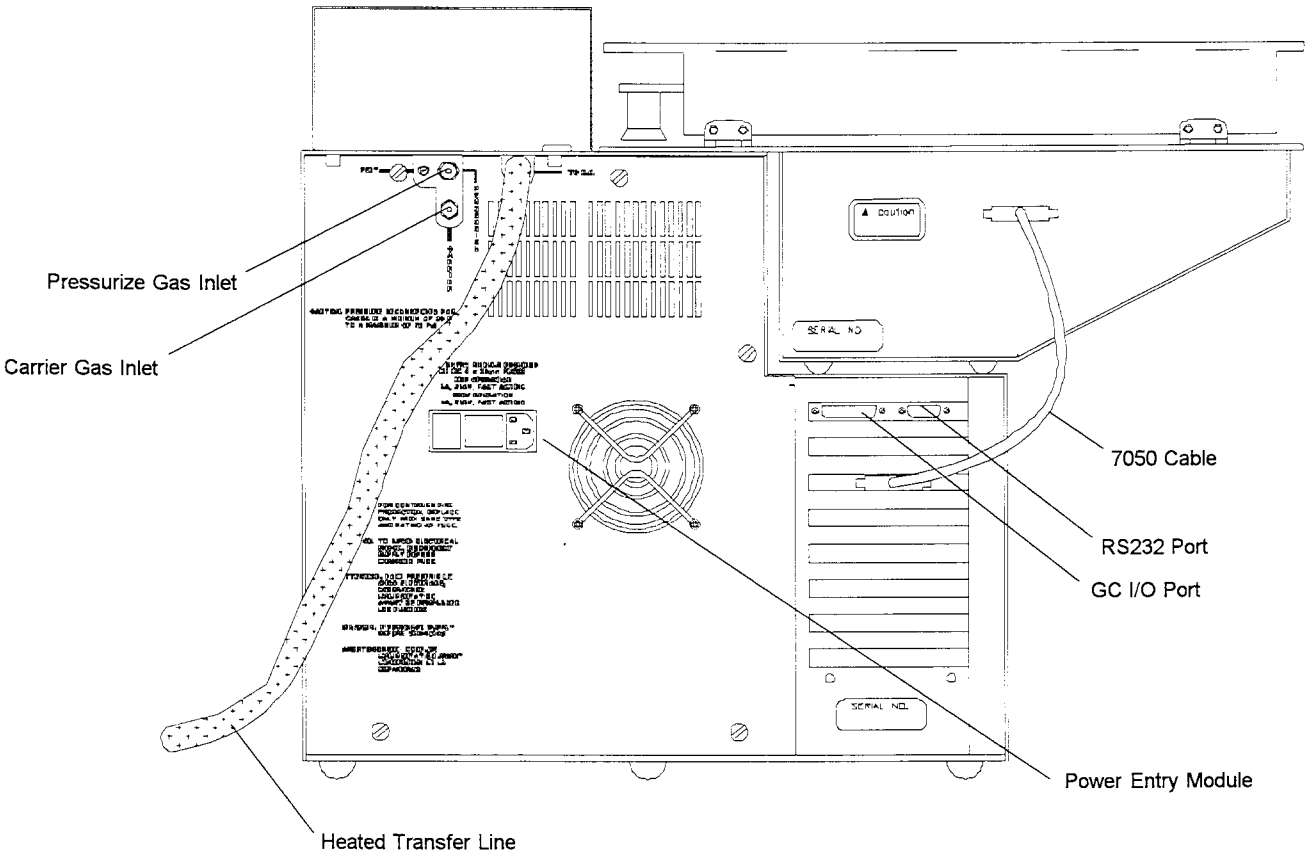


Figure 3-3 7000/7050 Rear View

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Plumbing Assembly/ Pneumatic Controls

The plumbing assembly area has the following components:

- **Pneumatic controls**
- **Pressurization valve** that supplies pressurization gas to the vial through the needle
- **Vent valve** that opens and fills the loop
- **Valve oven** (contains the 6-port valve, sample loop, and needle)
- **6-port valve and actuator** that route gas flow through the 7000 and separate the carrier system from the pressurization system
- **Stainless steel sample loop** (1 ml standard - optional sizes and materials available)
- **Sample needle** that punctures the septa to extract the headspace from the vial

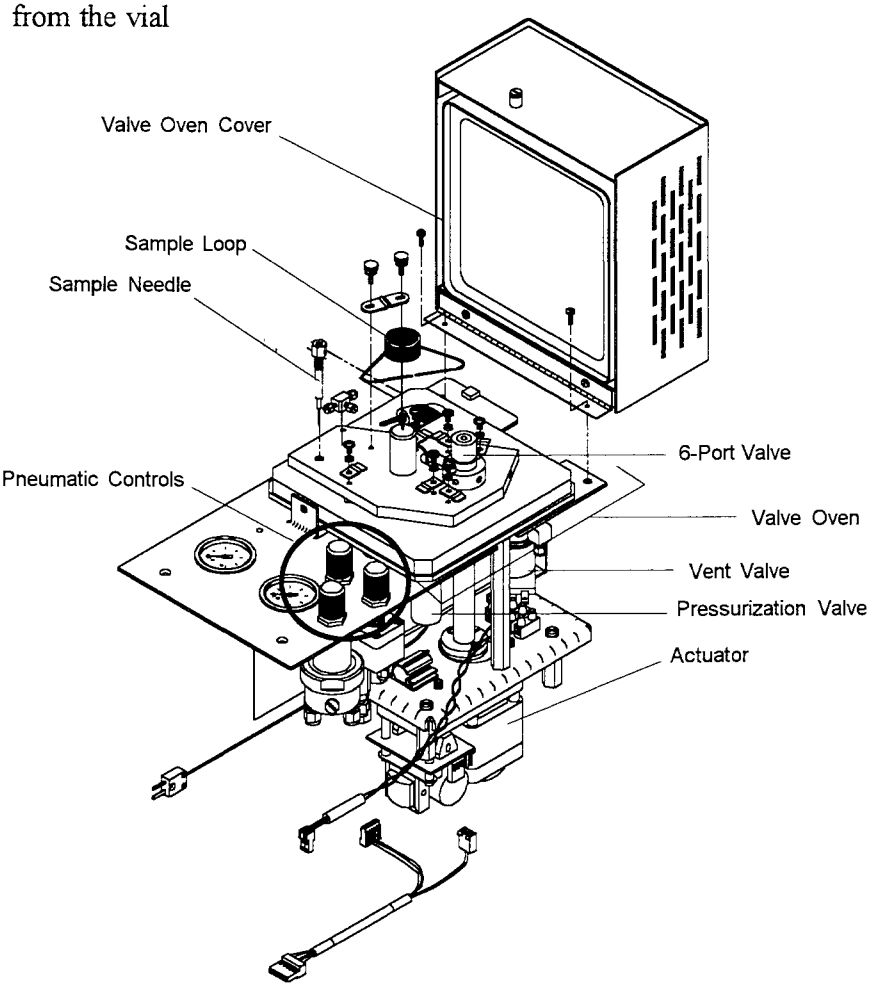


Figure 3-4 Valve Oven and Plumbing Assembly

continued

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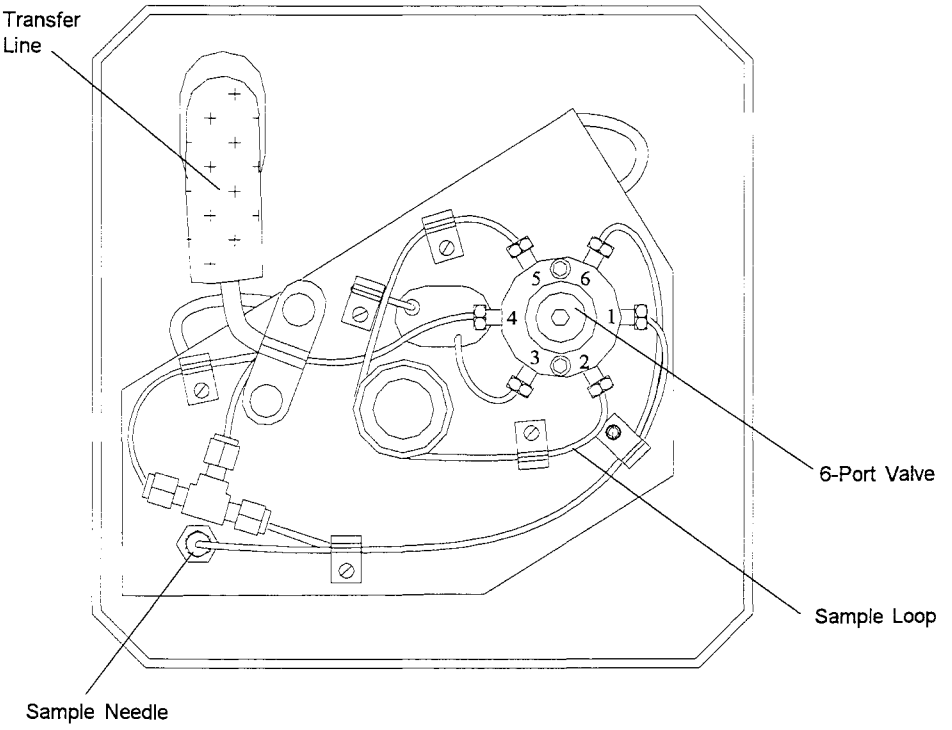


Figure 3-5 Valve Oven

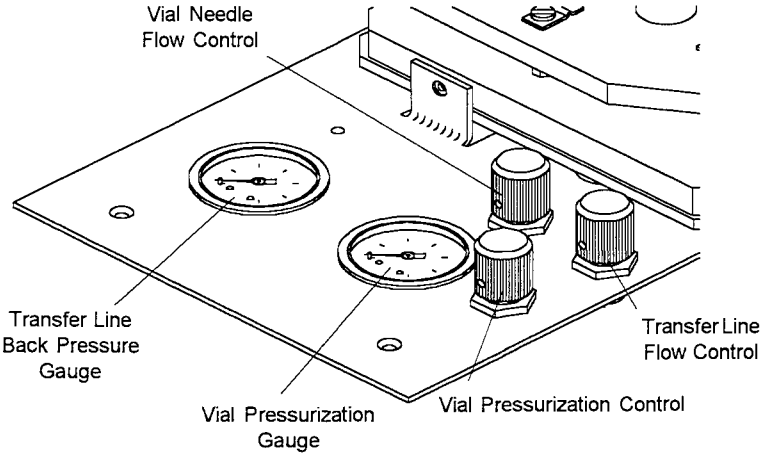


Figure 3-6 Pneumatic Controls

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7050 Carrousel

The optional 7050 Carrousel is used to automatically load and unload up to 50 vials into the heated platen. It is installed on the top left side of the 7000. The Carrousel has the following components:

- **Rack** to hold 50 vials
- **Indexing motor** to shift left or right
- **Carrousel motor** for rotating the rack
- **Carrousel encoder ring and circuit board** to sense rotary movement
- **Indexer board** to sense home/ forward/ reverse positions

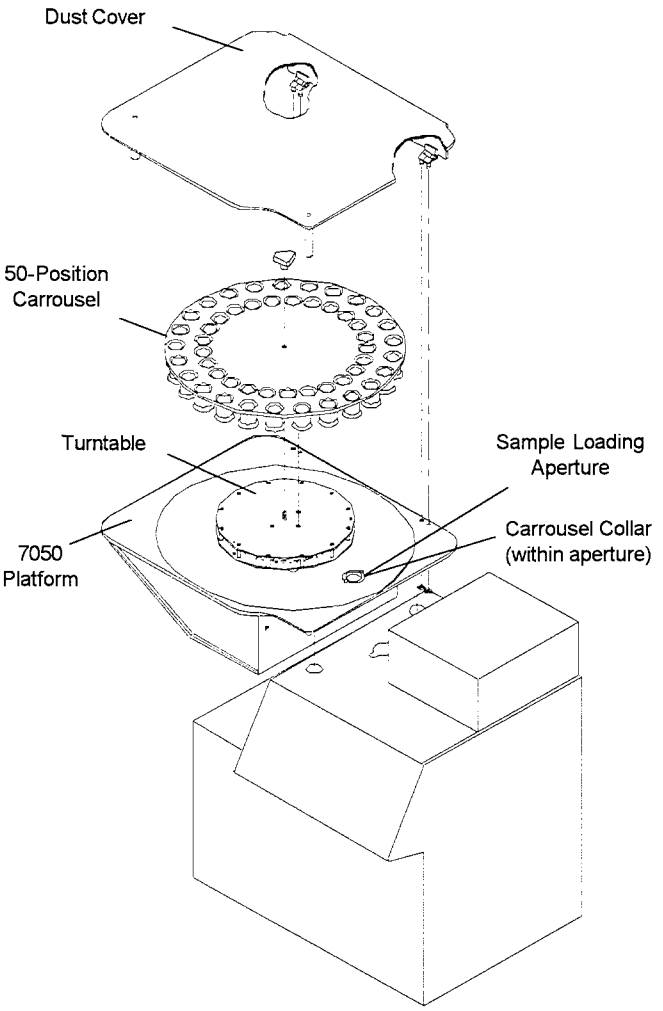


Figure 3-7 7050 Carrousel

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Platen Area

The platen is the 12-position, electrically-controlled device that holds vials as they are heated, equilibrated, mixed, and sampled.

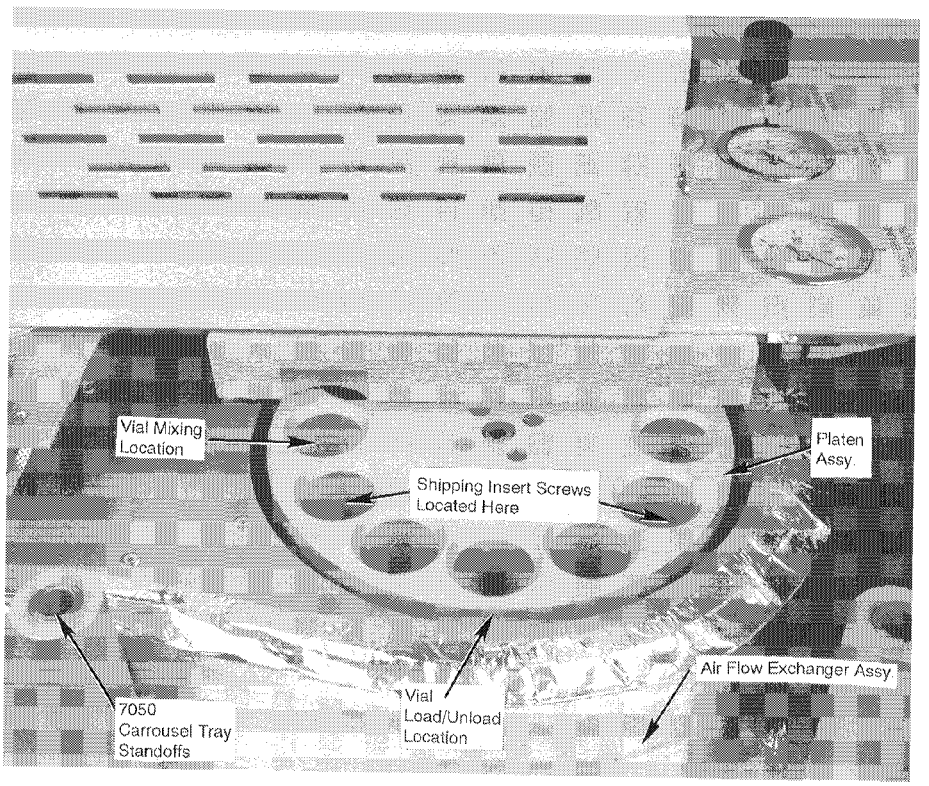


Figure 3-8 Platen Area

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Electronics Card Cage

The electronics card cage is located on the lower left-hand side of the 7000. The following circuit boards are in the card cage:

- The **power supply board**
- The **motor control board** - controls operation of the motors, motor overload, and the mixer output.
- The **logic/output board** - controls heater outputs, valves, and the platen fan.
- The **interface board** - allows the 7000 to communicate with other equipment, such as a GC, a mass spectrometer, data system, etc. This board accepts inputs to advance the 7000, and returns outputs on the status of the 7000.

The card cage has extra slots available for additional accessories, such as the Cryofocusing Module logic board.

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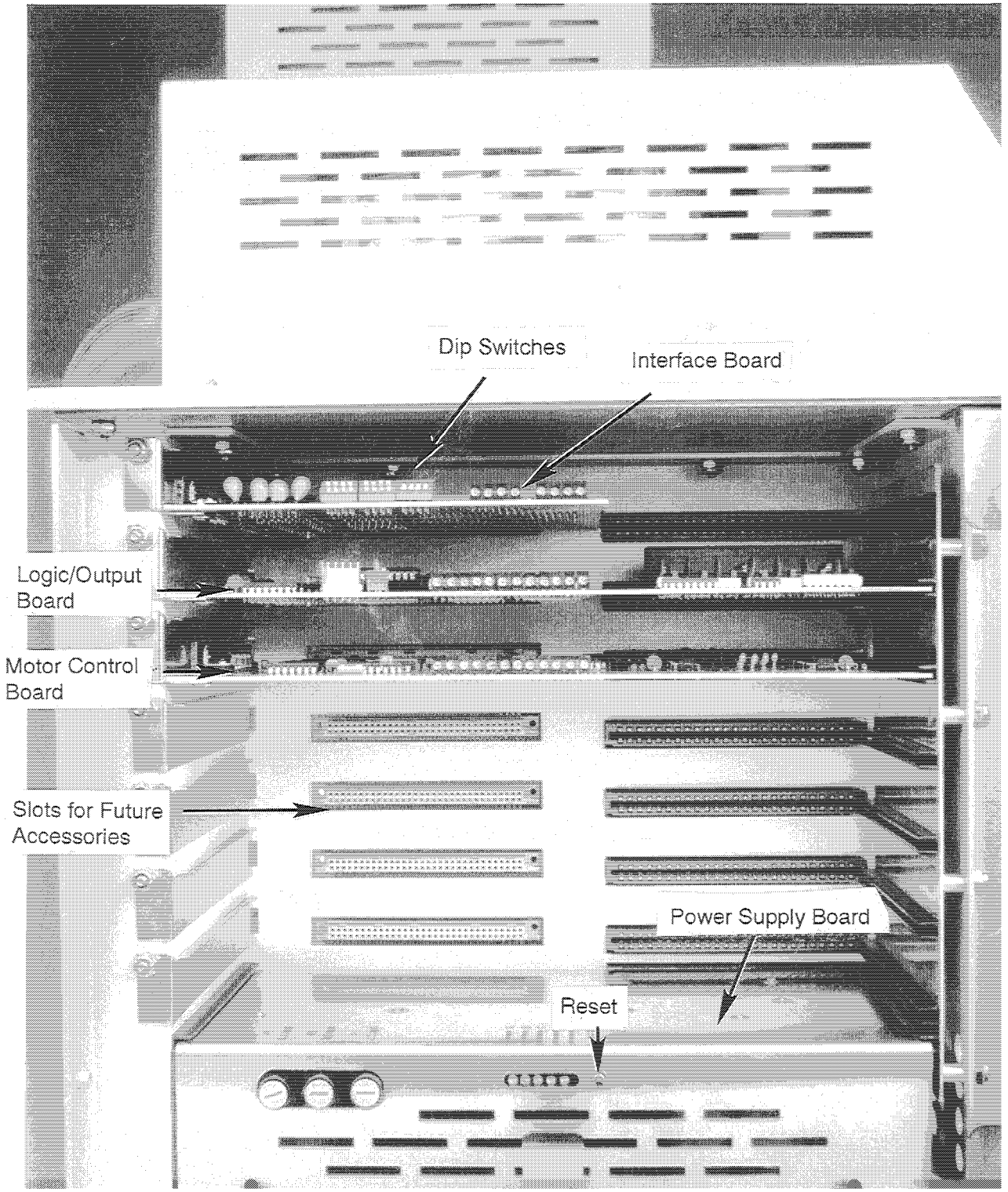


Figure 3-9 Electronics Card Cage (Left Side of Unit)

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4.1 Overview of the Section

This section on installing the 7000/7050 is intended to be as thorough as possible. Keep in mind, however, that certain tasks will vary depending upon the make and model of your gas chromatograph, mass spectrometer, and data system. If you are in doubt on any point, please contact Tekmar Service toll free at (800) 874-2004, or outside the U.S. at (513) 247-7000. When you are performing the 7000 installation yourself, you must be thoroughly familiar with this manual and all relevant sections of the gas chromatograph manual before proceeding.

Section 4 includes these topics:

- Preliminary setup procedures (unpacking, placing the unit on the bench)
- Vent outlet connections
- Choosing a gas supply
- Pressurization gas connections
- Carrier gas connections
- Transfer line connections
- Electronic connections
- Replacing the microprocessor ROM
- Installing the 7050 Carrousel
- Installing accessory boards
- Changing the sample loop
- Changing the sample needle
- Leak checking
- Checking the vent

4.2 Preliminary Procedures

To ensure proper operation of the 7000/7050, there are several preliminary steps that must be followed before making electronic and pneumatic connections. These steps are described on the following pages.

Materials Needed for Installation

You will need the following tools and materials, most of which are included in the assembly kit you received with your instrument or the Installation Kit you should have purchased separately (p/n 14-5093-000).

Assembly Kit Contents

Power Cord - Universal
Cap, Aluminum 20 mm Neck (pk of 125)
Septa, Silicon (pk of 125)
Vials, 22 ml (pk of 125)
Nut, Plug, Brass, 1/16"
Fuse, 2 AGC
Fuse, 8 AMP Rectifier
Fuse, GDB-8A, 250V, 5 x 20mm
Ferrule, Brass, 1/8"
Nut, Brass, 1/8"

continued

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Installation Kit Contents

Trap, Hydrocarbon
1/8" Copper Tubing
Ferrule Set, Brass, 1/8"
Reducer, 1/16" to 1/8"
Nut, Brass, 1/8"
Tee, Brass, 1/8"
Union, Tube, Brass, 1/8"
Union, SS, 1/16" - 1/16"
Bulkhead, 1/16", for Cap Plug
Wrench, 1/4 - 5/16 Open End
Wrench, 7/16 - 1/2 Open End
Slot Stubby Screwdriver
Phillips Screwdriver
Wrench, 3/8"
Nickel Tubing
Cap Crimper
0-10 ml/min Flow Element

Other Materials Needed for Installation

Two 5/16" Wrenches
Two 7/16" Wrenches
Extra 1/8" Copper Tubing for Venting

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Power Requirements

The 115V unit requires a 50 or 60 Hz single phase power source at 115V + 10%. The 230V unit requires a 50 or 60 Hz single phase power source at 230V + 10%. For the 115V unit, the maximum current draw is 8 amps and maximum power consumption is 920 watts (when accessories are included). For the 230V unit, the maximum current draw is 4 amps and maximum power consumption is 920 watts (when accessories are included). The 100V unit power cord terminates with a 3-prong straight blade plug and requires a matching receptacle. For the 100V unit, the maximum current draw is 10 amps and maximum power consumption is 1,000 watts (when accessories are included).

Unpacking the Instrument

To remove the 7000/7050 from its shipping container:

1. Cut the strapping from the box.
2. Remove the outer carton by lifting it up over the instrument.
3. Remove the shipping insert.
4. Remove the top foam pad.
5. Lift the instrument from the base of the box to the work space you have prepared.

> ⚠ **WARNING**
>
> **The unit weighs approximately 80 pounds. Do not attempt to lift the 7000/7050 by yourself.**

Inspecting the Instrument

Carefully examine the instrument. If you see that the 7000 or any of its accessories are damaged, notify both the shipping carrier and Tekmar Company immediately. If the instrument appears to be damaged, do not continue the installation until directed to do so by a Tekmar representative. **Failure to comply with these instructions may void your warranty on components damaged in shipment.** Do not return any materials to Tekmar without prior authorization. **(You can reach Tekmar Customer Service at (800) 543-4461.)**

Note: Save all shipping materials until you are certain the instrument is operating properly. Should you need to return the 7000, please remove the 7050 dust cover (see Section 4.5, Installing the 7050 Carrousel) before shipping.

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Removing the Shipping Inserts

There are two plastic shipping inserts that you will need to remove from the platen before proceeding with operation of this instrument. The diagram below shows the location of these inserts. There is a label on top of the instrument with instructions for removing the inserts. If you cannot locate the label, remove the inserts using the following directions:

1. Remove the four small Phillips head screws (two/front, two/rear) from the platen cover panel (it spans from the left side of the keypad to the rear of the instrument).

2. Find the two inserts and their hold-down screws in the platen (at the 7 and 11 o'clock positions when facing the front of the 7000).

3. Unfasten each hold-down screw with the Phillips head screwdriver.

4. Pull the insert and screw out together by placing pressure on the side of the insert with the screw as you pull up.

5. Replace the panel with the Phillips head screws and tighten down.

   Note: Be sure to replace the screws. Running the instrument without them could cause a misalignment of the system.

6. Store the inserts for future shipping needs.

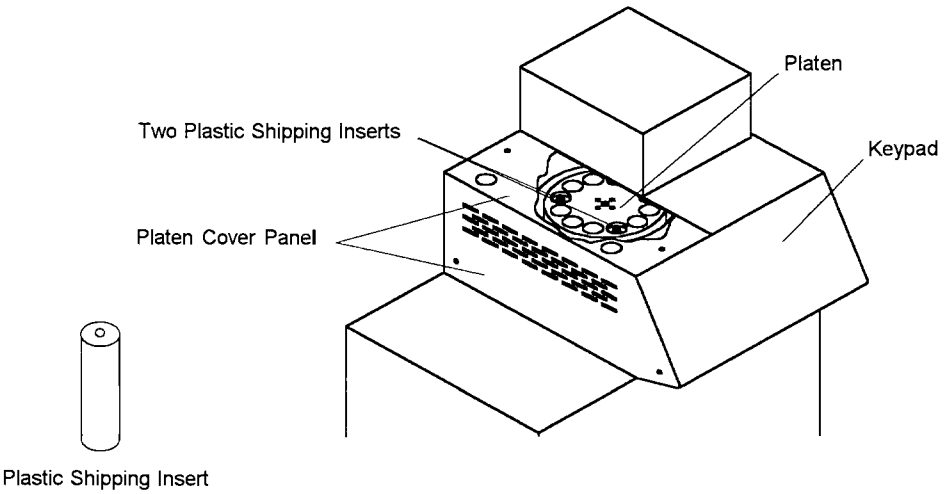


Figure 4-1 Location of the Platen Shipping Inserts

Placing the 7000 At Your Workstation

Place the instrument on a sturdy, stable bench surface beside the gas chromatograph. Place the 7000 close to the intended GC injection port. Allow sufficient space at the rear of the instrument for access and air circulation. Leave some clearance to the left of the 7000 for easy access to the electronics area.

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4.3 Pneumatic Connections

Venting the 7000

⚠ WARNING

During normal operation of the 7000, small portions of the sample's headspace may be vented to the laboratory air. If your samples contain toxic or other potentially hazardous compounds, you will need to protect lab personnel from contamination of the laboratory atmosphere.

There are two different ways to vent hazardous compounds:

1. Connect an adsorbent trap to the stainless steel fitting labeled "Vent" on the rear panel of the 7000. The adsorbent(s) should be effective in trapping hazardous materials known to be present in your samples. You should use a trap that will not create significant back pressure (e.g. the Tekmar green canister hydrocarbon trap available in the Installation Kit, p/n 14-5093-000).

2. Connect nickel tubing (p/n 14-5229-002) with a 1/16" stainless steel nut (p/n 14-0798-016) and 1/16" stainless steel ferrule (p/n 14-0799-016) to the 1/16" bulkhead labeled "VENT" on the back of the 7000. Route this tubing so that it terminates inside a fume hood.

   For information on recommended vial pressurization settings, please refer to Section 9.

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Choosing a Gas Supply

Choice of gas supply for your 7000 operation is one decision you must make before startup. Typically, the carrier gas is used for pressurization, but you may elect to use any non-flammable gas supply. Other related considerations are:

- **Control of Flow:** Column carrier gas flow can be supplied and regulated by either the 7000 or your GC. See the section **Connecting the Carrier Gas Supply** beginning on page 4-8 for instructions on these connection options.

  Note: If you plan to install a Cryofocusing Module to a Hewlett-Packard (HP) 5890A gas chromatograph (including the mass spec), or any back pressure-regulated GC, configure the 7000 to control flow with an external pressure regulator assembly (14-3938-000) with a 0-10 cc flow element in the 7000 transfer line flow controller.

- **Column Size and Flow Range**: See the section **Connecting the Carrier Gas Supply** (page 4-8) for detailed information.

- **Transfer Line/Inlet Connections:** See **Connecting the Transfer Line from the 7000 to your GC Inlet** (page 4-15).

Pressurization Gas Requirements:

Vial pressurization gas and column carrier gas are two separate connections on the 7000. Pressurization gas can be different from the carrier gas but should be of high purity. Connect the green hydrocarbon trap (p/n 14-1362-000, available in the Installation Kit, p/n 14-5093-000) to your pressurization supply gas to maintain purity. An acceptable flow range is 20-70 psi with 50 psi as the optimal setting.

⚠ DANGER



Never connect a flammable pressurization gas (such as hydrogen) to the pressurize bulkhead fitting. Venting during Standby mode may create an explosion or fire hazard.

Carrier Gas Requirements:

The 7000 requires a high purity (99.999% pure hydrocarbon tested, less than .5 ppm or better) helium or nitrogen carrier gas supply in the 5 to 70 psi range. The optimal setting will depend upon whether column carrier flow is supplied by the GC or the 7000. Choice of gas type (either helium or nitrogen) makes no difference to 7000 operation. However, this gas will also be used to supply carrier gas to the GC column, so you should consider the GC requirements before deciding. Another consideration is the type of gas used for leak detection. If you use an electronic leak detector, it is best to use helium for pressure and needle gas leak checks.

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Connecting the Pressurization Gas Supply

Here are the steps in connecting the 7000 to a pressurization gas supply:

1. Turn off the gas supply at its source. Cut the carrier gas line. Use the supplied 1/8" tee (p/n 12-0070-016) to connect the carrier gas line and the hydrocarbon trap (p/n 14-1362-000), available in the Installation Kit, p/n 14-5093-000). You can also connect the trap to the gas supply independent of the GC if you are not using the GC carrier gas for vial pressurization.

2. Continue the pressurization gas line from the trap to the fitting marked "Pressurize" at the rear of the 7000. Use an 1/8" brass nut (p/n 12-0069-016) and ferrule (12-0044-016) to make the connection. Tighten.

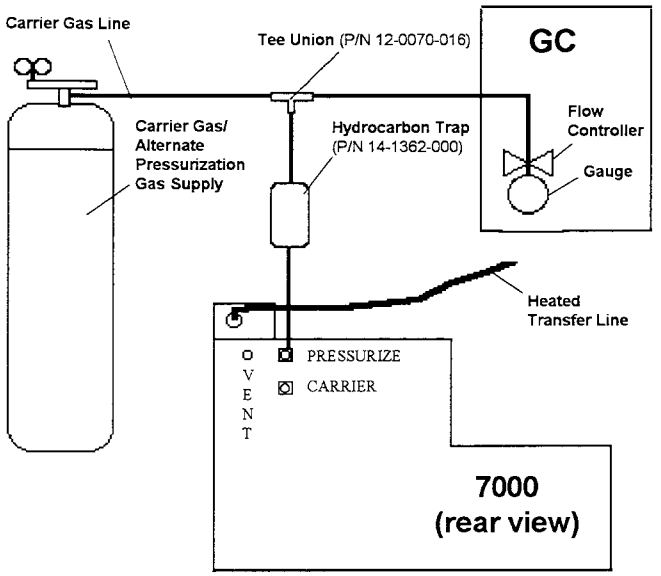


Figure 4-2 Typical Pressurization Gas Connection

Note: Before turning on the 7000, check that the pressure is in the range of 20 to 70 psi.

3. Turn on the gas supply via the flow control knob on the top of the 7000. See Section 9.5, ***Optimizing Headspace Sensitivity,*** for recommended settings.

⚠ DANGER

Never connect a flammable pressurization gas (such as hydrogen) to the pressurize bulkhead fitting. Venting during Standby mode may create an explosion or fire hazard.

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Connecting the Carrier Gas Supply

The carrier gas supply for the 7000/GC pneumatic interface can be configured in one of three ways, depending upon your particular application and preference:

- The carrier gas can be regulated by your GC inlet flow controller;
- The carrier gas can be regulated by the 7000 with an external pressure regulator assembly (p/n 14-3938-000);
- The carrier gas can be regulated by the transfer line flow controller of the 7000.

The following pages describe procedures for each of these connection methods, which require parts from the 7000 Installation Kit (p/n 14-5093-000).

7000 Flow Controller-Regulated Carrier Gas

The following instructions and diagram explain how to connect the carrier gas so that it is regulated by the 7000 flow controller.

To make the connection:

1. Turn the GC oven off and allow the column to cool.
2. Shut off the carrier supply to the GC from the two-stage regulator at the supply tank.
3. Cut the 1/8" copper line from the hydrocarbon trap to the "PRESSURIZE" bulkhead and install the supplied 1/8" tee (p/n 12-0077-016).
4. Connect the supplied 1/8" copper tubing (p/n 14-0546-002) to the open end of the tee.
5. Connect the other end of the tubing to the fitting on the back of the 7000 labeled "Carrier."
6. Connect the heated transfer line to the column via a:
   - Direct column connection inside the oven (GC flow controller is turned off)
   - Direct column connection using the Low Volume Injector
   - Septum needle adapter

   These methods are described later in this section.

continued

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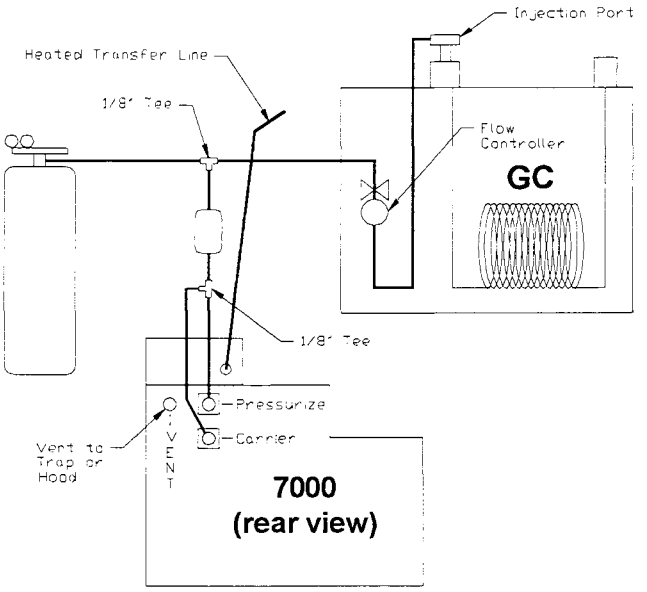


Figure 4-3 Carrier Gas Connection (7000-Controlled via Flow Controller)

The transfer line flow controller has a 0-60 ml factory-installed flow element which accurately controls flow from 6-54 ml/min. If the flow rate falls outside of this range, install one of the following flow elements:

Flow Element Part # and Description	*Range*
• p/n 14-4925-050, Blue	0 - 10 ml/min
• p/n 14-4936-050, Red w/Silver Dot	0 - 25 ml/min
• p/n 14-4928-050, Black	40 - 440 ml/min

Note: Flow elements need at least 50 psi inlet pressure to provide the full range of control.

continued

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Installing the Flow Element in the Transfer Line Flow Controller

1. Remove the six 1/4-turn fasteners from the right side panel of the 7000. Remove the panel.
2. Locate the base of the transfer line flow controller.
3. Unscrew the red color-coded flow element.
4. Replace with the new color-coded flow element.

Note: Do not over-tighten the flow element during installation. Over-tightening will increase flow restriction and hinder performance.

5. Replace the side panel.

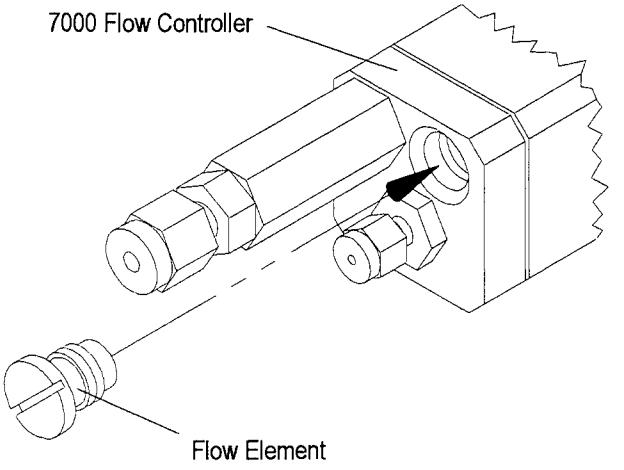


Figure 4-4 Flow Element Location

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Regulating Carrier Gas with an External Pressure Regulator

In some instances, (particularly when flow rates of 1 - 2 cc/min are being used), an external pressure regulator assembly (p/n 14-3938-000) may lead to higher flow precision. This is true only when the 7000 flow controller is used to control the GC carrier gas.

Note: The external pressure regulator is required when using a mass spectrometer (with flow rates of 1cc/min and below) or a cryofocusing module.

Note: If you are installing to a mass spectrometer, bypass the transfer line flow controller pneumatics entirely.

To connect carrier gas so that it is controlled by the external pressure regulator/7000 configuration:

1. Turn off the GC oven and allow the column to cool.
2. Turn off the GC carrier supply line at the two-stage regulator on the supply tank.
3. Cut in two the 1/8" copper line from the hydrocarbon trap to the bulkhead marked "PRESSURIZE". Install the supplied 1/8" brass tee (p/n 12-0070-016).
4. Connect the enclosed 1/8" line to the open end of the tee with an 1/8" nut and ferrule.
5. Use the 1/8" brass nut (p/n 12-0069-016) and ferrule (p/n 12-0044-016) to connect the other end of the line to the bulkhead marked "inlet" on the back of the external pressure regulator.
6. Connect one end of another piece of 1/8" line to the "outlet" bulkhead on the back of the external pressure regulator. Use the 1/8" brass nut (p/n 12-0069-016) and ferrule (12-0044-016) to connect the other end of the 1/8" line to the fitting labeled "Carrier" on the back of the 7000.
7. Place this assembly in a location which allows for easy access for viewing and changing column back pressure.
8. Turn the transfer line flow controller to its full open position.

Note: If you see significant backpressure using a standard flow element in the flow controller, remove the existing flow element and install the 40-440 ml flow element (black) using the procedures on the previous page.

continued

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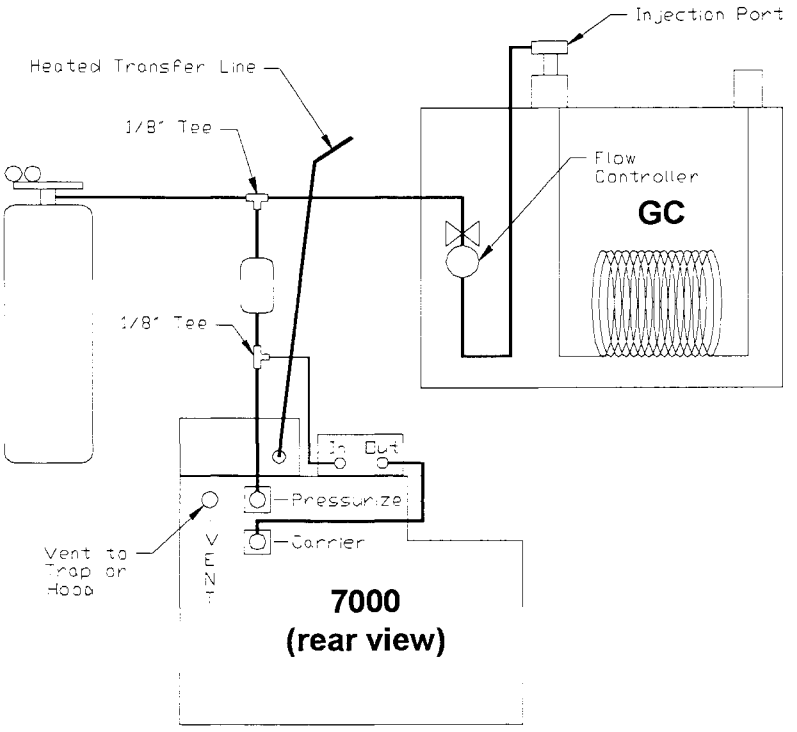


Figure 4-5 Carrier Gas Connection
(7000-Controlled via External Pressure Regulator)

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GC-Supplied Carrier Gas

The GC supplies and controls the carrier gas when it is connected to the 7000 in the following manner:

1. Turn off the GC oven and allow the column to cool.
2. Select the GC inlet that will be used for analysis and remove any covers around that inlet.
3. Locate the 1/16" regulated line supplying carrier gas into the inlet of the injector.
4. Cut this line approximately 1 - 2" from the injector housing.
5. Connect the regulated line coming from the GC flow control pneumatics to a 1/16" union (p/n 14-0051-016).
6. Connect one end of the supplied 5 ft. x 1/16" nickel tubing (p/n 14-5229-002) to this union with the 1/16" stainless steel nut and ferrule.
7. Connect the other end to the 1/8" to 1/16" reducing union (p/n 14-0050-016) which connects to the 1/8" copper tubing (p/n 14-0546-002). Connect the tubing to the bulkhead labeled "Carrier" on the back of the 7000 using an 1/8" brass nut (p/n 12-0069-016) and ferrule (p/n 12-0044-016).

Note: If you see significant back pressure using a standard flow element, install the 40-440 ml flow element (black) using the procedures on page 4-10.

8. Turn the transfer line flow controller on the 7000 counter-clockwise to its fully open position.

Note: When the GC is controlling the flow to the 7000 in packed column applications at higher flow rates, the 7000 transfer line back pressure gauge may exceed 30 psi. This is normal; it will not restrict flow rates.

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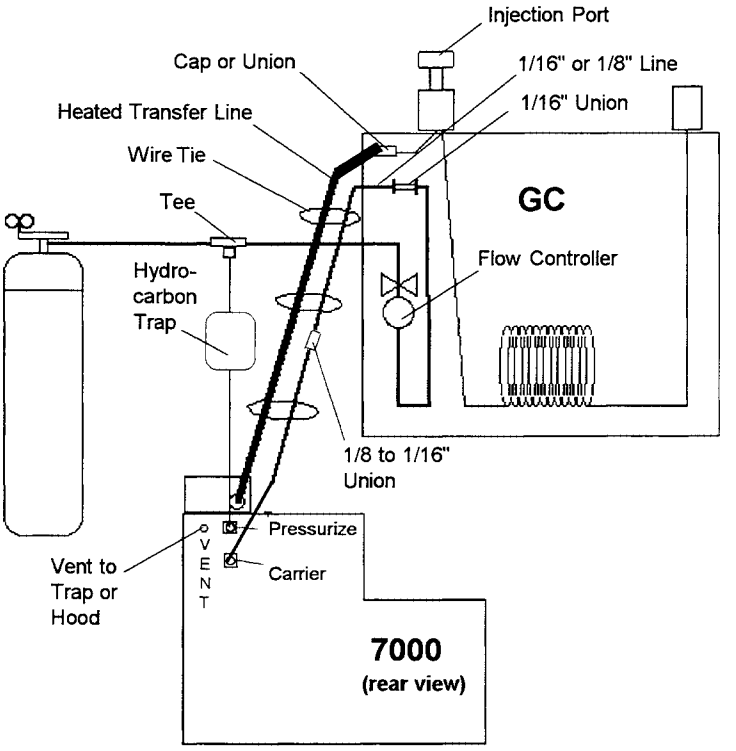


Figure 4-6 GC-Supplied Carrier Gas - Connections

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Connecting the Transfer Line from the 7000 to the GC Inlet

The heated nickel transfer line is coiled on the back of the 7000. Cut the wire tie from the transfer line and uncoil it from the 7000. If a column is already installed in the GC, turn off the oven and allow it to cool to room temperature. The carrier gas supply will be interrupted during installation, so the column must be cool to prevent damage.

There are five different methods of connecting the transfer line to the GC:

- Standard Inlet Line
- Direct Column
- Septum Needle Adapter
- Low Volume Injector
- Cryofocusing Module

Each has advantages and disadvantages. These are described in the following sections. Please read these sections completely before deciding on an installation method.

Note: You may install 0.32 mm ID (p/n 14-0539-002) or 0.53 mm ID (p/n 14-2072-002) fused silica lines in the transfer line by removing the installed nickel (0.5 mm ID) transfer line*. This procedure may be necessary when your application requires the inertness of deactivated fused silica.

***Note: To easily remove the nickel tubing, heat the transfer line to approximately 200°C, and use a pair of pliers to pull out the tubing.**

⚠ WARNING



Hot surfaces. Use caution when working with the instrument.

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Standard Inlet Line Connection

The advantages of the standard inlet line installation method are:

- It is the most leak-free operation.
- It is simple to set the carrier flow rate.
- The injection port remains open for manual injection methods or autosamplers.

The disadvantage is:

- It requires you to disconnect the transfer line from the 1/16" union to move the 7000 from GC to GC.

To make the standard inlet line connection:

1. Locate the 1/16" line that is connected to the GC injection port.
2. Use a 1/16" stainless steel union (p/n 14-0051-016) to connect the transfer line to the cut line entering the injection port.

   Note: The transfer line should be routed so that it doesn't interfere with normal GC operation.
3. Replace all of the GC covers to accommodate the transfer line.
4. Install the splitless liner into the capillary injection port, if applicable.

Note: If you are performing splitless runs, the split vent is capped off on the backpressure-regulated port. You will need to configure the 7000 to control flow from its transfer line flow controller (install the 0-10 ml flow element). You can also use the external pressure regulator (p/n 14-3938-000). See pages 4-10 to 4-11 for instructions on these two methods.

Note: If you are performing split runs, the split vent is not capped off. You will need to use a 40-440 ml/min flow element in the transfer line flow controller. An external pressure regulator is not necessary.

Note: Septum purge or split flow techniques are not recommended for capillary inlet operations. Therefore, the septum purge line and split vents should be turned off and capped. These recommendations are based on the following information:

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Septum purge flow leads to significant, non-reproducible loss in sample because the carrier inlet connection (from which the sample is introduced) is positioned directly across from the septum purge vent. If this vent is open, the sample will travel across the top of the injection port and out the septum purge vent.

Some analysts are uneasy about turning the septum purge off to help prevent septum bleed and carryover. But these problems are not seen with vapor phase samples introduced through the carrier inlet line.

Septum purge flow works best with liquid injections at temperatures that cause significant vaporization pressure pulse and flashback onto the septum. With the 7000, the vaporization step already occurs by the time the sample is introduced into the injection port. Therefore, flash vaporization-related septum bleed/carryover problems are not seen with the standard inlet line connection method.

Note: Tekmar recommends ultra low-bleed septa to prevent septum outgassing.

Split flow is recommended when sample concentrations are high enough to cause column and detector overloading. A split ratio of 50:1 means that 1/50th of the sample travels down the column and the rest of the sample is expelled from the split vent. The 50:1 split ratio reduces the amount of sample introduced to the column/detector and can help eliminate peak shape abnormalities and detector linearity problems. If column/detector overloading occurs with standard (carrier) inlet line-introduced vapor phase samples, you can try this procedure:

1. Dilute the sample to reduce the concentration of analytes introduced to the column and detector, or

2. Increase the vial pressurization setting to dilute the headspace, or

3. Reduce the injection volume (loop size), or

4. Adjust the phase ratio (volume of sample matrix).

The split ratio technique is ***not*** recommended for reducing vapor phase sample concentrations when a phenomenon known as "injection port fractionation error" occurs. This is when a sample is already in the vapor phase as it is introduced to the split capillary inlet (from the carrier inlet line), causing significant calibration errors.

continued

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Fractionation changes the split ratio of each analyte at different concentrations. The problem is compounded by the fact that fractionation is not reproducible, especially at low split ratios and/or low flow rates. So, you will see less precision when split flow is used with vapor phase samples introduced through the standard inlet line.

When vapor phase samples are exposed to low split ratios, the concentration and type of analyte in the vapor phase affects how each analyte is split. The split ratio injection technique usually works well for liquid injections.

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Direct Column Connection

In **direct column connection**, a zero dead-volume union is installed between the end of the transfer line and the GC column.

The advantages of this installation method are:

- There is a complete transfer of injection onto the column.
- Band broadening is reduced (as compared to connection to an injection port).

The disadvantage is:

- You cannot do injections directly on the column for problem isolation (without having to first disconnect, then re-connect the column to the injector).

To make the direct column connection:

1. Route the transfer line through any convenient opening in the oven (unused injection ports, for example) until the heater butts against the outside of the oven. If no ready-made openings are available, you can drill a small hole through the oven insulation near the injectors in the valve oven area.
2. Connect the transfer line to the column using the appropriate size zero dead volume union for 0.53, 0.32, 0.25 mm ID columns and 1/8 - 1/4" columns.
3. Adjust the flow rate from either the 7000 or the GC pneumatics and measure the carrier gas flow at the end of the column or detector.

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Septum Needle Adapter Method

The **septum needle adapter** connection method involves connecting the transfer line to the injection port through the GC inlet septum.

The advantage of this installation method is:

- It allows for quick disconnect of the transfer line from the GC (recommended when the 7000 must be transported between operations, for example).

The disadvantage is:

- The injection port is no longer available for use with autosamplers.
- It is prone to leaks.

To install the septum needle adapter:

1. Determine which injector is to be used and remove the nut from the top of the injector.

2. Select an injector adapter appropriate for your GC.

   Note: The Septum Needle Adapter is available for various Hewlett-Packard, Varian, Perkin Elmer, and Shimadzu GCs.

3. Replace the existing septum with the new one provided.

4. Screw the injector nut and septum into place. The septum nut should be lightly turned but not completely tightened at this point.
   Note: Overtightening the septum nut may core the septum when the needle goes in.

5. Screw the transfer line nut onto the 1/16" fitting on top of the needle interface.

   Note: You can use the optional transfer line guide when fused silica transfer lines are installed in the 7000. To make this connection:

   5a. Connect the fused silica line to the 1/16" fitting on top of the needle interface. Slide the needle and the transfer line through the top of the transfer line guide until the needle shroud butts against the bottom of the guide base.

   5b. Secure the needle hub by tightening the knob on the side of the transfer line guide.

6. Insert the transfer line with needle through the top of the injector nut. This will allow the needle to puncture the septum.

continued

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7. Finger-tighten the septum nut.

8. Check for leaks at the injection port (see Section 4.7 - Leak Checking the System.). If there is a leak, tighten the septum nut by turning 1/4-turn. If the leak persists, replace the septum and repeat the above procedures.

Note: If chromatograms show splitting or tailing peaks, set flows so that 70% of the column flow is through the transfer line, and the remaining 30% is supplied through the side inlet of the injector to eliminate discrimination in the column.

Septum Needle Adapter in the Splitless Capillary Operation Mode

When using capillary injectors, the splitless (vs. split mode) mode of operation is recommended.

To connect via a septum needle adapter in the splitless mode:

1. Make sure the splitless liner in the injection port is properly installed.

2. Verify that the septum purge line is turned off and capped.

3. Verify that the split vent is turned off and capped.

   Note: When you cap off the split vent, use the 7000 transfer line flow controller (0-10 ml flow element) or external pressure regulator (p/n 14-3938-000). See pages 4-8 to 4-12.

4. Turn off the GC carrier gas supply.

Column carrier flow is now supplied through the needle of the 7000.

Note: If you are using the GC controls for the carrier gas supply, withdrawing the needle from the injection port will halt column flow. Therefore, always turn off the GC oven and allow the oven and column to cool before removing the needle from the port.

Note: When the septum needle adapter is used in the capillary splitless mode or cap-on-column mode, Tekmar recommends you use either the 0-10 ml blue color-coded flow element (p/n 14-4925-050) in the transfer line controller, or the external pressure regulator (p/n 14-3938-000). Either will help ensure precise flow from the 7000 at a rate of approximately 1 ml/min.

continued

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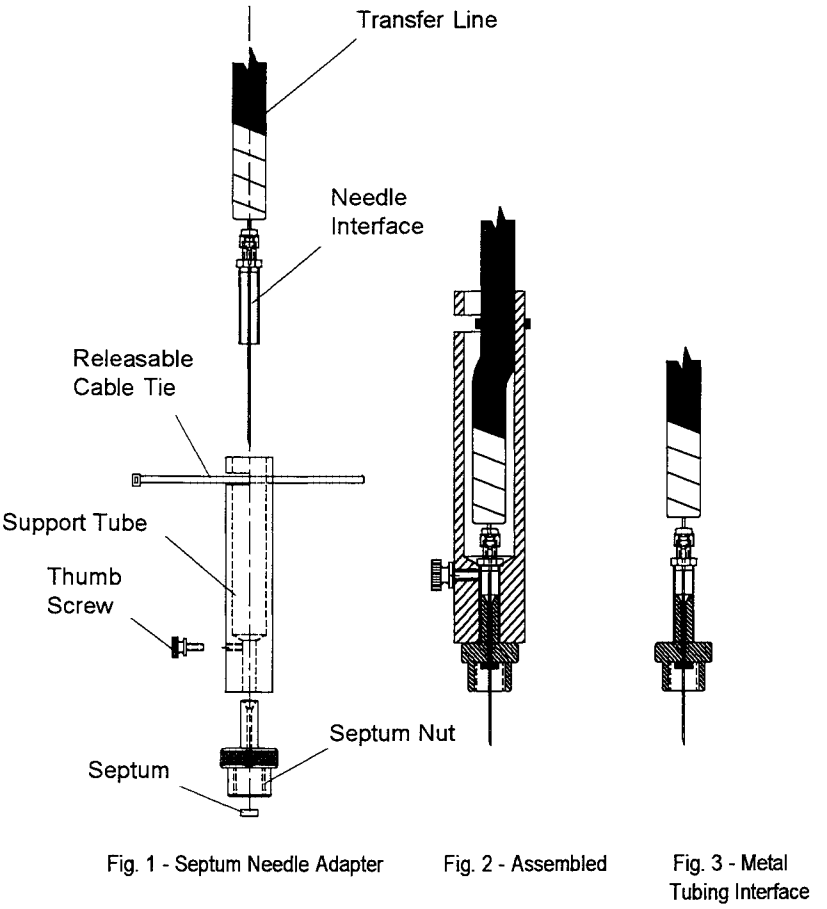


Figure 4-7 Septum Needle Adapter (pat. pending)

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Septum Needle Adapter in the Capillary Split Mode

Please note the caution at the bottom of page 4-17 regarding the use of the split injector with vapor phase samples.

To connect with a septum needle adapter in the split mode:

1. Turn off the oven and allow the column to cool.

2. Withdraw the septum needle adapter.

3. Install the 40-440 ml flow element (black color-coded - p/n 14-4928-050) on the 7000 transfer line flow controller by removing the right side panel and unscrewing the red color-coded 0-60 ml flow element from the flow controller.)

Note: The GC carrier flow is now reduced.

4. Turn on the GC flow pneumatics to the split injector and set up the injector and detector flow rate for anticipated split operation.

5. Adjust the split flow rate to 10 - 20 ml/min. (as measured from the split vent).

6. Connect the septum needle adapter to the injection port as explained in the instructions on page 4-20.

7. Adjust the 7000 transfer line flow controller until approximately 70-90 ml/min flow is measured out of the GC split vent.

Note: For septum needle adapter operations in the capillary split mode, Tekmar recommends you use the 40-440 ml flow element (black color coded-p/n 14-4928-050) in the transfer line controller.

Septum Needle Adapter in the Capillary Injector On-Column Mode

See the instructions for connecting in the splitless capillary mode on page 4-21 of this section.

continued

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Septum Needle Adapter in Packed Inlet/Packed Column Operation

1. Turn off the oven and allow the column to cool.

2. Withdraw the septum needle adapter from the injection port.

Note: Withdrawing the needle from the injection port will halt column flow. Always turn off the GC oven and allow the oven and column to cool before removing the needle from the port.

3. Turn all detector gases off.

4. Using the GC carrier pneumatics, adjust carrier flow rate to approximately 5-10 ml/min (measured at the end of the column or exit point of the detector).

5. Connect the septum needle adapter to the injection port (page 4-20).

6. Adjust the transfer line flow using the 7000 transfer line flow controller to a combined flow of 20-40 ml per min.

7. Turn the detector gases on.

Note: For septum needle adapter operations with packed inlet port operation, Tekmar recommends you use the standard installed 0-60 ml flow element (red color-coded - p/n 14-4926-050).

Septum Needle Adapter in the Packed Inlet/Megabore Column Mode

1. Turn off the oven and allow the column to cool.

2. Withdraw the septum needle adapter from the injection port.

Note: Withdrawing the needle from the injection port will halt column flow. Therefore, always turn off the GC oven and allow the oven and column to cool before removing the needle from the port.

3. Install the megabore lines and adapter to the injection port and connect the megabore column.

4. Turn the detector gases off.

5. Adjust the carrier flow rate from the GC pneumatics to approximately 25% of the final column flow rate (2-5 ml/min).

continued

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6. Connect the septum needle adapter to the injection port.

7. Adjust the carrier flow rate to achieve a final column flow rate of 8-20 ml/min.

8. Turn detector gases on to achieve a final flow of 3-15 ml/min.

Note: For septum needle adapter operations on packed injectors with megabore column flow between 8-20 ml/min, Tekmar recommends that you use the standard 0-60 ml red color-coded flow element (p/n 14-4926-050).

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Low Volume Injector (LVI)

A low volume injector lowers the volume of a standard GC-packed column injector, decreasing band broadening in the sample, and improving chromatography while leaving the septum free for direct injection.

Note: It is CRITICAL for proper installation of the injector that you follow these instructions exactly.

Note: The Low Volume Injector is available for various Hewlett-Packard, Varian, Perkin Elmer, and Tracor/Tremetrics injection ports.

Prepare the GC

1. Turn off the GC.
2. Remove the column from the injection port line inside the GC.
3. Remove the injection port nut and septum from the GC.
4. Remove the injection port liner, 9/16" liner nut, and 1/4" graphitized vespel ferrule from the inside of the GC. Save the nut and ferrule for later use.

Assemble and install the insert

1. Slide the 1/16" graphitized vespel seal ring onto the glass-lined insert tube. Be careful -- the glass-lined tubing will break if bent.
2. Slide the large injection port nut (supplied with the LVI) onto the insert tube.
3. Tighten the injection port nut 1/4-turn past finger-tight.
4. Unscrew the small septum nut on top of the assembly.
5. With a microsyringe, slide the septum nut and pre-drilled low volume injector septum through the end of the syringe. Be sure the teflon surface of the septum is positioned face-up as you place it in the nut.
6. With the syringe still in place, finger-tighten the nut onto the assembly.
7. Remove the syringe. This is **critical** in preventing septum particles from plugging the glass-lined insert.
8. Slide the low volume insert body down into the injection port, and hand-tighten the injection port nut.

   Note: Tighten only the large brass nut. Do not use the side arm transfer line nut and ferrule for leverage while tightening.

continued

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9. Place the 9/16" nut (face up) and the 1/4" graphitized vespel ferrule (cone-side up) over the top of the support tube. The nut and ferrule were set aside in Step 4 of the GC preparation instructions.

10. Slide the support tube up onto the glass-lined insert tube and into the injection port nut ferrule until it stops -- do not tighten yet.

    Note: Be very careful that you do not bend the glass-lined insert tube!

11. Slide the 1/16" graphitized vespel seal ring onto the glass-lined insert tube.

12. Screw the lower insert nut over the ferrule and onto the glass-lined insert tube.

13. Adjust the position of the support tube so that the glass-lined insert tube is flush with the bottom of the lower insert nut.

    Note: The glass-lined tube must not protrude past the end of the lower insert nut, but should be flush with the bottom of the nut.

14. Use a 1/4" wrench to hold onto the support tube. Use the other 1/4" wrench to tighten the lower insert nut 1/4-turn past finger-tight.

15. Tighten the 9/16" nut that you installed onto the GC in steps 9 and 10 1/4-turn past finger-tight.

    Note: Be sure the glass-lined tube is still even with the lower insert nut.

16. Slide the provided column nut and ferrule onto the column.

Install the column

Note: Use the appropriate ferrule and be sure it is placed cone-side down.

1. Cut appproximately 2" off the end of the column to create a clean edge and to remove any ferrule residue.

Note: Do the following step as precisely as possible to ensure the highest level of accuracy in your analytical run.

2. Slide the column up into the lower insert nut and up into the glass-lined insert tube until it reaches the restrictor in the glass-lined insert tube. The restrictor is about 1 mm from the end of the tubing.

continued

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You will be able to feel the column "drop" into place inside the restrictor -- **be sure the column tip is secured in the restrictor!**

3. Use a 1/4" wrench to tighten the column nut and ferrule 1/4-turn past finger-tight.

Install the 7000 transfer line

1. Slide the provided side arm transfer line nut and appropriate ferrule onto the 7000 transfer line. Be sure the ferrule is placed into the nut cone-side in.

2. Tighten the nut and ferrule of the transfer line to the side arm of the insert body.

3. Leak check all connection points.

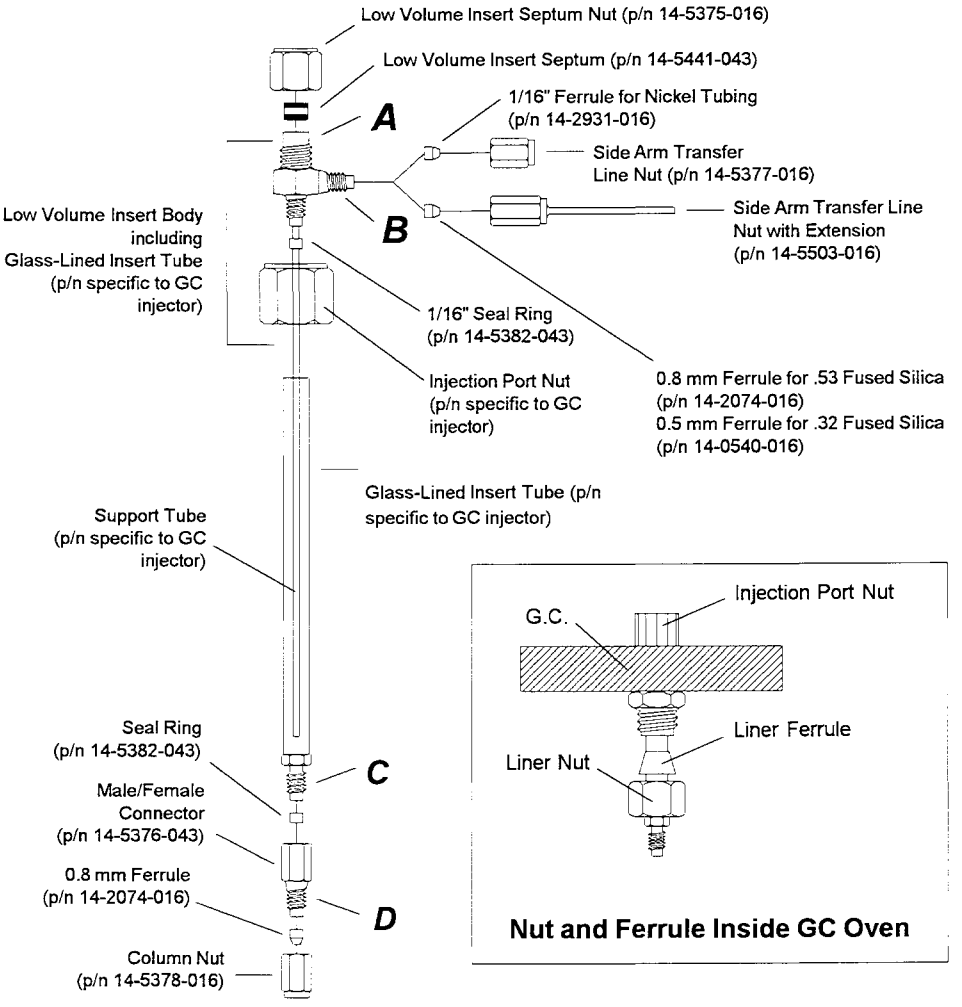


Figure 4-8 Low Volume Injector Assembly

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Cryofocusing Module

The Cryofocusing Module transfer line connection method is ideal for high sensitivity/high resolution capillary chromatographic analysis, such as when injected volumes exceed 2 ml and capillary column ID's are .32 mm or smaller.

Consider these variables when deciding whether to use the Cryofocusing Module:

- Injection volume
- Sample concentration within the sampling loop
- Column phase film thickness (column loading capacity)
- Column ID
- Column length

Refer to the following pages for pneumatic and electronic connections.

To connect the transfer line to the Cryofocusing Module:

1. Remove the outer and inner covers of the Cryofocusing Module by loosening the nut on the front of each cover and carefully sliding each cover up and forward.

2. There are two different mounting brackets in the accessory kit:

   - **The General mounting bracket (p/n 14-2781-000)**
     Use this bracket for all GCs except the HP 5890. Use the flat head screws (p/n 12-0323-VO1) to mount the bracket over the unused injection port.

     Note: On non-Varian installations, you will need to drill two holes to mount this bracket onto the GC.

   - **HP 5890 GC mounting bracket (p/n 14-3522-000)**
     Use the two #6-32 x 1/2" long pan head screws (p/n 12-0323-C01) and the two #6-32 locking nuts (p/n 12-0325-910) to mount the slotted portion of the mounting bracket to the slotted vent holes at the right of the injection ports. Align the bracket over the unused injection port.

     Note: HP Series II GC's do not have slotted vent holes on the top cover -- you will need to drill two holes.

Note: If neither of these brackets can be modified for your GC, contact Tekmar Service at 800-874-2004 for further help.

3. Connect the 1/4" insulated line (provided) to the bottom bulkhead union labeled "IN" on the rear of the Cryofocusing Module.

continued

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4. Connect the other end of this line to the fitting on the cryogenic valve assembly which sends the coolant to the Cryofocusing Module.

5. Remove the septum nut and the septum and pass the column up through the injector and out of the GC.

6. Place the septum nut over the column and tighten it to the injector.

7. Carefully mount the Cryofocusing Module onto the bracket so that the column passes through the 1/16" conduit of the cryofocusing heater.

8. Loosely fasten the nuts that hold the Cryofocusing Module to the bracket.

9. Pass the column through the cryofocusing assembly, carefully sliding the nut and ferrule onto the column.

10. Use a capillary cutting tool to carefully cut approximately 1/4" off the end of the column to remove any ferrule fragments.

11. Slide the column halfway into the bottom of the gold-plated union. Attach it with the nut and graphite vespel ferrule.

12. Loosen the four thumb screws on the back of the Cryofocusing Module and carefully slide the assembly down until it is approximately 1/8" from the injector. Re-tighten the screws.

13. Lower the assembly to its lowest point without putting undue stress on the column or touching the injector body. Tighten the screws. If the exposed section of the column is more than 1/4" long, you may need to pack some insulation around it to bring more heat to the exposed area.

14. Pass the transfer line from the 7000 down through the rubber grommet on top of the Cryofocusing Module.

15. Slide the appropriate size nut and graphite vespel ferrule onto the fused silica transfer line. Make a straight (90°) cut across the transfer line (if fused silica) to remove shavings from the ferrule. Slide the fused silica transfer line into the top of the gold-plated union until it is positioned midway through the union. Tighten the nut and ferrule about 1/4 to 1/2-turn past finger-tight.

    Note: The goal is to achieve a low volume connection between the fused silica and the column. The two should meet each other in the center of the union, but not touch, or the stress may damage the ends of the tubing. Cut the tips of the fused silica and the column straight across, not angled.

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16. Turn on the carrier gas flow and leak check the union according to Section 4.7 of this manual.

17. Replace the inner and outer covers of the module.

Note: Supply pressure and line length influence cooldown time. Therefore, the line supply for the Cryofocusing Module must be within 20-70 psi at all times and the maximum cryo line length must not exceed five feet.

continued

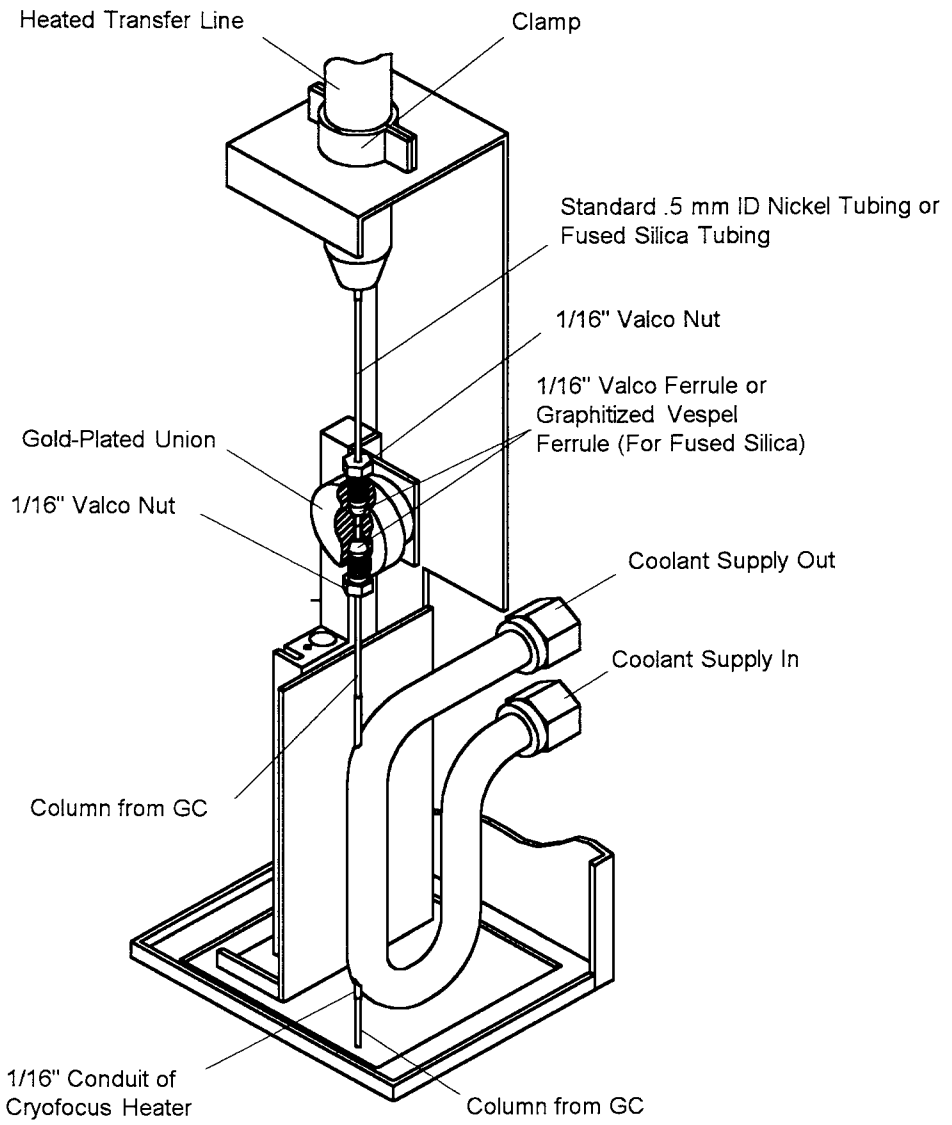


Figure 4-9 Cryofocusing Module

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Electronic Connections - Cryofocusing Module

To connect the Cryofocusing Module to the 7000 electronically you will need to install the Logic Board:

1. Turn the power off to the 7000 and unplug the unit.

2. Locate the electronics card cage -- it is on the left side of the 7000 (as viewed from the front). Remove the two 1/4-turn fasteners from the left side panel.

3. Pull the left side panel forward toward the front panel and away from the instrument.

4. Remove the cover from any unused expansion slot by unscrewing the hold-down nut.

   Note: All accessory boards can be damaged by static discharge. To prevent static damage, the boards are wrapped in anti-static bags.

5. Hold the board (still wrapped in its anti-static bag) in one hand and touch one of the expansion slots with the other hand to ground any potential static discharge.

6. Carefully remove the board from the bag.

   Note: Hold the accessory board by the bracket or edge of the board only. Avoid touching the components or connections to prevent damage from static discharge.

   Note: Any expansion slot with the exception of the bottom slot can be used for installing accessory boards.

7. Hold the expansion board by its edges or by the mounting bracket, and slide it into the slot until the card's edge or pin connector is fully seated.

8. Secure the board in place by reinstalling the hold-down nut.

9. Reinstall the left side panel by lining up the retaining clips to the posts in the expansion chassis.

10. Press the panel back onto the locating pins (on the rear panel).

11. Secure the panel with the two 1/4-turn fasteners.

continued

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12. Connect one end of the 15-pin D type logic interface cable (included with the Cryofocusing Module kit) to the Cryo Logic Board connector on the rear of the 7000. Connect the other end of the cable to the connector on the rear of the Cryofocusing Module.

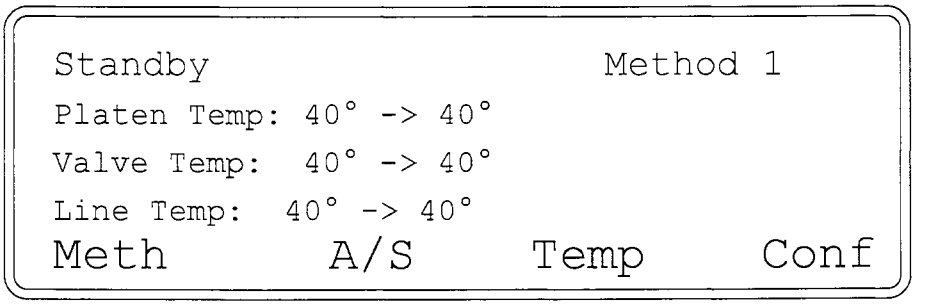
13. Connect one end of the two-pin connector to the rear of the main body of the Cryofocusing Module labeled "TO LN2". Connect the other end of this cable to the connector on the cryogenic valve.

14. Connect a power cord to the rear of the Cryofocusing Module.

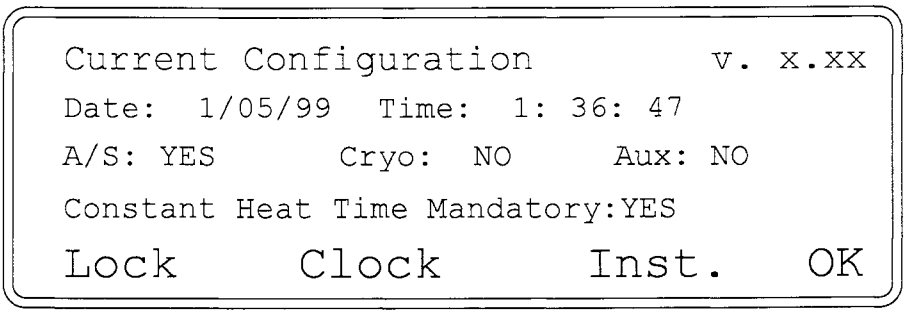
15. Plug in both main power cords for the 7000 and the Cryofocusing Module.

Configuring the 7000 to acknowledge the Cryofocusing Module: Once the Cryofocusing Module has been installed, you will need to configure the 7000 to acknowledge the Cryofocusing Module. To do this:

1. Power up the 7000 and the Cryofocusing Module. The 7000 will conduct routine self tests and then proceed to the Standby mode screen.



2. Press **F4 (Conf)**. The Configuration screen will appear:



3. On the Configuration screen, use the arrow key to move the highlighted cursor to Cryo. Press ENTER. Press Y to acknowledge the Cryofocusing Module.

4. Refer to Section 14.1, "Reviewing and Changing Instrument and Accessory Configurations" for additional information.

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Variable Injection Pressure Regulator (VIPR)

The Variable Injection Pressure Regulator (VIPR), p/n 14-5045-000, is a back pressure regulator module used to control the injection pressure of the headspace sample during the 7000 Loop Fill Mode. The VIPR offers more precise control of injection pressure in constant volume injections. A high VIPR setting yields a loop pressure that compresses the sample within the loop.

This compression leads to higher analyte concentration, and therefore, greater area count response (high sensitivity) from the detector. The extent of area count increase depends upon chromatographic and detector condition as well as vapor pressure of the analytes.

To install the VIPR:
On the rear of the VIPR are two bulkheads. One is labeled "INLET" and the other "OUTLET".

1. Remove the cap plug from the "INLET" bulkhead.

2. Connect the 1/8" nut and ferrule and the 1/8 - 1/16" reducer to this bulkhead (see the illustration below).

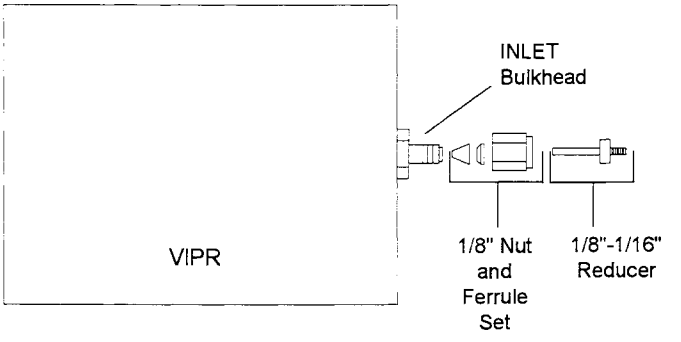


Figure 4-10 VIPR Connections

3. Remove the cap plug from the "OUTLET" bulkhead.

4. Connect a proper venting exhaust system (i.e. a hood) to this bulkhead with a length of 1/8" copper tubing (not included - p/n 14-0546-002) and the other 1/8" nut (p/n 14-0243-016) and ferrule (p/n 14-0241-016) set.

5. Connect 1/16" nickel tubing (p/n 14-5229-002) to the "VENT" bulkhead on the back of the 7000, using the 1/16" nuts (p/n 14-0243-016) and ferrules (p/n 14-0241-016). Connect the other end to the 1/16" end of the reducer assembly on the "INLET" bulkhead of the VIPR (see the illustration on the next page).

continued

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To set the VIPR to the desired pressure:

1. In the Standby screen, press **3** on the keypad and then press **3** again for **Vent Check**.

2. Insert an empty vial in position #1 of the 7050 Carrousel and press **LOAD**. This will allow pressurized flow out of the vent and to the VIPR.

3. Use the dial on the front of the VIPR to set the pressure to about 2-3 psi less than the vial pressurization settingon the 7000 (i.e. a 7 psi setting at the VIPR with a 9 psi setting on the 7000).

⚠ CAUTION

Note: Do not completely open the regulator. This could damage it, causing inaccurate readings or leaks.

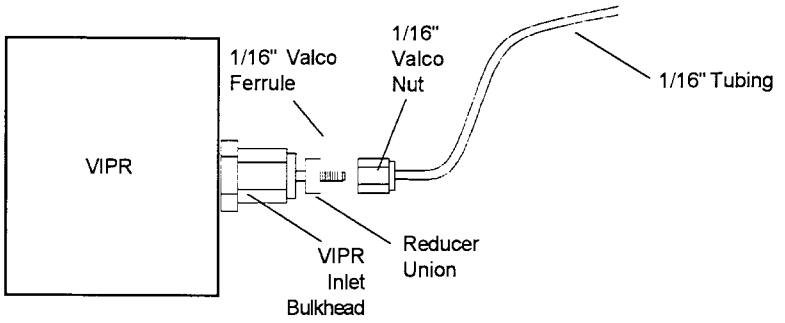


Figure 4-11 VIPR Connections

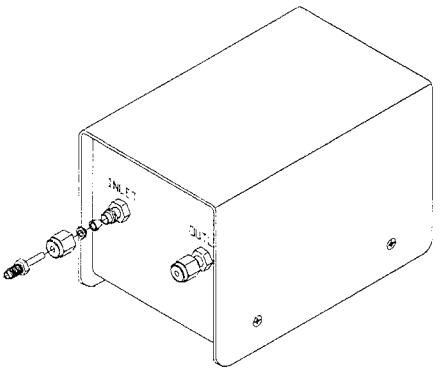


Figure 4-12 VIPR Back View

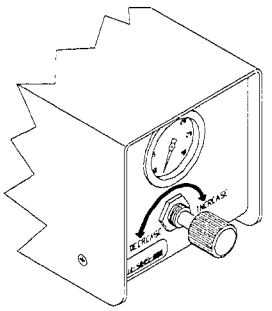


Figure 4-13 VIPR Front View

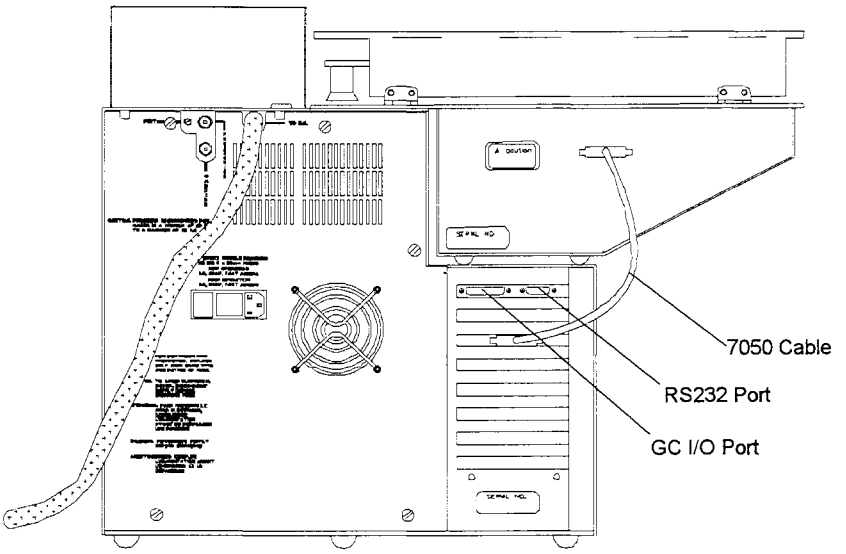
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4.4 Electronic Connections

Connecting the 7000 to the GC

Electronic connections are made through interface cables. These cables are specific to each particular GC make and model. Instructions for connecting a 7000 to a particular gas chromatograph accompany the interface cable. If you need to order an interface cable for your GC setup, call our Sales Support Department at (800) 543-4461; outside the U.S. at (513) 247-7000.

When you receive your cable, you will also need to set the DIP switches on the 7000 I/O board so that the 7000 will be able to acknowledge the GC. Installation instructions including DIP switch settings are included with every interface cable. If you do not have instructions, refer to the settings on the next two pages.



7000/7050 Rear View

continued

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Accessing the interface (I/O) board to set the dip switches

To access the I/O Board, remove the two 1/4-turn fasteners on the lower left side panel and slide the panel forward to release it from the studs.

⚠ WARNING

Electrical shock hazard inside this instrument.
Unplug power cord before servicing.

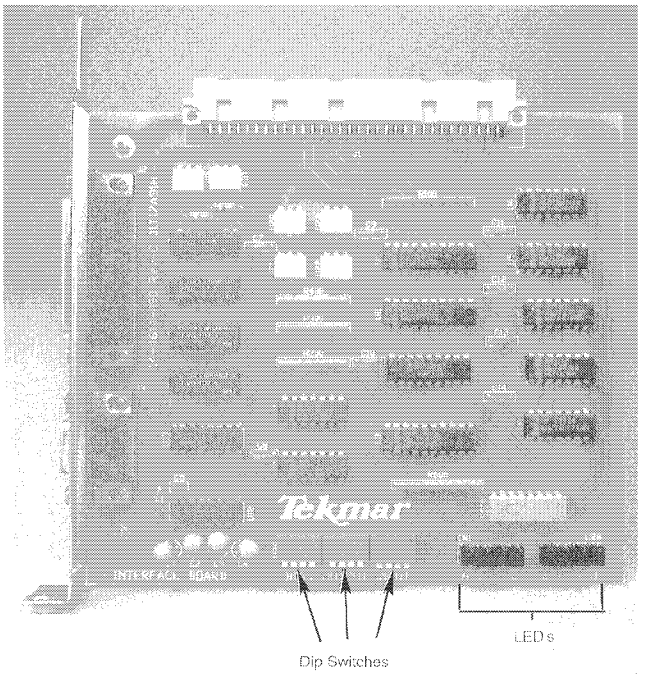


Figure 4-14 7000 Interface Board

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Setting the DIP Switches

The DIP switches that allow you to set up the GC interface port for communication to your specific GC are on the Interface Board. You should set these switches as specified on the instructions shipped with your cable.

There are three sets of four DIP switches on the 7000 I/O board. These DIP switches are labeled "BIAS", "OUTPUT", and "INPUT". The DIP OUTPUT switch controls output information to the GC. The BIAS and INPUT switches control signals from the GC to the CPU (central processing unit) of the 7000 microprocessor board. These settings must correspond to the particular configuration that your gas chromatograph/data system requires.

Find out if your GC's relay closure is normally open or normally closed when the GC is ready to start a run. Your gas chromatograph manual should contain this information. The corresponding 7000 DIP switch settings are on the next page.

If you want your 7000 to execute automated runs:

1. Press the **AUTO** key on the 7000.

2. Select the following dip switch positions on the I/O Board to yield a constant start signal:

   BIAS 4 = UP (open)
   INPUT 3 = DOWN (closed)

If you configure your I/O board this way, you won't have to press the Start key on your system when the 7000 is in Auto mode.

continued

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Here are the corresponding 7000 DIP switch settings:

	Signal	Signal Condition	DIP Switch Setting	LED
INPUTS	*Start*	Relay Closure N.O.	BIAS 4 open INPUT 3 open	F
		Relay Closure N.C.	BIAS 4 open INPUT 3 closed	F
		True Positive Signal	BIAS 4 closed INPUT 3 closed	F
		True Ground Signal	BIAS 4 open INPUT 3 open	F
	Continue	Relay Closure N.O.	BIAS 3 open INPUT 4 open	J
		Relay Closure N.C.	BIAS 3 open INPUT 4 closed	J
		True Positive Signal	BIAS 3 closed INPUT 4 closed	J
		True Ground Signal	BIAS 3 open INPUT 4 open	J
OUTPUTS	*System Ready*	Relay Closure N.O.	OUTPUT 3 open	A
		Relay Closure N.C.	OUTPUT 3 closed	A
	Inject Ready	Relay Closure N.O.	OUTPUT 2 open	B
		Relay Closure N.C.	OUTPUT 2 closed	B
	Inject	Relay Pins 21 & 22 TTL Pins 9 & 10 Closure N.O.	OUTPUT 4 open	C
		Relay Pins 21 & 22 TTL Pins 9 & 10 Closure N.C.	OUTPUT 4 closed	C
		Relay Pins 23 & 24 TTL Pins 11 & 12 Closure N.O.	OUTPUT 1 open	D
		Relay Pins 23 & 24 TTL Pins 11 & 12 Closure N.C.	OUTPUT 1 closed	D

LED is normally off but is on when active input is received.

LED is normally on, but is off when active output is available.

* Inject outputs are active at the **beginning** of Inject if the Cryofocusing Module is not installed and at the **end** of Inject if the Cryofocusing Module is installed.

Figure 4-15 DIP Switch Settings

continued

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Replacing the ROM

You may need to replace the microprocessor ROM chip to upgrade the operating parameters of the 7000. The accessories for the 7000 require a minimum version of 1.02 on the ROM. Versions 1.02 or greater will appear printed in the upper right hand corner of the Configuration screen. You can access this screen anytime by pressing **F4 (Conf)** when "Conf" is displayed on the bottom of the Standby screen.

Note: Make a hard copy of all your method parameters (other than default). The parameters will be lost and will need to be re-entered when a new ROM chip is installed.

Note: The following instructions assume that you will be working from the right side of the 7000.

To replace the ROM chip:

1. Turn off the power to the 7000 and unplug the power cord.

2. Remove the right side panel by unfastening the six 1/4-turn fasteners and pulling the panel away.

3. Locate the microprocessor. It sits directly behind the LCD display panel. You will be viewing the microprocessor from its right side.

4. Locate the ROM chip. Viewed from the right of the 7000, the chip is closest to you at the top of the microprocessor. It is held in by a blue ZIF (Zero Insertion Force) socket. There should be a label on the chip.

5. **Do not pull the chip out!** You will need to unlock the ZIF socket before removing the chip. Locate the levers on each side of the socket.

   a. Flip one of the ZIF levers down to release pressure on the pins. This will disconnect the ROM chip.

   b. Remove the chip by holding it by its sides and pulling it straight out away from the microprocessor.

Note: Before installing the new ROM, clear the program that was saved before you shut down the 7000. To clear the program, plug in the power cord and turn the instrument on for approximately 10 seconds. The unit will beep and the display will flash. Turn the instrument off and unplug the power cord. Proceed with ROM installation.

continued

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⚠ WARNING

Electrical shock hazard inside this instrument.
Replace the side panel before powering up.

6. Hold the ROM chip so that its u-shaped notch is farthest away from you, as viewed from the right side of the 7000. (Pin 1 will be in the upper right hand corner of the socket when viewing the chip from the rear of the 7000).

Note: The chip MUST NOT be installed with the notch oriented to the left side of the ZIF socket -- improper installation will destroy the chip!

7. Carefully align the pins with the sockets and push the chip in until it seats, being careful not to bend the pins.

8. While applying finger pressure, push the ZIF lever up until it clicks to lock the ROM chip in place.

9. Replace the side panel using the six 1/4-turn fasteners.

10. Plug in the power cord and turn on the instrument.

11. Proceed through the self tests (Section 12.3). If there are errors, refer to Section 13, Failure and Error Screens.

12. Reinstall your parameters (Section 8.7) after all of the self tests have been completed.

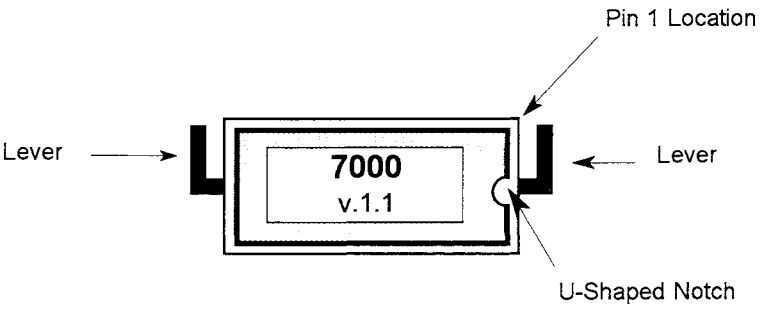


Figure 4-16 ZIF Socket/ROM Chip Orientation

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Installing Accessory Boards

This section contains instructions for handling accessory boards and installing them in the 7000. The microprocessor ROM may need to be upgraded when installing accessories. If so, see the previous two pages.

The electronics card cage is located on the left side of the 7000 (as viewed from the front).

1. Turn power off to the 7000 and unplug the unit.

> **⚠ WARNING**
>
> **Electrical shock hazard inside this instrument.**
> **Unplug power cord before servicing.**

2. Remove the two 1/4-turn fasteners on the left side panel.
3. Pull the left side panel forward toward the front panel and away from the instrument.
4. Remove the cover from any unused expansion slot (with the exception of the bottom slot) by unscrewing the hold-down nut.

   Note: All accessory boards can be damaged by static discharge. To prevent damage, the boards are wrapped in anti-static bags.

5. Hold the board (still wrapped in its anti-static bag) in one hand and touch one of the expansion slots with the other hand.
6. Carefully remove the board from the bag.

   Note: Hold the accessory board by the bracket or edge of the board only. Do not touch the components or connections.

7. Hold the expansion board by its edges or mounting bracket. Slide it into the slot until the board's edge or pin connector is fully seated.

   Note: You can install accessory boards in any expansion slot except the bottom one.

8. Secure the board in place by reinstalling the hold-down nut.
9. Reinstall the left side panel by lining up the retaining clips to the posts in the expansion chassis.
10. Press the panel back onto the locating pins (on the rear panel).
11. Secure the panel with the two 1/4-turn fasteners.

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TekLink™ Option

TekLink is a software program offered by Tekmar which allows you to automatically schedule and monitor up to four 7000 Headspace Autosamplers and their accessories from your PC in the Microsoft® Windows™ environment. The TekLink program displays all operation times and temperatures in "real time". It also offers 10 pre-defined methods for frequently-used applications which can be customized to meet your needs.

Some of the benefits of TekLink include:

- centralized control of GC accessories
- compatibility with most data stations and PCs
- storage and quick access of method parameters
- storage of methods in computer memory or to floppy disks

For more information about TekLink, or for a demo disk, please call Tekmar Sales Support at (800) 543-4461 or outside the U.S. at (513)247-7000.

RS232 Interface Option

The RS232 protocols option with ROM version 1.1 or greater lets you interface your 7000 with a data station/personal computer and gives you complete remote control over your 7000 operations.

RS232 requires ROM version 1.1 or higher and a cable (p/n 14-5106-186 for a 25-pin RS232 Interface Cable and p/n 14-5107-086 for a 9-pin cable).

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4.5 Installing the 7050 Carrousel

The diagram on the following page illustrates the 7050 Carrousel installation procedures.

> **⚠ CAUTION**
>
> **Turn off the 7000 before installing the carrousel to avoid damaging the equipment.**

1. Position the carrousel onto the 7000 platform and place its two locating pins into the two receptacle openings on the 7000.

2. Press the 7050 down gently to lock it into place.

3. Locate the 25-pin D type cable in the accessory pack shipped with the 7050.

4. Plug one end of this cable into the 25-pin female connector on the rear panel of the 7050. Plug the other end into the single 25-pin male connector on the back of the 7000.

Note: Your system is configured for 22 ml vials. If you plan to use 9 or 12 ml vials, you will need to install the 9 and 12 ml carrousel collar (p/n 14-4522-079) and the 9 and 12 ml platen inserts (p/n 14-4048-079). These are available in the kit (p/n 14-5083-000). Refer to Section 6 for more information on preparing the carrousel for vials. If you plan to use 22 ml vials, you may proceed with installation of the 7050 as described below.

To install the 50-position carrousel deck:

1. Position the deck over the turntable, centering it over the threaded stud.

2. Rotate the deck until the locating pin locks into the turntable.

3. Secure the deck with the holding knob found in the assembly kit box.

continued

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***To install the dust cover*:**

1. Remove the protective paper from the dust cover.
2. Install the hinges onto the dust cover using the four #10 nuts.
3. Install the standoffs onto the dust cover using two 6-32 x 3/8" screws.
4. Slide the dust cover hinge studs into the two sets of holes on the 7050 platform. Secure with four #10 nuts.

Note: If you need to return the 7050 to Tekmar for any reason, remove the dust cover before shipping.

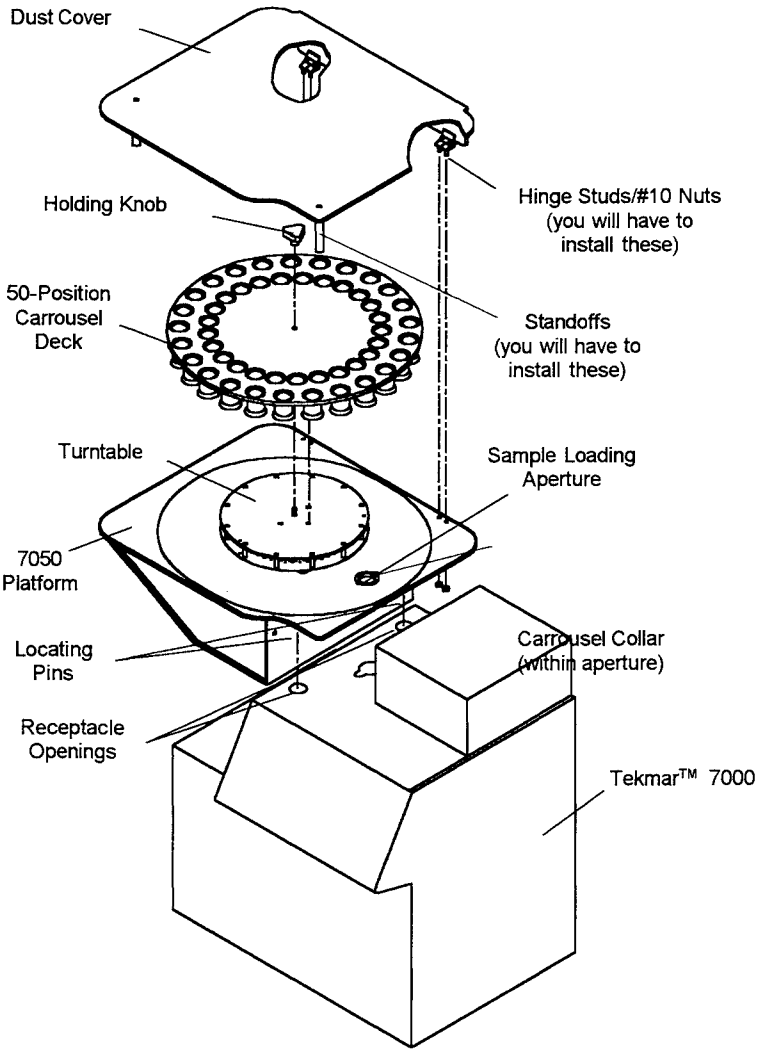


Figure 4-17 7050 Carrousel Installation

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4.6 Changing the Sample Loop and Sample Needle

This section gives instructions on changing the sample loop and sample needle.

The 7000 is shipped with a 1.0 ml sample loop installed. Other loops available are 0.1, 0.25, 0.5, 2 , 3, 5 ml.

To install alternate loops:

1. Open the valve oven cover.

> **⚠ WARNING**
>
> **When accessing the valve oven area, be certain that the valve is cool before opening the cover.**

2. Loosen the retaining clips and the nuts on each end of the loop (at ports 2 and 5 of the 6-port valve) and remove the loop.

3. Slide the new loop/adapter sleeve assembly over the mandrel base (required for 3 and 5 ml loops only). Connect one side of the loop to port 2 and the other side to port 5.

4. Use a 1/8" Allen wrench to tighten the adapter in place over the sample post.

5. Reinstall the retaining clips.

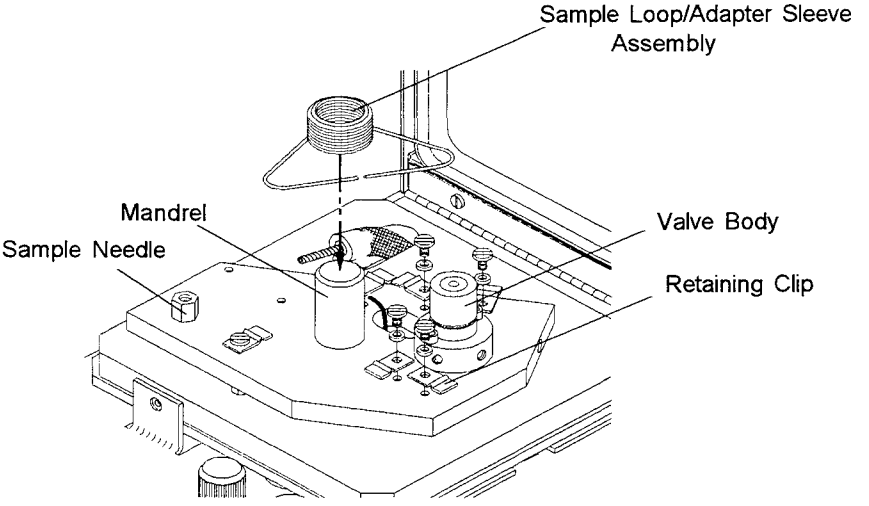


Figure 4-18 Valve Oven Area

continued

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Removing the sample needle:

1. Open the valve oven cover.

> **⚠ WARNING** 
>
> **When accessing the valve oven area, be certain that the valve is cool before opening the oven cover.**

2. Remove the sample line from the needle using a 5/16" wrench.
3. Unscrew the needle hub with a 3/8" open end wrench.
4. Pull the sample needle up and out of the valve oven area.

Installing a new needle:

1. Install the new needle in the mounting plate with a 3/8" wrench.
2. Reconnect the sample line to the needle hub and do a leak check (see Section 4.7 of this manual).

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4.7 Leak Checking the System

The 7000 is not a leak-prone system, however, it is very leak-sensitive. You should take special care before operating to ensure the system is leak tight. Check all fittings thoroughly.

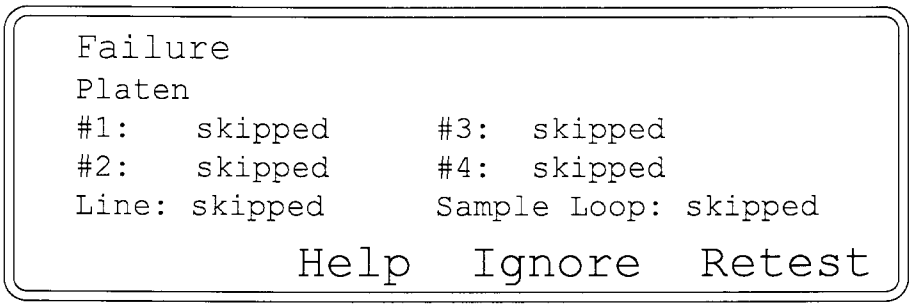
Note: Leak checking is most effective when performed with an electronic thermal conductivity detector (Tekmar p/n 21-0052-000).

Do not use soap solutions (e.g. Snoop™ or Detect) to leak check. If these solutions get into the lines, increased background and adsorption are likely to occur.

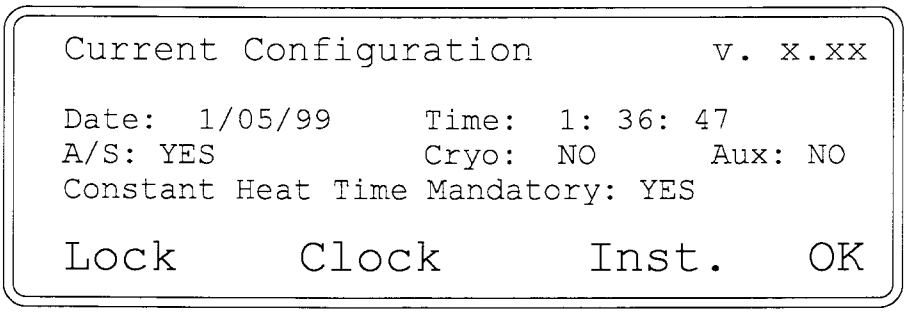
Electronic leak detectors do not work well when nitrogen is the pressurize or carrier gas. If possible, use helium when leak checking. If you do not have an electronic leak detector, you can use a 1:1 solution of iso-propanol:water on the suspect fitting, ***if done so sparingly***. If you have trouble locating a leak, please contact the Tekmar Service Department for assistance at (800) 874-2004, and locally or outside the U.S. at (513) 247-7000.

Accessing the Leak Check Mode:

1. Power up the 7000 and allow it to conduct the self tests -- or press **F4 (Skip)** to skip each of the self tests. If you choose **F4 (Skip)**, the following screen will appear:



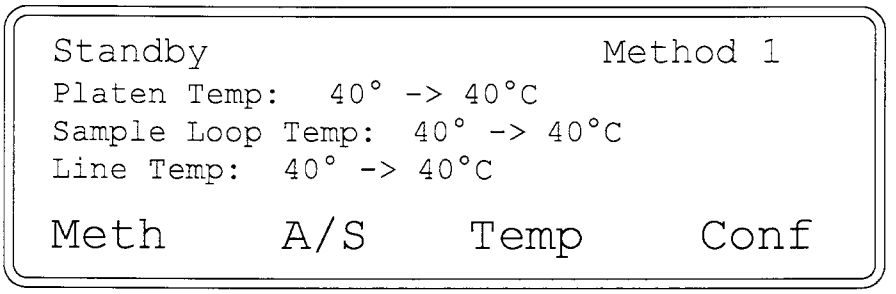
2. Press **F3 (Ignore)** to acknowledge that you intentionally skipped the self test. The 7000 then brings up the Current Configuration screen:



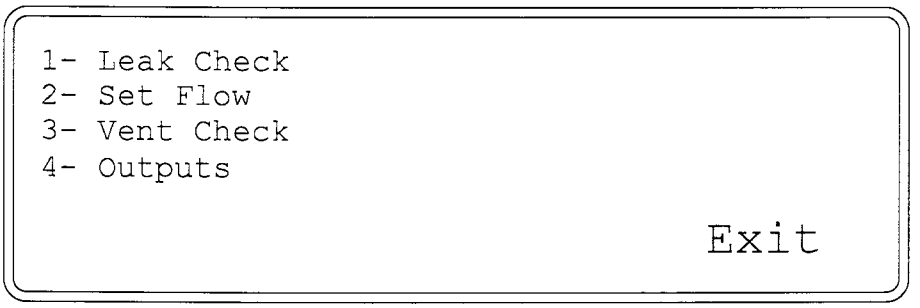
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3. From the current configuration screen, press **OK** to advance to the Standby screen.

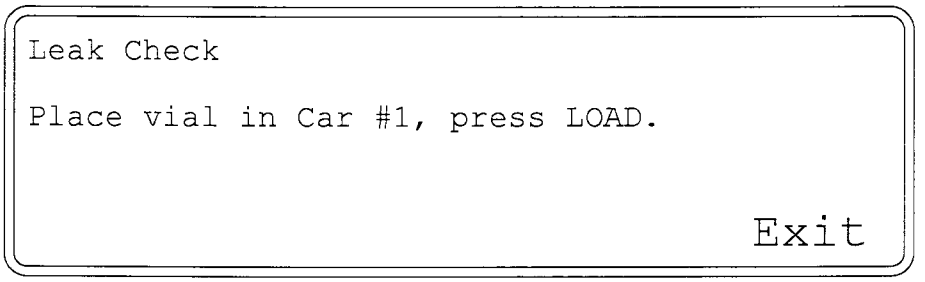


4. While in the Standby mode, press **3** on the keypad to access the Pneumatic/Output Diagnostic screen:



5. Prepare a properly sealed vial with the provided cap, septa and crimper **(see Section 6 for more information on vial preparation).**

6. Press **1** on the keypad for the Leak Check screen. ***If you have the 7050 installed,*** this screen will appear:



continued

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If you do not have the 7050 installed, the 7000 displays this screen:

```
Leak Check

Load vial, press LOAD


                                        Exit
```

7. ***Procedures to follow if you have the 7000 with the 7050:*** Place the vial in position #1, press **LOAD.**

8. ***Procedures to follow if you have the 7000 without the 7050:*** Place the vial in the platen and press **LOAD** and the following screen will appear:

```
Leak Check

Plug transfer & vent, then check


                                    Exit
```

9. Plug the transfer line and the vent on the back of the instrument with the cap nuts supplied in the installation kit.

10. Adjust the vial pressurization to approximately 10 psi. Wait 3-5 minutes for the system to pressurize. See Section 5.6 for information on setting vial pressurization.

11. Begin your leak check with a leak detector (Tekmar p/n 21-0052-000). Check all fittings in the valve oven area and in the right interior of the instrument.

continued

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12. After leak checking, press **F4 (Exit).** ***The following screen will appear when you are using the 7000 only.***

```
Leak Check
Remove vial


                              Exit
```

13. ***If you have the 7050 installed***, the unit will return to the Diagnostic screen automatically after you press **F4 (Exit)**.

14. ***If you do not have the 7050 installed***, remove the vial from the 7000 and press **F4 (Exit)** to return to the Diagnostic menu.

15. Press **F4 (Exit)** to get back to the Standby screen.

⚠ WARNING



Use extreme care when unloading hot vials from the platen chambers. Improper handling of hot vials may cause a burn.

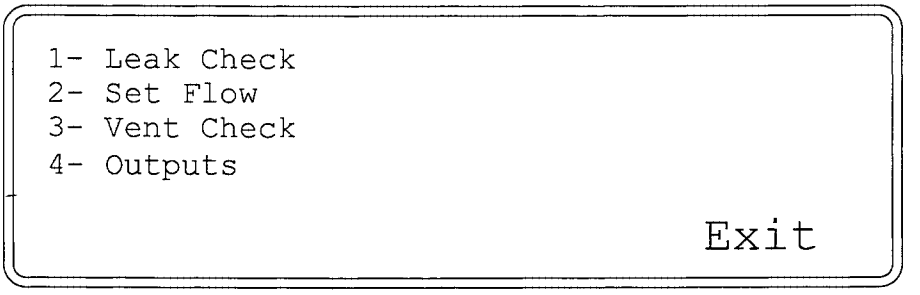
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4.8 Checking the Vent

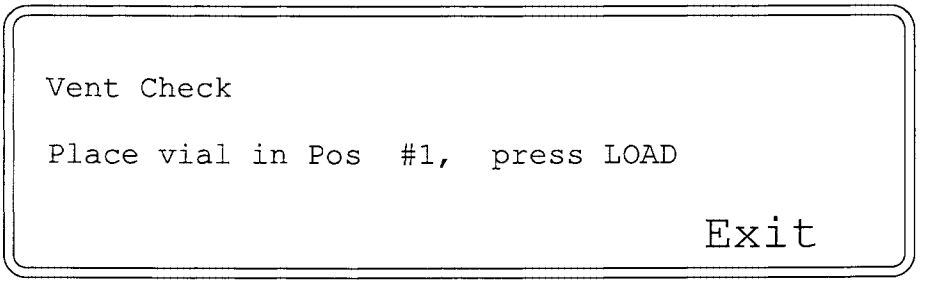
Check the vent outlet operation to verify that there is a clear flow path from the sample loop to the vent. The following instructions tell you how to use the vent check mode to check the vent.

To access the Vent Check Mode:

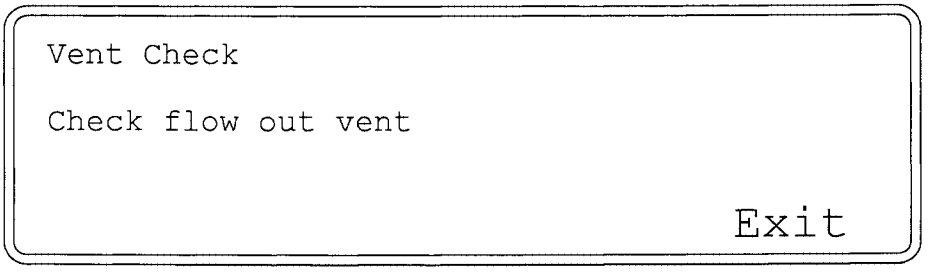
1. From Standby mode, press **3** on the keypad to access the Diagnostic screen:



2. Press **3** again for the Vent Check mode. This is the screen you will see ***when the 7050 is installed:***



3. This is the screen you will see ***when the 7050 is not installed:***



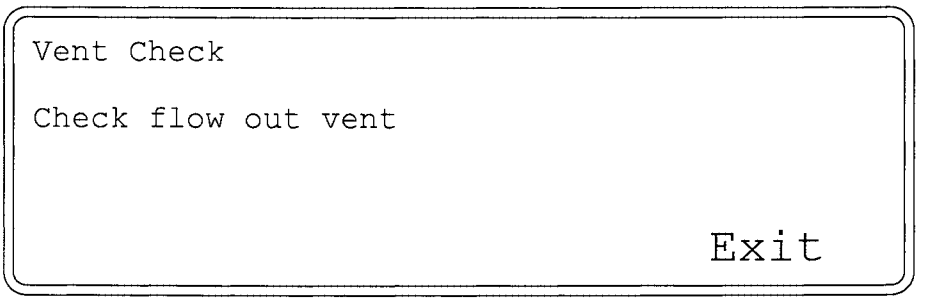
4. Prepare a properly sealed vial with the provided cap, septum and crimper. **See Section 6 for vial preparation instructions.**

5. Load a vial in the appropriate manner, depending upon whether or not you have the 7050 installed with the 7000 (see Section 6).

continued

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6. Press **LOAD** and the following screen will appear:



7. Turn the vial needle flow controller (on the top of the unit) to mid-range.

To check the vent:

1. Connect a flow meter (Tekmar p/n 13-0079-000) to the VENT bulkhead on the back of the 7000.

2. Turn on the flow meter.

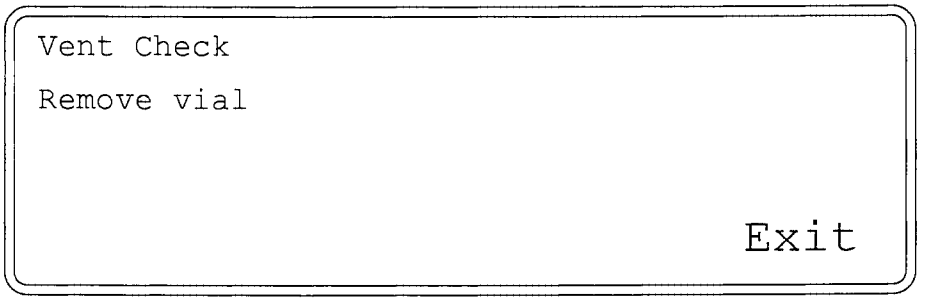
3. Make sure the flow rate on the flow meter is the same as the desired pressurization flow (see Section 5.6, Setting Vial Pressurization).

4. Measure the flow rate.

5. Press **F4 (Exit)**. The 7000 will wait 15 seconds before exiting.

6. ***If you have the 7050 with the 7000***, the vial will automatically unload and the control panel will return to the Diagnostic screen.

7. ***If you don't have the 7050 installed,*** the following screen will appear:



8. Remove the vial by pressing **LOAD/UNLOAD** (***for the 7000 only***) then press **F4 (Exit)** to return to the Diagnostic screen.

Note: If you cannot measure flow at the vent, call the Tekmar Service Department at (800) 874-2004 or (513) 247-7000 (locally or outside the U.S.).

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5.1 Overview of the Section

This section of the manual provides information on and procedures for setting pneumatics on the 7000.

The 7000 Headspace Autosampler is built on the "valve and loop" sampling approach. It has separate (and independent) gas flow systems for vial pressurization and carrier gas functions. Separating pressurization and carrier gases lets you optimize each flow system individually for the best possible analytical results.

The flow diagram (Figure 5-1) on the next page shows the 7000's gas flow system. Gases enter through two inlets labeled "CARRIER" and "PRESSURIZE".

From the ***carrier*** inlet on the back of the 7000, gas is directed through a flow controller (**F2**). A pressure gauge (**G2**) measures pressure in this line. The flow passes into the heated valve oven section (**H1**) through the 6-port valve (**V6**), through the heated transfer line (**H2**), and into the GC. This provides the column with carrier gas at a rate set at **F2**. The back pressure of the column can be read at **G2**.

Note: When the 7000 is controlling flow to the column, the carrier supply line must be regulated to 40-50 psi from the carrier source (i.e. stage regulator on the supply tank). The flow elements supplied with the 7000 are calibrated against 40-50 psi for accurate operation.

You can also use the GC flow controller to regulate column carrier flow. You do this by connecting the 7000 carrier inlet to the regulated supply line coming from the GC inlet pneumatics. You'll need to deactivate the flow controller (**F2**) by turning it to its fully open position.

Note: When using the GC flow controller to regulate carrier flow, Tekmar also recommends that you install the 40-400 cc flow element (p/n 14-4928-050) to reduce backpressure created by the flow controller. See Section 4 for information on installing the flow element.

continued

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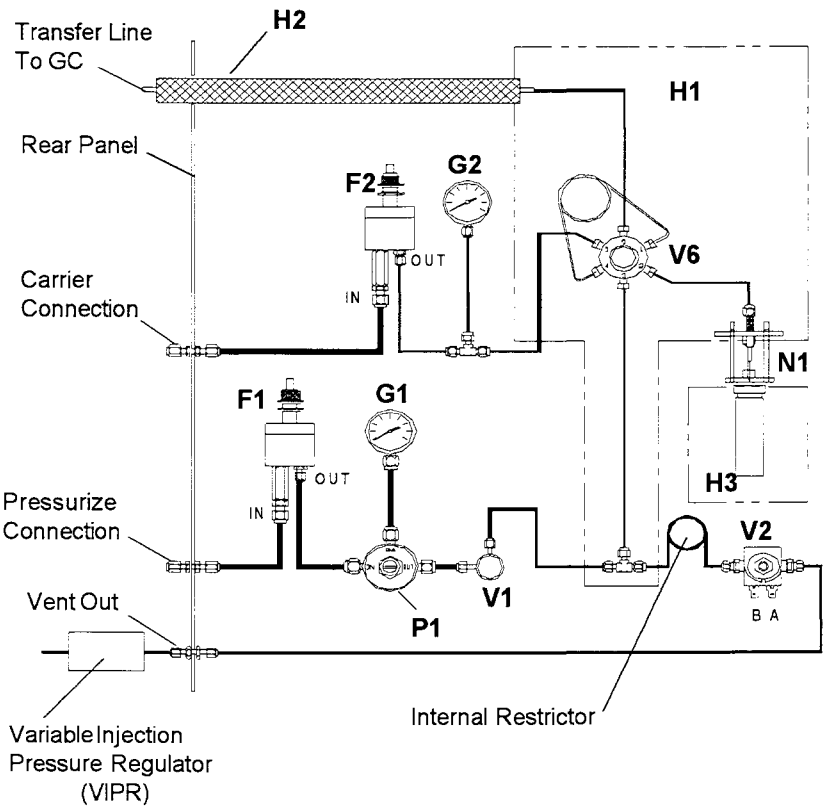


Figure 5-1 7000 Flow Diagram

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5.2 Pneumatic Controls and Gauges on the 7000

Following is an overview of the pneumatic controls located on the top panel of the 7000.

Vial Needle Flow Control - adjusts the amount of gas sweeping through the 7000 to pressurize the vial and to reduce carryover between injections.

Transfer Line Flow Control - controls gas flow through the transfer line. May also be used for total column carrier flow -- with interchangeable flow elements to accommodate different flow rate ranges. **Note: The GC can also control column flow. In this case, the transfer line flow controller is completely opened.**

Vial Pressurization Control - adjusts the final vial pressure after the vial is heated.

Vial Pressurization Setting Gauge - indicates the vial pressure before injection onto the GC.

Transfer Line Back Pressure Gauge - indicates transfer line and/or GC column back pressure.

continued

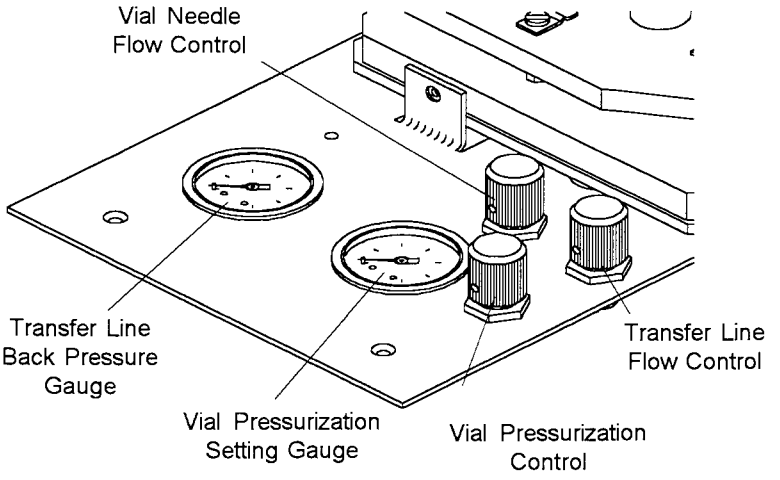


Figure 5-2 Pneumatic Controls and Gauges

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The 7000 flow and pressure pneumatics control panel on the top of the instrument allows you to independently regulate the following:

- **Column carrier flow (ml/min)*** - Use the Transfer Line Flow Controller.
- **Vial Needle/Standby Flow (ml/min)** - Use the Vial Needle Flow Controller.
- **Vial Pressurization (psi)** - Use the Vial Pressurization Controller and Vial Pressurization Setting Gauge.

***Note: The transfer line flow may be controlled by the 7000 or by the GC (see Section 4 of this manual).**

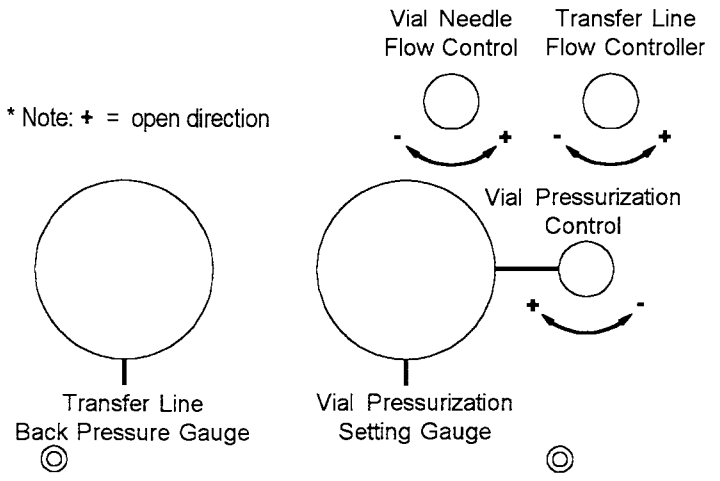


Figure 5-3 7000 Pneumatic Controls

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5.3 Principles of Operation - Column Carrier Flow

Column carrier flow may be regulated from the 7000 or GC flow control or by backpressure regulator pneumatics (see Section 4 of this manual). The following chart summarizes these pneumatic control options.

Instrument	*Column Flow Control (Back Pressure-Regulated)*	*Column Flow Control (Flow Controller-Regulated)*
Tekmar™ 7000	Yes (with optional external pressure regulator assembly - p/n 14-3938-000).	Yes (for both capillary and packed column flow rates. Select optimal flow element range for best precision - see Section 4).
GC	Yes (usually for capillary column flow rates - see pages 4-11 to 4-12).	Yes (usually for wide bore capillary and packed column flow rates - see pages 4-8 to 4-10).

Column carrier flow rate and back pressure are the gas flow rate and back pressure generated by the GC column. Optimal flow rates and back pressure depend upon the column geometry and desired separation. Common column sizes, flow rates and their resulting back pressures are illustrated below:

Column Geometry	*Flow Rate*	*Back Pressure*
I.D. = 1/4" - .1mm Length = 60 m - 6 ft.	40 ml/min - 1ml/min	40 psi - 4 psi

The 7000 allows you to use any column that falls within the above categories.

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5.4 Principles of Operation - Vial Needle Flow

Vial needle flow is the flow rate of the pressurization gas as it sweeps the loop and exits the side port of the vial needle (see Figure 5-4). This flow has two primary functions: 1) to reduce carryover; and 2) to pressurize the vial. These functions are explained below:

Function 1:

Vial needle flow sweeps the sample path between injections to reduce sample-to-sample carryover. How well carryover is reduced or eliminated through Standby vial needle flow depends upon the total sweep volume between injections. Total sweep volume is calculated by:

vial needle flow rate (ml/min) x time between injections (GC cycle time = sweep volume ml)

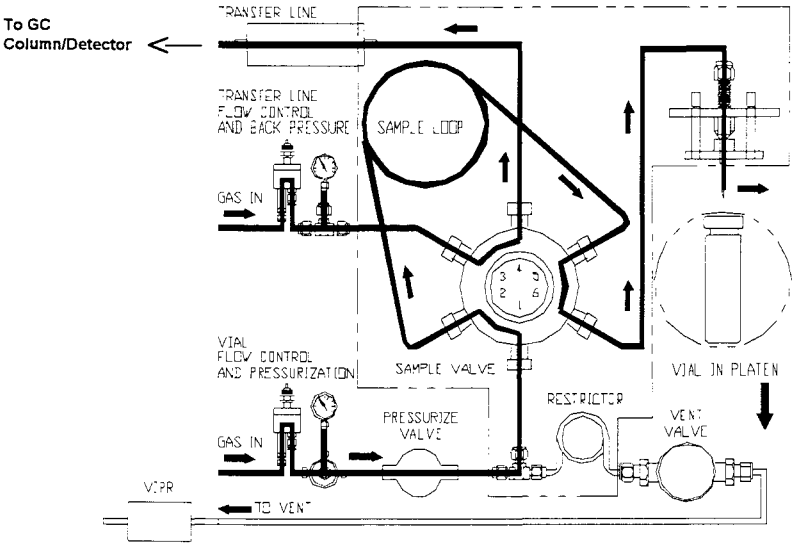


Figure 5-4 Valve Configuration of the 7000 in Standby Mode

continued

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For an average analysis, 800 ml sweep volume will reduce carryover. The following chart shows the relationship between flow, GC cycle time, and sweep volume.

Vial Needle Flow	*GC Cycle Time*	*Sweep Volume (approx.)*
10 ml/min	80 min	800 ml
15 ml/min	53 min	800 ml
20 ml/min	40 min	800 ml
30 ml/min	27 min	800 ml
40 ml/min	20 min	800 ml
60 ml/min	13 min	800 ml

A given analysis may require a low pressurization setting and a high vial needle flow rate, such as 5 psi vial pressurization and a 5-minute GC cycle time. This will yield a vial needle flow rate of 160 ml/min to provide an 800 ml sweep volume. If you separate vial pressurization from vial needle flow, you can modify these two parameters independently.

Function 2:

The second function of vial needle flow is to contribute to the rate at which pressurization gas is introduced to the vial. As the vial reaches the assigned pressurization setting (in psi), the vial needle flow rate drops from its Standby rate to zero. Refer to Figure 5-5.

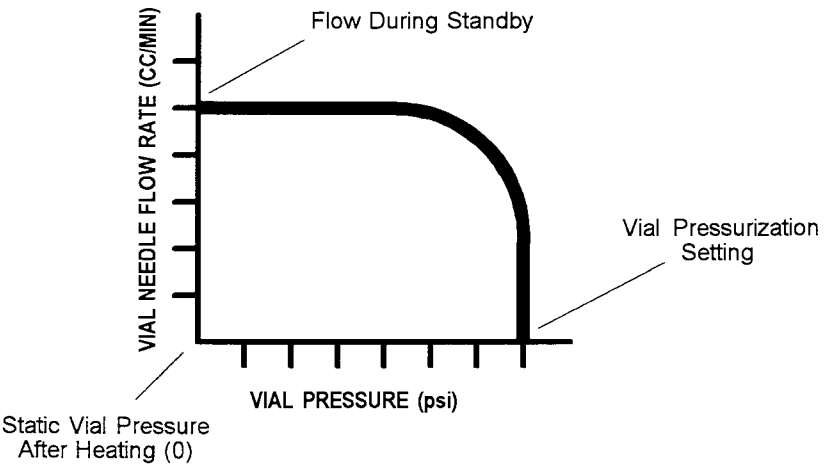


Figure 5-5 Vial Needle Flow Rate and Vial Pressure

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5.5 Principles of Operation- Static Vial Pressure

When certain samples are heated, pressure builds in the vial. This pressure is referred to as the **static vial pressure**, which exists ***before*** the vial is raised onto the needle.

Static vial pressure is directly related to the temperature of the vial and the boiling point of the solvent in the vial. However, in some cases (particularly with dry solids) heating causes insignificant static vial pressure, keeping the pressure at or near zero. Note that the vial pressure axis in Figure 5-5 on the previous page does not start at zero.

Some samples, although they are of the same type, may show slightly different static vial pressures after being heated at a given temperature for a specified period of time. Therefore, be sure to consider static vial pressure for the following reasons:

- Sample-to-sample static vial pressure variances may lead to different loop flush values. In these situations you may be able to achieve more precision in your analysis by adding 2 - 10 psi vial pressure above the static vial pressure. This ensures a uniform flush of the sample loop during the Loop Fill step. To adjust vial pressurization, use the Vial Pressurization Control knob on the pneumatics control panel on top of the 7000 (see Figure 5-2).

- The pressure within the vial is used to expand the headspace atmosphere into the loop and out of the vent before injection (see Figure 5-6). For maximum precision, there must be enough pressure in the vial to flush the sample loop with ***at least twice the sample loop's volume.***

- For samples that do not have enough static vial pressure to expand twice the total loop volume, you will need to add more pressure to the sample vial to yield a proper loop flush. Additional pressurization may also be needed to fill larger loop volumes.

In general, increasing vial pressure improves precision and in some instances sensitivity when:

- The vial static pressure is too low to flush twice the loop volume during the Loop Fill step (precision).

- You are using larger sample loops which require larger vial exhaust volumes for reproducible loop fills (sensitivity).

continued

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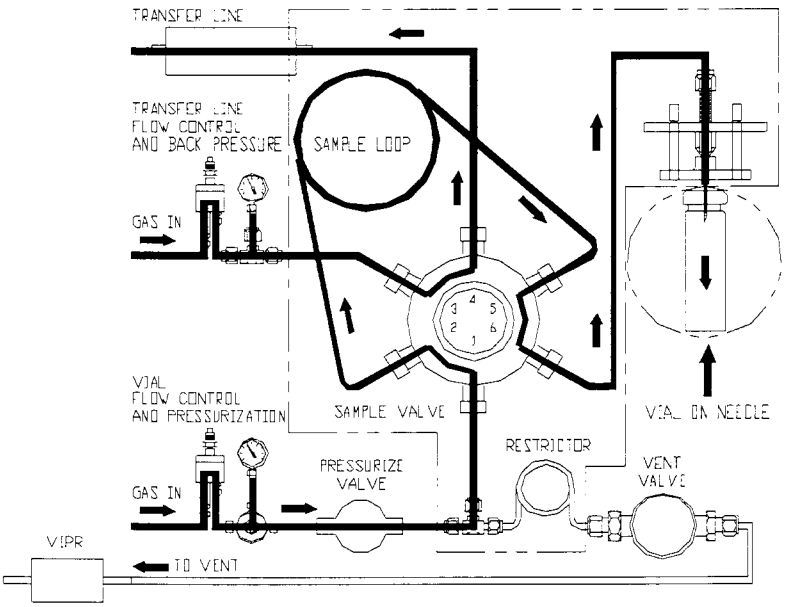


Figure 5-6 7000 Flow in Vial Pressurization Mode

To determine static vial pressure:

1. Turn the vial needle flow controller completely off.
2. Set up a method for your application.
3. Place a vial containing your sample into the carrousel.
4. Start the run, letting the sample equilibrate for the same amount of time and temperature that the sample will be run.
5. When the vial is pressurized, record the pressure that the Vial Pressurization gauge reaches. This is the static vial pressure.

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5.6 Setting Vial Pressurization

Set vial pressurization about 2 - 4 psi above the static vial pressure.

To set the vial pressurization:

1. Press **3** on the keypad from the Standby Mode screen.
2. Press **4 (Outputs)** on the keypad.
3. Open the Vial Needle Flow Control knob (by turning clockwise). The precise needle flow will be set later.
4. **PAGE DOWN** until you see "Press" for Vial Pressurization.
5. Press **"Y"** on the keypad for "yes" to turn on the pressurization valve.
6. Dial in the appropriate vial pressurization setting using the Vial Pressurization Control knob and pressurization setting gauge on the pneumatics control panel.
7. You can adjust the vial pressurization setting by pressing **"Y"** to turn the valve on or **"N"** to turn the valve off.

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5.7 Setting Standby Vial Needle Flow Rate

To determine the optimal Standby vial needle flow rate, see Section 5.4 of this manual.

To set the Standby vial needle flow rate:

1. Use a 1/16" union to attach a flow meter (p/n 13-0079-000) to the VENT bulkhead on the back of the 7000.
2. Press **3** on the keypad while in the Standby screen.
3. Press **3**, Vent Check.
4. Place an empty, sealed vial in the Carrousel and push "Load".
5. Dial in the correct vial needle flow rate using the Vial Needle Flow Control knob on the 7000 pneumatic panel. Note the flow rate from the vent on the flow meter.
6. Measure the flow coming out of the vent.

Your vial needle flow and vial pressurization settings should now be properly set.

5.8 Setting the Variable Injection Pressure Regulator

The Variable Injection Pressure Regulator (VIPR) is an optional accessory for the 7000 that is designed to increase sensitivity. Install the VIPR according to the instructions in Section 4.

To set the VIPR:

1. Press **3** on the keypad while in Standby mode.
2. Press **3**, Vent Check.
3. Place an empty, sealed vial in the Carrousel and press "Load".
4. Adjust the pressure using the knob on the front panel of the VIPR.

   Note: Generally, the VIPR is set 2 - 3 psi lower than the Vial Pressurization setting.

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6.1 Overview of the Section

Section 6 explains how to prepare and load vials into the 7000/7050.

6.2 Preparing the 7000 for 9 ml and/or 12 ml Vials (with 7050 Installed)

Note: If you are using the 7000 without the 7050 Carrousel, please refer to Section 6.4 - Preparing the 7000 for 9 ml and 12 ml Vials without 7050.

Note: Sample weight should not exceed approximately 40 grams per vial.

Note: The 7050 Carrousel is pre-set to accept 22 ml vials into the platen. If you plan to use 9 ml and/or 12 ml vials, complete the following procedures.

Change the carrousel collar in the 7050 sample loading aperture to reduce the hole size to accommodate the smaller vials. Then install platen sleeve inserts in the 12 heater chambers of the sample platen to ensure the 9 ml and 12 ml vials fit securely in the platen.

To change the collar and install the platen sleeve inserts:

> **WARNING**
>
> **Turn the instrument off and allow the platen to cool before proceeding.**

1. Lift the dust cover.
2. Unscrew the holding knob on the 7050 Carrousel deck and remove the carrousel deck from the turntable.
3. Locate the 22 ml carrousel collar in the sample loading aperture on the 7050 base plate. It is held in by two screws.
4. Remove these screws, then remove the existing collar from the base plate.

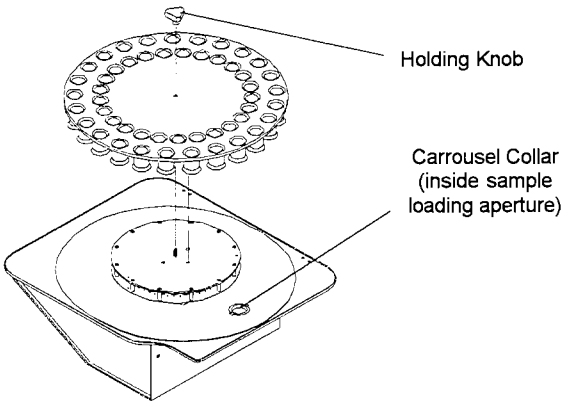


Figure 6-1 Carrousel Collar Location

continued

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5. Use the provided insert removal tool (p/n 14-4365-027) to install a platen sleeve insert into the first heater chamber of the platen.

6. To move the platen to each position, press **F2 (A/S).** The following screen will appear:

```
Automatic Sampler Control
Current  position: 1   Platen: 3
Start:  1              Stop:  50
Enable Method Schedule : N

Help       Sched     - >     Exit
```

7. Press STEP on the keypad to advance the platen to the next position.

8. Install the next insert into the next chamber.

9. Repeat this procedure until all 12 inserts are installed.

10. Position the 9/12 ml carrousel collar in the sample loading aperture on the base plate and secure it with the two screws.

11. Position the carrousel deck over the turntable, centering it over the threaded stud.

12. Rotate the carrousel deck until the locating pin locks into the turntable.

13. Secure the 7050 deck to the base with the holding knob.

continued

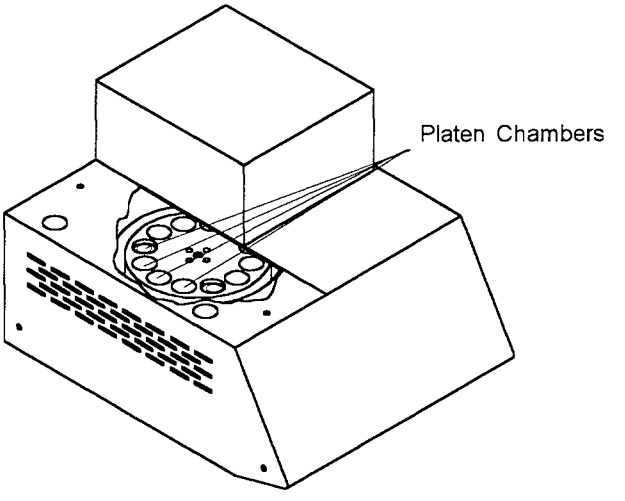


Figure 6-2 Platen Chambers

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> **⚠ CAUTION**
>
> **Be sure that all 12 chambers contain sleeves. Operating the instrument without a sleeve insert in each chamber may cause a platen or loader error which will abort runs.**

14. You can now use either 9 ml or 12 ml vials. The system will not accept 22 ml vials with the platen sleeves installed. To prepare the system for 22 ml vials, please refer to Section 6.3.

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6.3 Preparing the 7000 for 22 ml Vials (with 7050)

Note: 22 ml vials are used alone -- not in combination with any other vial size. You do not need the platen sleeve inserts when using 22 ml vials.

Note: The 7000 is shipped with the 22 ml vial carrousel collar. If you plan to use 9 ml and/or 12 ml vials, read the following instructions.

Here are instructions for installing the 22 ml carrousel collar and removing the platen sleeve inserts if at some time the system was configured for 9 and 12 ml vials and you want to switch back. You remove the 9 ml and 12 ml sleeve inserts from the platen automatically using the 7000 keypad.

To re-configure for 22 ml vials:

1. Lift the dust cover.

2. Unscrew the holding knob on the 7050 Carrousel deck and remove the deck from the unit's turntable.

3. Locate the 9/12 ml carrousel collar inside the sample loading aperture on the top of the base plate. It is held in by two screws (see Figure 6-1).

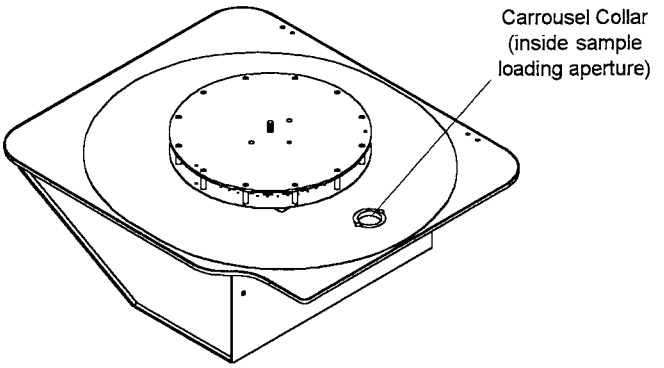


Figure 6-1 Carrousel Collar Location

4. Remove these screws, then remove the carrousel collar from the 7050 base plate.

5. Remove the first sleeve insert from the heater chamber of the platen with the insert removal tool (p/n 14-4365-027) provided in the insert kit (p/n 14-5083-000).

6. To automatically move the platen to each position, press **F2 (A/S)** then press **STEP** on the keypad.

continued

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7. Step the platen to the next position and remove the next sleeve insert from its chamber with the insert removal tool.

8. Repeat until all 12 sleeves are removed.

> **⚠ CAUTION**
>
> **Be sure that all of the sleeves are out of the platen. If any are left, a platen or loader error could occur, which will abort runs.**

9. Position the 22 ml carrousel collar in the opening of the base plate and secure it with the two supplied screws.

10. Position the 7050 deck over the turntable, centering it over the threaded stud.

11. Rotate the deck until the locating pin locks into the turntable.

12. Secure the deck with the holding knob.

> **⚠ WARNING** 
>
> **Always use the insert removal tool (Tekmar p/n 14-4365-027) when removing sleeve inserts from the platen heater. The inserts remain the same temperature as the platen heater and may cause a burn if handled improperly.**

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6.4 Preparing the 7000 for 9 ml and/or 12 ml Vials (without 7050)

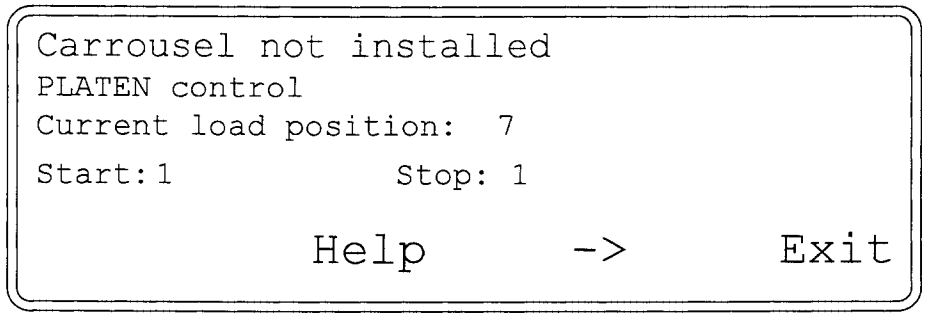
Before operating the 7000, you will need to determine the vial size(s) you are going to use and how to configure the instrument accordingly:

Note: 9 and 12 ml vials may be combined using the 7000 method scheduling function. Sleeve inserts are required to use 9 and 12 ml vials in the 7000 12-position platen.

Following are the instructions for installing sleeve inserts in the 12 heater chambers of the sample platen. You will need to use the microprocessor to complete the installation.

1. Using the provided insert removal tool (p/n 14-4365-027), install a sleeve adapter into the first heater chamber of the platen.

2. Press **F2 (A/S)** to move the platen to each position. The following screen will appear:



3. Press **STEP** on the keypad to advance the platen to the next position.

4. Install the next sleeve insert into that chamber.

5. Repeat the procedure until all 12 inserts are installed.

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6.5 Preparing the 7000 for 22 ml Vials (without 7050)

The 7000 is set up at the factory to run 22 ml vials. The following instructions explain how to reconfigure the 7000 for 22 ml vials if you set it up at one time to accept 9 and 12 ml vials.

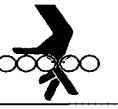
Note: The 22 ml vials are to be used by themselves and not combined with any other vial size. Do not attempt to use the sleeve inserts when running 22 ml vials.

To configure the 7000 for 22 ml operation (and to remove the sleeve inserts):

1. Press the **A/S** key and note the current position.
2. Use the insert removal tool (p/n 14-4365-027) to remove the insert from the first platen position.

> ⚠ **WARNING** 
>
> **Always use the insert removal tool (Tekmar p/n 14-4365-027) when removing sleeve inserts from the platen heater. The inserts remain the same temperature as the platen heater and may cause a burn if handled improperly.**

3. Press the **STEP** key to advance the platen to the next position.
4. Remove the insert and repeat until all 12 positions are without inserts.

> ⚠ **WARNING** 
>
> **The platen may rotate and can pinch or grab fingers. Be sure to use the insert removal tool to remove sleeve inserts.**

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6.6 Preparing Vials for Sampling

A properly capped and sealed vial is critical in maintaining sample integrity during headspace autosampling. Following are guidelines for proper vial preparation.

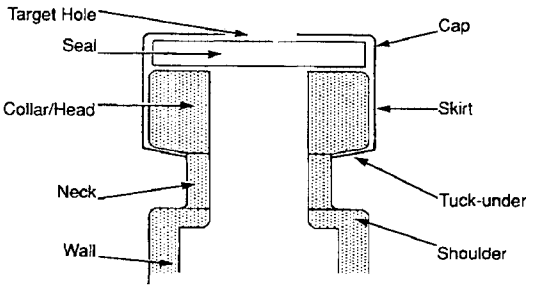


Figure 6-3 Properly Sealed Vial

When a vial is **undercrimped**, the cap shape will be concave and the cap can be easily rotated. The "tuck-under" on the vial will be at an angle to -- not in contact with -- the underside of the collar. If you feel some resistance when trying to rotate the cap, it is probably sufficiently crimped.

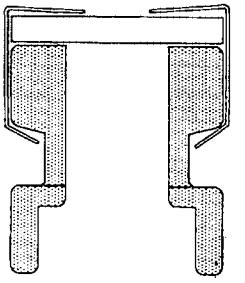


Figure 6-4 Undercrimped Vial

continued

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An **overcrimped** vial may cause a cored septum or bent needle. The hole in the teflon face may be 50% larger than in a properly crimped seal.

In an overcrimped vial, the top surface of the vial will be convex and may have a small, raised ridge around the edge. The seal will be partially extruded from the target hole in the vial and have a concave shape. In some cases, the skirt of the vial may have pleats around the collar.

If the seal is only slightly overcrimped, the small, raised ridge may be the only symptom. This can be corrected by reducing crimp pressure.

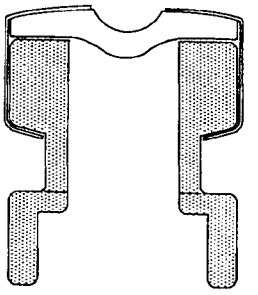


Figure 6-5 Overcrimped Vial

Several factors affect the quality of a seal:

- Seal/vial variations
- Amount of effort needed to make the seal
- Quality of the crimping tool

High quality glass vials may have a tolerance of ± 0.2 mm collar height and the seal may range in thicknesses of 2.5 mm to 3.0 mm. Tekmar recommends that you use an adjustable crimping tool to allow for these variations (Tekmar p/n 14-4863-027).

Hand or plier-type crimpers accommodate varying seals and vial thicknesses, but require more effort than automatic crimpers, especially when you have a lot of vials to crimp at one time.

Tekmar recommends the Crimp Station (Tekmar p/n 14-4865-027) to efficiently cap and seal vials. It has steel jaws that provide more stability.

For more information on vial preparation or for product information, please call Tekmar at (800) 543-4461 or (513) 247-7000 locally and outside the U.S.

continued

Information on pages 6-6 and 6-7 courtesy of Chromacol Ltd.©

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To prepare vials for sampling:

1. Place the cap on a firm surface.

2. Insert the septum into the cap (avoid contamination of the septum face -- the shiny silicone face should always be face down toward the vial.)

3. Place the septum and cap together on top of the vial opening.

4. Place the crimper on top of the capped vial with the crimp head resting on the cap.

5. Squeeze the crimper handles firmly in a slow and smooth motion -- do not squeeze quickly.

6. Remove the crimper.

7. Check for a proper crimp seal by lightly rotating the cap. If some resistance exists, it is properly sealed. See the additional information on crimping and crimp tools on pages 6-7 and 6-8.

Note: Sample weight should not exceed approximately 40 grams per vial.

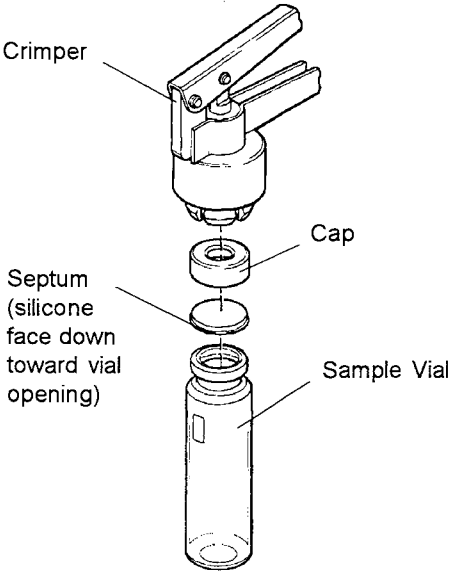


Figure 6-6 Hand Crimper & Vial

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6.7 Loading Vials Into the 7000 (without 7050 Installed)

To load vials into the 7000 platen when the 7050 Carrousel is not installed:

1. Make sure the platen is prepared to accept the vial size you are loading. Refer to Sections 6.1 and 6.2 before proceeding.

2. Press **F2 (A/S)** in any operating mode and the following screen will appear:

```
Carrousel not installed
PLATEN control
Current load position: 7
Start: 1      Stop: 1

          Help      ->          Exit
```

Current load position (e.g. position 7) designates which opening in the platen is in the loading position. **Start** and **Stop** designate which platen chambers you wish to start on and stop on. You will need to adjust all three of these parameters before loading vials into the platen.

Setting your start and stop positions:

1. Press **F3 (->)** to move the highlighted box to the **Start** parameter and press **ENTER.**

2. Enter the appropriate number, press **ENTER** again and repeat for the **Stop** parameter.

continued

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Setting the current load position:
To move the **current load position** to your chosen **start** position:

1. Press **STEP** to move the platen forward or press " ±" on the keypad to make the platen move in reverse to the start position. For a 12-position load, the screen will look like this:

```
Carrousel not installed
PLATEN control
Current load position:  1
Start: 1          Stop: 12
        Help        ->        Exit
```

To load vials into the platen:
1. Press **LOAD/UNLOAD**. The vial ram will raise up through the platen and stop just short of the top of the platen.

2. Place a prepared vial in the opening.

3. Press **LOAD/UNLOAD** again to lower the vial into the platen. The vial ram will lower the vial into the platen position, the platen will rotate to the next position, and the vial ram will raise up again to accept the next prepared vial.

4. Repeat these steps for all designated **Start** to **Stop** positions.

Using STEP in the LOAD/UNLOAD sequence:
This option offers you more control during your loading operations. Press **STEP** to lower the vial ram (when it is in the raised position) and advance the platen (without having to raise the vial ram back to the load position. **STEP** will also advance the platen to a particular position without going through the load/unload sequences.

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6.8 Loading Vials into the 7000 (with 7050 Installed)

1. Manually place the desired size and quantity of prepared vials into the appropriate 7050 Carrousel positions (from 1-50).

2. Make sure the platen is prepared to accept the vial size you are loading. Refer to Sections 6.1 and 6.2 before proceeding.

3. Press the **A/S** key to select the start and stop positions corresponding to the vial positions filled in the carrousel.

4. Press **SCHED** to verify the proper start/stop method numbers in the scheduling screen.

Note: If all vials in the carrousel are to be analyzed together under one method, you can disable the scheduling feature. Refer to Section 10 of this manual for more information.

5. Once the vials are in place and you activate the desired method, the 7000 will automatically load and unload vials.

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6.9 Unloading Vials from the 7000 (without 7050 Installed)

You will need to manually unload the sample vials from the platen after Inject mode. To do this:

1. Use the **STEP** command on the 7000 keypad to move the platen to the vial position you want to unload.

2. Press the **LOAD/UNLOAD** key to raise the vial up through the aperture.

> **⚠ WARNING**
>
> 
>
> **Use extreme care when unloading hot vials. During a sampling, the vials are heated to the same temperature as platen chambers and may be hot enough to cause a burn.**

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6.10 Unloading Vials from the 7000 (with 7050 Installed)

During routine methods when the 7050 is installed, the 7000 will automatically unload sample vials from the platen after Inject mode, placing them back in position in the carrousel.

⚠ WARNING



Use extreme care when unloading hot vials from the carrousel. During a sampling, the vials are heated to the same temperature as platen chambers and may be hot enough to cause a burn.

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7.1 The Microprocessor

The 7000 control system consists of:

- an 8-bit microprocessor with 48K of program ROM (read only memory); 2K of RAM (random access memory),
- a membrane keypad to modify program parameter values,
- a 240 x 64 dot matrix LCD that displays the modes of operation.

The controller uses a 6303 eight-bit CMOS-type microprocessor to manage operation of the various functions of the 7000/7050 system. Instructions for the microprocessor are stored in ROM. After each initial power-up, you can review and change program parameters. Battery back-up saves changed parameters in the event of a power failure.

Default Methods/ Modified Methods

There are four default methods that you can customize for greater convenience and more efficient process time. Parameter values can also be copied from method to method (see Section 8). The new method values you enter will be stored in RAM. The programmed default values for all four methods are never lost and may be reinstated at any time (see Section 8.7 for detailed information on this topic).

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7.2 Program Panel

The program panel controls and/or displays the following:

- Method Parameters
- Time/Date Configurations
- Accessory Configurations
- Troubleshooting Checks
- Timing Functions
- Electronic Outputs
- Viewing Angle of Screen
- Error Signals
- Failure Signals

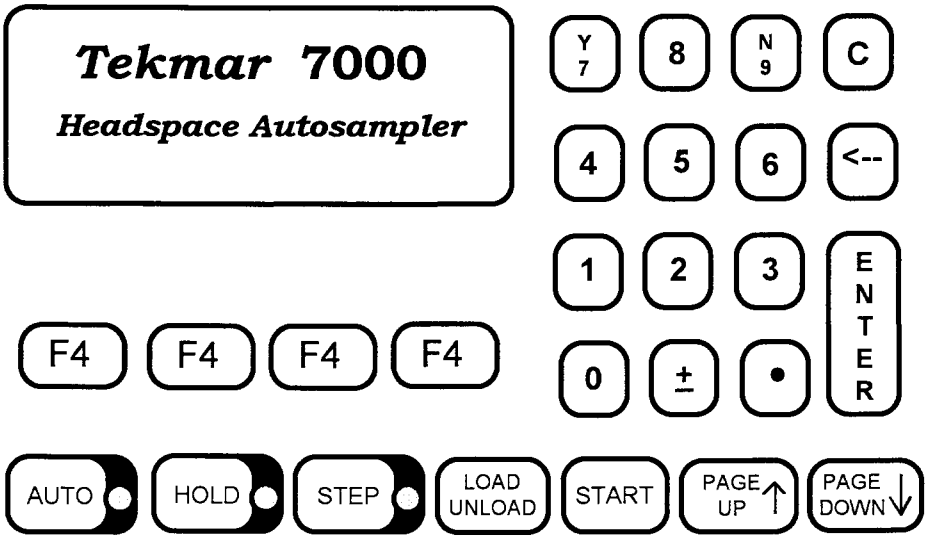


Figure 7-1 7000 Keypad

Keypad Description



AUTO signals the system to proceed through the run automatically.



HOLD is used to make the 7000 pause in a particular mode of operation. When you press **HOLD**, the 7000 stays in its present mode of operation until you press either the **STEP** or **AUTO** key.

continued

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STEP signals the 7000 to leave the mode of operation it was in and go to the next mode. This function is also used to load and unload the vials into the 7000 platen.



LOAD/UNLOAD enables the 7000 to position vials for manual loading and unloading of the platen.



START signals the 7000 to leave the Standby mode and begin a run.

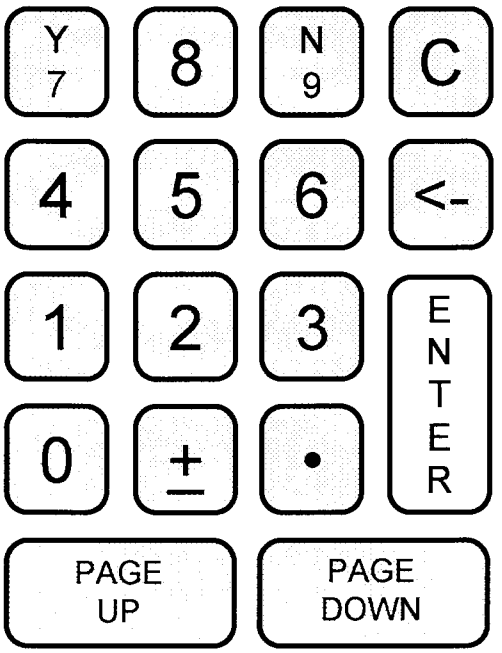


Figure 7-2 7000 Numbered Keypad

continued

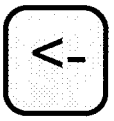
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The numbered keypad has two functions:

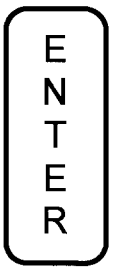
- The numerals are used to enter time, temperature, and mix power values for developing methods. Keys **Y/7** and **N/9** are used to enter "Yes" and "No" for parameter ON/OFF commands.
- It enables you to view the diagnostic screens: troubleshooting, pneumatic, and electronic output setting. See Section 5, Setting Pneumatics, for more information about accessing these screens.



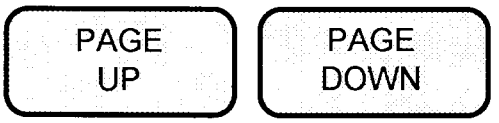
COPY enables you to copy parameters from one method to another. (Enter the method number you want to develop, then press **C**, then press the method number you want to copy.)



<- **(BACKSPACE)** removes a parameter value that appears in a highlighted box on the screen.



Press **ENTER** after you've indicated a value or highlighted a feature that you want to key into the method.



The **PAGE UP** and **PAGE DOWN** keys serve two separate functions:

- To change the viewing angle of the screen (see Section 14 for detailed information on this procedure).
- To view parameter listings (e.g. the Method Parameters screen) press **PAGE UP** or **PAGE DOWN** when the screen prompt "<PAGE UP/ DOWN for more >" appears.

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8.1 Overview of the Section

This section of the manual covers procedures for setting parameters on the 7000 Headspace Autosampler. Topics covered include Constant Heat Time Feature, Reviewing and Changing Instrument and Accessory Configurations, Reviewing, Setting, and Copying Method Parameter Values, Multiple Headspace Extraction, and Method Optimization Mode™.

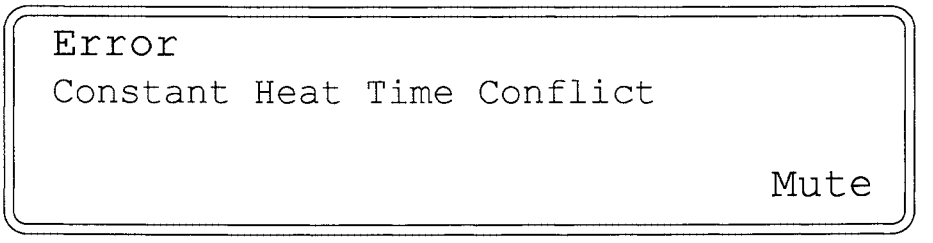
Upon routine power up, the 7000 conducts self tests to confirm that all of its heated components are working properly.

8.2 Constant Heat Time

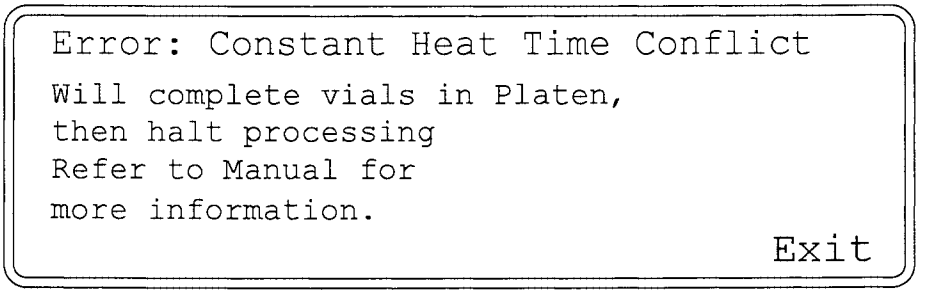
In its standard operation, the 7000 exposes each sample to identical thermal exposure times -- called **Constant Heat Time***. To ensure identical thermal treatment of each sample during automated runs, the 7000 checks for a ready signal from the GC before injection (during Loop Equilibrate mode if Constant Heat Time is mandatory; during Sample Equilibrate if Constant Heat Time is not mandatory**).

If the GC Ready signal is not present, the 7000 microprocessor assumes that either the GC is disabled or the cycle times between the 7000 and the GC are mismatched. In either case, the 7000 protects the vials that have not yet been analyzed by yielding a **Constant Heat Time Conflict** error message. The system then aborts the scheduled analysis.

The 7000 will beep to alert you of the problem, and the following screens will appear before the system reverts to the Standby mode.



1. Press **F4 (Mute)** to silence the alarm.



2. Press **F4 (Exit)** to clear. The 7000 will wait for the GC Ready signal.

*** Note: Constant Heat Time mode requires the 7050 carrousel.**

**** Note: See page 8-2 for information on Constant Heat Time mandatory/not mandatory.**

continued

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Constant Heat Time Mandatory / Not Mandatory

You may choose to have the 7000 ignore GC Cycle Time discrepancies by "telling" the 7000 that constant heat time is not mandatory. To do this:

1. Press **F4 (Conf)** from the Standby screen to review the Current Configuration screen.

```
Current Configuration                v. x.xx

Date:  1/05/99                Time:  1: 36: 47
A/S: YES       Cryo:  NO             Aux: NO
Constant Heat Time Mandatory?: YES

Lock      Clock      Inst.      OK
```

2. Press **F3 (Inst)** for the Instrument Configuration screen.

```
Automatic Sampler: Y
Cryofocusing: not installed
Aux Heater: not installed
Constant Heat Time Mandatory: Y

Help        <-          ->          Exit
```

3. Position the cursor by pressing **F3** (->) to highlight **Constant Heat Time Mandatory: Y.**

4. Press **N/9** on the keypad for **No**.

5. Press **F4 (Exit).** The following screen will appear:

```
Current Configuration                v. x.xx

Date:  1/05/99                Time:  1: 36: 47
A/S: YES       Cryo:  NO             Aux: NO
Constant Heat Time Mandatory?: NO

Lock      Clock      Inst.      OK
```

6. Press **F4 (OK)**. The system will go to the Standby screen.

The 7000 will now ignore any GC/7000 cycle time discrepancies. If you have the GC Cycle Time Conflict feature enabled on your 7000 system (***Constant Heat Time Mandatory: Y***), the system will alert you to take appropriate corrective action. See the following pages.

continued

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What to do in the event of a constant heat time conflict:
If the 7000 detects the Constant Heat Time Error and aborts samples, you will need to:

- Record the time it takes the GC to go from start of Injection/ Integration to the end of the run and then back to initial conditions. This value must be equal to or less than the programmed GC cycle time in your method.

Once your actual and programmed GC cycle times are synchronized, the GC should be ready for the 7000 injection and will not need to flag a cycle time error on your next run.

If your programmed GC cycle time and actual GC cycle time are the same, and you still encounter a GC cycle time error, it is probably because the 7000 for some reason could not receive the GC Ready signal.

To troubleshoot:
- Make sure that the GC I/O Board dip switches are set correctly. See Section 4.4 of this manual. Verify that the GC is ready prior to injection.

If the above actions do not solve the problem, please call the Tekmar Service Department at (800) 874-2004, or (513) 247-7000 locally and outside the U.S.

Note: You may disable the GC Cycle Time Conflict function whenever your GC cycle times will vary intentionally throughout a scheduled run. See the next page for additional information on this topic.

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8.3 Varying GC Cycle Times and GC Cycle Time Conflict Errors

When GC cycle time varies due to fluctuation in ambient temperature, you can disable the **Constant Heat Time Error** feature on your instrument by choosing **"Constant Heat Time Mandatory: N"** (see Section 8.2). This will place the 7000 in a waiting mode during Sample Equilibrate mode.

The system will pressurize your sample when it receives a GC Ready signal. Each sample may receive varying amounts of sample equilibration time. Therefore, when Constant Heat Time Error is **disabled** on your instrument, your system is no longer operating in the **Constant Heat Time** mode.

Note: This is only an acceptable situation when samples have reached true equilibrium (see Section 11).

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8.4 Constant Heat Time and Parallel Sample Introduction - Two 7000s and One GC

If you want to maximize the number of samples analyzed in a certain time period, you can hook up two 7000 systems to a single GC. This way, each 7000 is interfaced to an injection port column and detector. Two samples are introduced at the same time, separated on two columns and detectors in a single GC oven, and analyzed concurrently.

This configuration reduces large sample batch analysis time to about half the time required for single injection port analysis.

Note: For parallel operation, you will need to order a separate interface (I/O) cable:

- **Tekmar p/n 14-4830-086 for the HP 5890**
- **Tekmar p/n 14-5044-086 for the Varian 3400**
- **Tekmar p/n 14-4655-086 for the Tremetrics 540**

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8.5 Reviewing Method Parameter Values

To view default or other stored methods:

1. Press **F1 (Meth)** during the Standby mode.

2. Select a Method number from the keypad and then press **F3 (Edit)** to see the Method Parameters screen. If you wish to edit method parameters, you should review Sections 8 and 10 thoroughly.

Note: If you are viewing the screen during a run, the RUN option will not appear over the command key F2.

```
            Method 1 Parameters
Platen:  40°C PlatEquil:  10.00
SmplEquil:  60.0
Vial Size:  22 ml
(PAGE DOWN for more)

Help       Run        ->      Exit
```

Page down...

```
Mixer:  OFF              Mix:  -NO-
Mixer Power:  -NO-     Stabilize:  -NO-
Cryo Cooldown:  -NI-  min at -NI-

(PAGE UP/DOWN for more)
Help        Run     ->      Exit
```

Page down...

```
Press:  0.25     Press Equil:  0.25
Loop:  0.25      Loop Equil:  0.20
Inject:  1.00
Cryo Inject:  -NI-   min at -NI-
(PAGE UP/DOWN for more)
Help       Run        ->       Exit
```

continued

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Page down...

```
Sample Loop:  40°C
Line: 40°C
Cryo Union Htr:  -NI-
Aux Heater: -NI-
(PAGE UP/DOWN for more)

Help      Run      ->      Exit
```

Page down...

```
Inj per Vial:  1

GC Cycle Time:  30

(PAGE UP/DOWN for more)
Help      Run      ->      Exit
```

Page down...

```
Method Optimization Mode:   ON
- ->   Sampl Equil          First: 40.0
Max:   80.0                 Range: 40.0
Run 3 time(s) then inc by 5.0
(PAGE UP for more)
Help      Run      ->      Exit
```


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8.6 Setting New Method Parameter Values

To modify the default (or existing) times and temperature parameters:

1. Press **F1 (Meth)** from the Standby screen or from any method mode screen (Equilibrium, Mixing, Stabilize, etc.) and choose a method number.

2. Press **F3 (Edit)** to view the Method Parameters screen:

```
          Method 1 Parameters
Platen:  40.0°C    PlatEquil:  10.00
SmplEquil:  60.0
Vial Size:  22 ml
      (PAGE DOWN for more)

Help       Run        ->    Exit
```

3. Press **F3 (->)** to move the highlighted box to select the parameter you wish to modify and press **ENTER** on the keypad. Minimum and maximum limits are shown on the screen.

```
Current:        40°C
Minimum          0°C      Maximum  200°C
Platen Temp:      °C

      <Press ENTER for no change>
```

4. Use the keypad to enter a new value and press the **ENTER** key again.

The 7000 has similar screens for each temperature and time parameter.

continued

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Mixer Mode or Method Optimization On/Off

Two parameters - Mixing and Method Optimization - need to be turned on or off (See Section 9 for an explanation of Parameter Optimization).

To do this:

1. Press **F1 (Meth)** from the Standby screen or from any method mode screen (Equilibrium, Mixing, Stabilize, etc.) then choose a method number.

2. Press **F3 (Edit),** then press **PAGE DOWN** for the Mixer parameter in the second screen or Method Optimization in the last screen (the Mixer parameter will be used in the example below):

```
Mixer:  OFF                Mix:  -NO-
Mixer Power:  -NO-         Stabilize:  -NO-
Cap Cooldown:  -NI-        min. at -NI-

(PAGE UP/DOWN for more)
Help       Run      ->        Exit
```

To turn the mixer on or off:

1. Press **F3 (->)** to move the highlighted box to the "mixer" and press **ENTER** on the keypad. The following screen will appear:

```
Current:   OFF

Mixer: Choose ON/OFF

ON         OFF
```

2. Press **F1 (ON)** or **F2 (OFF)** as desired. The Method Parameter screen will be shown again.

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Multiple Headspace Extraction (MHE)

Multiple Headspace Extraction lets you run multiple samplings on one vial. You can do up to nine samplings (injections) from one vial. You cannot change the number of injections per vial while a method is running.

Injections (Samplings) Per Vial - Single or Multi-Puncture

There are two options with Multiple Headspace Extraction (MHE) mode: **Multi-Puncture** and **Single-Puncture** modes. Each option has its advantages and disadvantages -- please review these before deciding which best suits your particular sample characteristics.

Option 1 - MHE Single-Puncture Mode:
The vial's septum is punctured by the needle **one time only.**

Advantages of Single-Puncture Mode:

- Since the septum is punctured only once, the vial seal stays secure.
- The secure seal and lowered risk of leaking leads to better quantitation, especially with high vapor pressure samples.

Disadvantages of Single-Puncture Mode:

- Since the sample vial remains on the needle, all injections after the first injection will not include mixing (if mixing was activated in the method).
- In all subsequent injections after the first, the sample must wait for the GC to be ready (if the GC cycle time is longer than the re-equilibrate time).

Option 2 - MHE Multi-Puncture Mode:
The vial's septa is punctured by the sampling needle multiple times.

Advantages of Multi-Puncture Mode:

- There is identical re-heating time from the first injection to all subsequent injections.
- The mixing function is repeated for all injections (if mixing is activated within the method).

Disadvantage of Multi-Puncture Mode:

- The septa could leak between injections, leading to quantitation errors with high vapor pressure samples.

continued

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In Single-Puncture mode, the needle punctures the septum ***one time only***, but ***remains on the vial*** as it continues to extract the desired number of samples (up to 9 per vial). The 7000 then pressurizes the sample, fills the loop, equilibrates, and injects the sample into the GC. The vial is pressurized and re-pressurizes for the next injection. Each successive injection from a single vial occurs when the GC is ready.

To operate in Single-Puncture mode:

1. Press **F1 (Meth)**.

2. Choose a method number.

3. Press **F3 (Edit)** after choosing a method number.

4. Press **PAGE DOWN** to the fourth screen:

Note: If this is not an initial start-up, the 7000 will "remember" the previous parameter setting (either Single or Multi-Puncture) which may require you to switch from Multi-Puncture to Single-Puncture or vice versa. (See the following pages for further instructions.) If there is no parameter setting in the 7000's memory, the following screen will appear:

```
Inj per Vial:  1

GC Cycle time:  15
(PAGE UP/DOWN for more)

Help      Run      ->      Exit
```

5. Press **F3 (->)** to move the highlighted box to the **Injections per Vial** parameter. Press ENTER. The following screen will appear:

```
Current:     1
Minimum:     1        Maximum:  9
Injections per Vial:___

<Press ENTER for no change>
```

Note: You can run from 1 to 9 injections per vial.

continued

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6. Press a number (from 2 - 9) on the keypad to indicate the desired number of injections per vial. Press ENTER. The following screen will appear:

```
Inj per Vial:     3 w/ Multi Puncture
Sampling Option: Concentrate
Sample Re-Equil: 60
GC Cycle time:    30
(PAGE UP/DOWN for more)
Help        Run      ->    Exit
```

To operate in Single-Puncture mode, you will need to:

7. Press **F3 (->)** to move the cursor to highlight the "Multi-Puncture" prompt.

8. Press **ENTER**. The following screen will appear:

```
Current: Multi Puncture
Puncture: Choose SINGLE/MULTI

SINGLE     to re-Equil & Inject
MULTI      to re-Agitate also
SINGLE    MULTI
```

9. Press **F1 (SINGLE)**.

10. Press **F2 (Run)** to begin sampling.

continued

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Multiple Headspace Extraction in the **Standard** mode* includes an "intermediate vent" feature to further optimize headspace analysis. Intermediate vent operates as follows:

- For the first half of Inject mode, there is gas flow through the pressurization valve to the vial.
- At the halfway point, the flow is turned ***off*** at the pressurization valve and ***on*** at the vent valve to vent pressure from the vial.
- At the end of the Inject step, the vent is off, leaving the 7000 in a static mode (no flow through either the pressurization or vent valve until the pressurize mode for the next injection).

Note: In Single-Puncture mode, the vial will not be mixed in all injections following the first one.

***See pages 8-16 through 8-18 for more information on Standard (and Concentrate) modes.**

continued

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Multiple Headspace Extraction In Multi-Puncture mode (using Method 1 for illustration purposes)

In Multi-Puncture mode, the needle punctures the vial septa as many times as you program the 7000 to sample (inject). If you choose nine injections per vial with Multi-Puncture, for example, the needle punctures the septa nine times in a row. It draws a new sample each time as the vial comes up onto the needle and backs off again. Multi-Puncture adds a re-equilibration and a mix step (if activated) to the sampling run.

To operate in Multi-Puncture mode:

1. Follow steps 1-8 on pages 8-11 to 8-12.

2. The following screen will appear:

```
Current: Multi-Puncture
Puncture: Choose SINGLE/MULTI

SINGLE     to re-Equil
MULTI      to re-Agitate also
SINGLE    MULTI
```

3. Press **F2 (MULTI).** The following screen will appear:

```
Inj per Vial: 9  w/ MultiPuncture
Sampling Option: Concentrate
Sample Re-equil: 60.0
GC Cycle time:  30
(PAGE UP/DOWN for more)
Help      Run      ->      Exit
```

4. Exit Method Editing by pressing **F4 (Exit)** or **F2 (Run)** to begin sampling.

continued

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To go from Single-Puncture back to Multi-Puncture and vice versa:

1. Press **F3** to move the --> cursor to highlight "Single Puncture" or "Multi-Puncture".

```
Inj per Vial: 9  w/ MultiPuncture
Sampling Option: Concentrate
Sample Re-Equil: 60.0
GC Cycle time:  30
(PAGE UP/DOWN for more)
Help        Run    ->     Exit
```

2. Press **ENTER** and the following screen will appear:

```
Current: MultiPuncture
Puncture: Choose SINGLE/MULTI

SINGLE     to re-equil & Inject
MULTI      to re-Agitate also
SINGLE    MULTI
```

3. Press **F1 (SINGLE)** or **F2 (MULTI)** depending upon your desired parameters. The following screen will appear:

```
Inj per Vial: 9  w/ MultiPuncture
Sampling Option: Concentrate
Sample Re-equil: 60.0
GC Cycle time:  30
(PAGE UP/DOWN for more)
Help       Run     ->     Exit
```

4. Exit Method Editing by pressing **F4 (Exit)** or **F2 (Run)** to begin sampling.

Note: An "M" will flash in the lower left hand corner of the screen to remind you that you are in the Multiple Headspace Extraction mode.

continued

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Multiple Headspace Extraction (MHE) - Standard and Concentrate Modes

In Multiple Headspace Extraction, you can also choose "Standard" or "Concentrate" modes, which are described below. Each mode can be used with either single or multiple injections.

Standard Mode provides an extra equilibration step before a subsequent sample is injected. Standard mode runs the GC after each injection. It vents the headspace off of the sample before "Re-Equil" and allows you to select the time at which the sample being heated in the platen should reach a ***second*** equilibrium. This value is generally less than that of the original Platen Equilibration time.

Concentrate Mode is used to collect multiple injections from a single vial on a Cryofocusing Module (Tekmar p/n 14-2530-200) or in a subambient oven. In Concentrate mode, the GC runs only on the last injection. This feature increases sensitivity, area counts and signal-to-noise ratios of certain compounds by as much as 50%. It is recommended for analysis on compounds in very low concentrations. With Concentrate mode, you can also pick a setpoint for "Sample Re-Equil"*.

To choose single or multiple headspace injections:

1. From the Method Parameters screen, **PAGE DOWN** to "**Inj to Vial".**

2. The following screen will appear:

```
Inj per Vial:  1
GC Cycle time:  15
(PAGE UP/DOWN for more)

Help        Run        ->      Exit
```

***See page 8-19 for more information on Sample Re-Equil Mode.**

continued

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3. Press **F3** to move the --> cursor to highlight "**Inj per Vial**". Press **ENTER.** The following screen will appear:

```
Current:    1
Minimum:    1     Maximum:  9
Injections per Vial:

      (Press ENTER for no change)
```

4. Press a number (from 2 - 9) on the keypad to indicate the number of injections per vial you want to run. Press **ENTER.** The following screen will appear:

```
Inj per Vial:   3  w/ MultiPuncture
Sampling Option: Concentrate
Sample Re-equil: 60
GC Cycle time:  15
(PAGE UP/DOWN for more)
Help       Run      ->       Exit
```

5. Press **F3** to move the --> cursor to highlight the "**MultiPuncture**" prompt.

6. Press **ENTER**. The following screen will appear:

```
Current:   MultiPuncture
Puncture:  Choose SINGLE/MULTI

SINGLE    to re-equil and Inject
MULTI     to re-Agitate also
SINGLE      MULTI
```

continued

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7. Press **F1 (SINGLE)** or **F2 (MULTI)** and the parameter will automatically change. The method parameters screen will come up again:

```
Inj per Vial:     9 w/ MultiPuncture
Sampling Option: Concentrate
Sample Re-equil: 60
GC Cycle time:   15
          (PAGE UP/DOWN for more)
 Help       Run        ->        Exit
```

8. Press **F3** to move the --> cursor to highlight "**Concentrate**" on the previous screen. Press **ENTER.** The following screen will appear:

```
Current: Standard
Choose STANDARD/CONCENTRATE
STNDRD runs GC at each Inject
CONC accumulates injections --
running GC only at last Inject
STNDRD      CONC
```

9. Press **F1 (STNDRD)** or **F2 (CONC)** and the parameter will automatically change. The Method Parameter screen will come up again.

10. Press **F2 (Run)** to begin sampling.

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Multiple Headspace Extraction (MHE) - Sample Re-Equilibrate Mode

In Multiple Headspace Extraction, you can program the 7000 to run an additional equilibration step on the sample (which is still in the heated zone) after the sample is injected onto the GC or onto the subambient oven. This is called "Re-Equil". The second equilibration time is generally shorter than the first equilibration.

You can activate Re-Equil in Single or Multi-Puncture, Standard or Concentrate modes.

To program Re-Equil:

1. Follow steps 1-10 on pages 8-16 to 8-18.

2. Press **F3 (-->)** to highlight the "Sample Re-Equil" prompt.

```
Inj per Vial:   2  w/ MultiPuncture
Sampling Option: Concentrate
Sample Re-equil: 60
GC Cycle time:  15
(PAGE UP/DOWN for more)
Help      Run      ->      Exit
```

3. Press **ENTER** on the keypad. The following screen appears:

```
Current:  60.0 minutes
Minimum: 0.0   Maximum: 999.9
Sample Re-equil Time:   Min
```

4. Enter the value for Re-Equil and press **ENTER.**

5. Press **F2 (Run)** to begin sampling.

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Programming Vial Sizes

You can enter **Vial Size** as a parameter in the Method Parameters screen to tell the 7000 which size vial you plan to use.

1. Press **F1 (Meth)** and the desired method number.

2. Press **F3 (Edit)**. The following screen will appear:

```
Method 1 Parameters
Platen:        40°C    Plat Equil: 1.0
Smpl Equil:    15.0
Vial Size:     22ml

Help        Run     ->        Exit
```

3. In the Method Parameters screen, press **F3 (-->)** to move the cursor to highlight "Vial Size" and press **ENTER.**

4. The next screen shows you the vial size currently programmed.

```
Current:      22ml
Vial Size:    Choose 9ml/12ml/22ml
9ml & 12ml need platen & carrousel
      transfer inserts
For 22ml vials, remove inserts
9ml          12ml              22ml
```

5. Press the function key **(F1, F2, or F3)** that corresponds to the vial size you plan to use. The 7000 will acknowledge the new vial size.

Note: If changing vial sizes from previous runs, see Section 6 to set up.

6. Press **F2 (Run)** to begin sampling.

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8.7 Copying Method Parameters

This option allows you to copy parameters from one method to another. ***It does not copy individual parameters.*** It transfers all of the parameters from one method to another, creating two identical methods. You may want to keep a hard copy of the values you are replacing.

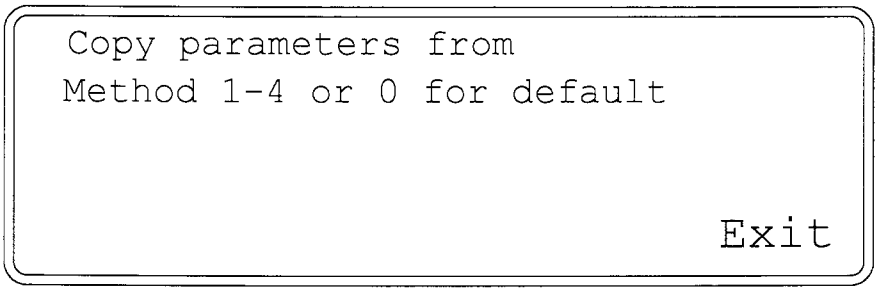
To Copy Methods:

1. Press **F1 (Meth)** from the Standby screen or from any method mode screen.

2. Select the method you want to copy to and press the number on the keypad.

3. Press **F3 (Edit)**.

4. Press the **C** on the keypad and the Copy Parameters screen will appear:



5. Choose the method you want to copy from and press the number on the keypad.

A Method Parameters screen will appear with the new method and copied parameters. Press **F4 (Exit)** if you do not want to copy the parameters.

To re-enter default values:

To revert back to all default values in a given method, enter 0 in the Copy Parameters screen.

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8.8 Method Optimization Mode™ (MOM)

Method Optimization Mode™ (MOM) is a unique control program on the 7000 that allows you to optimize time, temperature, and mix settings for the highest sensitivity and lowest RSD values.

In MOM, successive samples undergo incremental changes according to a pre-assigned setpoint. After reviewing the results of the programmed runs, you can determine the optimal setpoints and assign them to that parameter. MOM's automation lets you, for example, test and review all possible setpoints overnight. Parameters that you can select for incremental change include:

Time	*Temperature/Mix Power*
Platen Equilibrate Time	Platen Temperature*
Sample Equilibrate Time	Mix Power
Mixing Time	Valve Temperature
Stabilize Time	Transfer Line Temperature
Pressurize Time	Capillary Interface Cooldown Temperature
Pressurize Equilibrate Time	Capillary Interface Injection Temperature
Loop Fill Time	Capillary Interface Union Heater Temperature
Loop Equilibrate Time	
Inject Time	
Capillary Interface Injection Time	
Re-Equil Time	

⚠ CAUTION

*** Platen temperature should never exceed 10° below the boiling point of the solvent within the vial. If you exceed this value, the cap and septum assembly may leak and/or pop off of the top of the vial. The maximum optimal platen temperature is usually 15 - 20° below the boiling point of the sample solvent. Do not over-increment the platen temperature beyond 10° below the boiling point of the solvent in the vial.**

Note: Full Evaporation Technique is an exception to the above due to the limited volume of solvent (< 20 ul).

continued

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***To program MOM*:**

1. Select a method and press **F3 (Edit)** for the Method Parameters screen. Press **PAGE DOWN** to the last screen:

```
Method Optimization Mode:  OFF
-->      -No-      First: -No-
Max:     -No-      Range: -No-
Run -NO- time(s) then inc by -NO-
PAGE UP for more  ->
Help          Run        ->        Exit
```

2. With **OFF** highlighted, press **ENTER** on the keypad for the Optimization ON/OFF screen. Press **F1 (ON)**. The unit will return to the optimization parameter in the Method Parameters screen.

Note: When you are in Method Optimization Mode, an "O" will flash in the lower right hand corner of the Standby screen.

```
Method Optimization Mode:  ON
-->   Platen                First: 40°C
Max:    0°                  Range: 0°
Run 1 time(s) then inc by 0°
PAGE UP for more  ->          Tracking: OFF
Help          Run          ->      Exit
```

3. Press **F3 (->)** to highlight the parameter heading (such as "Platen") and press **ENTER**.

```
Method Optimization Mode:
-->  Platen

(PAGE DOWN & PAGE UP to change)
         (ENTER to lock in choice)
```

4. Press **PAGE UP** or **PAGE DOWN** to find the parameter you wish to optimize. Press **ENTER** to select the parameter.

continued

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5. Once you select the parameter, the value shown on the screen is the time or temperature at which the parameter will begin optimizing. This is the "first" -- or minimum -- value that you entered when you first set up your method parameters (see Section 8.6). You can change the minimum value by going back into the Method Parameters screen.

6. Enter the maximum value (at what time or temperature optimizing will stop) at "MAX" in the Method Optimization Mode screen. Press **F3 (->)** to move the cursor to highlight the "MAX" prompt and press **ENTER**. Use the keypad to enter the value and press **ENTER.**

```
Current: 40°C
Minimum: 40°C  Maximum 200°C
Maximum for Opt:

      <Press Enter for no change>
```

7. The 7000 calculates the "RANGE" by subtracting the minimum or first value from the maximum as shown in the following screen.

```
Method Optimization Mode: ON
-->        Platen:          First: 40°
Max:      140°              Range: 100°
Run 1 time(s) then inc by °   Tracking: OFF
Help        Run        ->      Exit
```

You need to tell the 7000 how many times to run that increment and at what increments you want it to optimize the time or temperature parameter.

To conduct multiple runs at each time or temperature increment so that you can validate your data:

1. Press **F3 (->)** to highlight **Run __ time(s)**. Press **ENTER**. The following screen will appear:

```
Current: 1
Minimum: 0  Maximum 50
Times to run each increment:

      <Press Enter for no change>
```

continued

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2. Enter the desired number and press **ENTER**. The Method Optimization Mode screen will come up again and show you the values you selected.

```
Method Optimization Mode: ON
-->     Platen:              First: 40°C
Max:    140°C                Range: 100°C
Run 4 time(s) then inc by    °  Tracking: OFF
Help          Run            ->      Exit
```

3. Press **F3 (->)** in the Method Optimization Screen to highlight "Inc by:". Press **ENTER**. The following screen will appear:

```
Current: 0°C
Minimum: 0°C  Maximum 140°C
Increment By:

      <Press Enter for no change>
```

4. Enter the increment at which you want the parameter to be optimized. Press **ENTER**. The Method Optimization Mode screen will come up again to show you the values you selected.

Note: If you enter an increment value that does not divide evenly into the range or exceeds the maximum, the 7000 sends an error message (see the screen below). For example, if the range is 100°, and you enter a 15° increment, you will get the error because 15 is not a multiple of 100.

```
ERROR: Increments do not fit
       range.
Select Increment or Max to
provide a whole number.
                              Exit
```

5. Press **F4 (Exit)**. The system will return to the Method Optimization Mode screen. Press ENTER to input another value.

continued

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Note: If you enter a number larger than the range or out of the temperature/ time limit, the following screen will appear:

```
Current: 0°C
Minimum: 0°C  Maximum 140°C
Increment By: 150°C
<Value Out of Range>
    <Press Enter for no change>
```

5. The "Value out of Range" message will flash, and then the increment (Inc By) prompt will be highlighted so that you can re-enter your value.

Note: All parameters can be optimized like the platen example used above, but only one parameter can be optimized at a time.

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Tracking

In Method Optimization Mode, a "tracking" feature allows you to program the platen, sample loop, and line temperatures to increase by the same increment during each run. These are the only parameters that can be "tracked." For example, if you want to increment all three parameters from 40°C to 140°C in 10° increments, you would set up the parameters in the Method Optimization screen as follows:

```
Method Optimization Mode: ON
Platen:                          First: 40°
Max:      140°                   Range: 100°
Run 4 time(s) then inc by 10°    Tracking: OFF
<PAGE UP For More>
Help          Run          ->          Exit
```

1. Press **F3** to move the cursor (->) to highlight "Tracking".

2. Press **ENTER.** The following screen will appear:

```
Current:     OFF
Tracking:    Choose ON/OFF


ON          OFF
```

3. Press **F1 (ON)** to enable tracking of the line, valve, and sample loop.

With tracking enabled for the **Platen** in Method Optimization Mode and the "increment by" parameter set for 10°C, the method would run with platen, sample loop, and line temperatures at 40°C, then 50°C, then 60°C, etc.

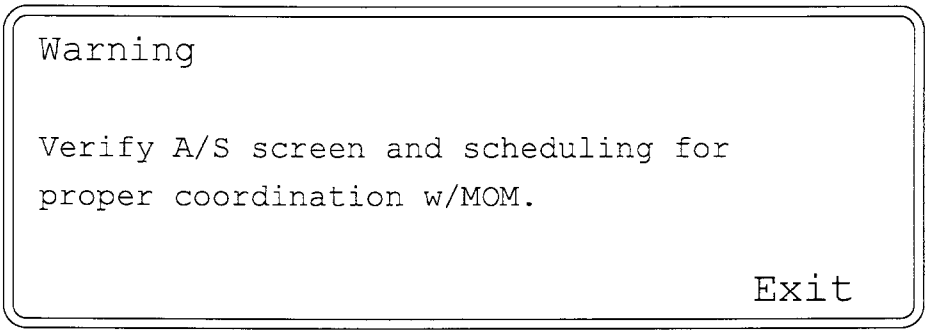
You may also enable tracking at the sample loop temperature screen. If you do this, only the sample loop and line temperatures will increment the same amount in each run. The platen temperature parameter will not increment, rather, it will run at the temperature set up in the method.

You can disable tracking by keying in **F2 (OFF)** at the screeen prompt. With tracking disabled, you can increment platen, sample loop, and line temperatures separately without affecting one another.

continued

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When you exit the MOM screen, verify that the values in the Autosampler Control (A/S) screen coordinate with MOM. The following screen will come up as a reminder after you exit MOM:

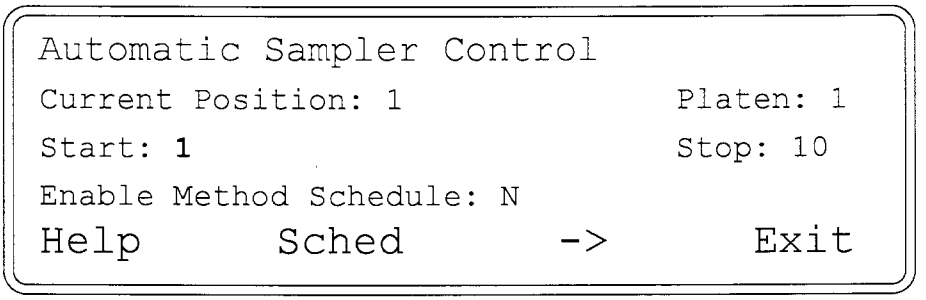


Use the A/S screen to program the number of vials that you want to run in Method Optimization Mode. For example, if you intend to run MOM while optimizing the Platen Equilibrate mode from 40° to 140° four times, in increments of 10°, you will need 40 vials in the carrousel. Therefore, set the A/S screen to start at vial position 1 and end at vial position 40.

To set up the A/S screen:

1. Bring up the A/S screen by pressing **F2 (A/S)** in the Standby screen.

2. Press **F3** (-->) to move the cursor to highlight "Start" and press **ENTER**.



3. Enter the carrousel vial position number you want to begin with and press **ENTER.**

4. Do the same for the "Stop" position.

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9.1 Overview of the Section

This section of the manual explains some of the theory as well as step-by-step procedures in developing methods for the Tekmar™ 7000 Headspace Autosampler. Specific topics included are:

- Fundamentals of Headspace
- Injection Techniques
  - Pressure
  - Temperature
  - Full Evaporation Technique
- Quantitation of Headspace
- Optimization of Headspace Sensitivity
- Example Application Parameters

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9.2 Fundamentals of Headspace

This section provides an overview of the fundamentals of headspace analysis. For further investigation into gas chromatography/headspace analysis, there are several recommended books and other documents available, including:

- Headspace Analysis and Related Methods in Gas Chromatography, Ioffe and Vitenberg; John Wiley and Sons; 1984
- "Design and Performance of an Automatic Static Headspace Analyzer", R. Westendorf and H. Lehan, Tekmar Company, 1990

An overview of headspace analysis:

In the headspace analysis process, sealed vials containing samples are heated, allowing the volatile organic compounds to diffuse into the vial's atmosphere or vapor space, known as the "headspace". A portion of the vapor is then removed from the vial for injection into a gas chromatograph.

In a sealed vial, the analytes diffuse from the sample into the vapor space and also from the vapor back into the sample. Eventually equilibrium occurs as the two diffusion rates equal and the concentration of analytes in the vapor remains constant.

Injections to the gas chromatograph are usually made after the sample has reached equilibrium. This yields the highest sensitivity and precision possible.

The equilibrium concentration depends heavily on sample temperature. Small changes in temperature can cause major changes in vapor concentration, so temperature is the variable most likely to affect headspace procedures.

The speed with which equilibrium is reached depends primarily on the diffusion rate of volatiles in the sample matrix. Some solids (such as plastic) need substantial time to reach equilibrium. In this instance, it is not practical to wait until equilibrium occurs before sampling. Therefore, a technique such as Constant Heat Time is used. In Constant Heat Time, each sample is heated at identical times and temperatures before injection to the gas chromatograph. This yields good reproducibility in samples that are otherwise difficult to analyze.

continued

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Early applications of headspace analysis began in the 1960's. By the 1970's, headspace analysis was being used with residual solvents, monomers, plastics, and food samples. Later, headspace became a solution for water, blood alcohol, and flavor analysis. Within the last few years, static headspace applications have expanded to include Full Evaporation Technique, environmental soil methods, and pharmaceutical methods.

In all applications, headspace eliminates cumbersome, expensive sample preparation and extraction procedures and results in less instrument down time and less contamination than other techniques.

Definitions

There is a glossary at the back of the manual, but following are five terms that are used frequently in the following pages. If you are not already familiar with these, please take the time to review them before reading on.

Partition Coefficient (K) - The ratio of concentration of an analyte in the sample matrix to concentration in the gas phase at equilibrium, $K = C_M/C_G$. The partition coefficient is dependent on a number of factors, including temperature and solvent.

Distribution Constant - A constant that describes the equilibrium considerations for a particular analyte/matrix combination, at a given temperature. The distribution constant is independent of any other variables. It can be used to predict partition coefficients, but is not generally used in daily method calculations.

Phase Ratio - The ratio of the volume of vapor space in a vial to the volume of sample, V_G/V_L.

Equilibrium - The state achieved when the concentrate of analyte is constant in the gas phase within the sample vial.

Static Vial Pressure - The pressure in a vial during equilibrium. The pressure in most vials will rise above ambient as the sample is heated and the partial pressures of the analytes and solvent increase.

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9.3 Injection Techniques

Once a sample has reached equilibrium, its headspace is **injected** into the gas chromatograph as a gaseous sample. Gas injections are significantly different from liquid injections. With liquids, the total mass injected is a function of injection volume and analyte concentration. No other factors are involved.

Since gases are compressible, the actual quantity injected can vary depending upon the amount of gas that is compressed into the loop, even though there is a fixed sample loop size. Factors such as pressure and temperature affect the quantity of the gaseous sample injected.

Pressure, temperature, and "Full Evaporation Technique" are the injection considerations covered in this section.

Pressure

Backpressure at the vent port determines the pressure in the sample loop at injection. If you are not using a backpressure device, the sample loop pressure will fall to atmospheric pressure. For the majority of headspace samples, this is acceptable. But changes in atmospheric pressure will cause variations in sample loop pressure and, ultimately, the amount of sample injected. This will affect quantitative reproducibility.

One simple way to improve reproducibility of the sample loop pressure is to apply a fixed backpressure device at the vent port, such as Tekmar's Variable Injection Pressure Regulator (VIPR™). The VIPR maintains constant pressure in the sample loop regardless of atmospheric pressure, yielding better day-to-day reproducibility. The VIPR also lets you increase pressure in the sample loop. Higher pressure leads to more sample in the loop and therefore, greater sensitivity. (Instructions on setting the VIPR can be found later in this section.)

There is another way to improve sample loop pressure reproducibility, but it is a manual method that requires daily (or even more frequent) attention, depending on your laboratory conditions. Here are the instructions:

1. Attach a length of 1/16" O.D. tubing to the vent port.
2. Place the other end of the tubing in a graduated cylinder so that the end of the tubing is stable at the bottom of the cylinder.
3. Fill the cylinder at least half full with water.
4. Record this level.
5. The water column will now exert a head pressure on the vent that is independent of atmospheric pressure.

Note: The water level must remain constant or the backpressure will vary.

This method does not increase pressure in the loop as effectively as the VIPR does, but it will smooth out differences caused by fluctuating weather conditions.

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Temperature

The temperature of the sample loop also affects the amount of sample in the loop. Higher temperatures cause the sample gas to expand, resulting in less sample in the loop. Temperatures that are too low may cause carryover of some analytes, or condensation of the solvent vapors in the sample valve and loop. The actual temperature you choose must be at least as high as sample temperature. This will usually eliminate condensation and will prevent carryover. Some analytes, however, may be prone to adsorption on sample path surfaces. For samples of this type, you need to raise the temperature enough to eliminate carryover.

Full Evaporation Technique

The Full Evaporation Technique (FET) completely vaporizes a small aliquot of liquid sample in a sealed headspace vial. It is often the first step in the headspace method development process. FET is used both as a quantitation tool for standard headspace samples and a complete analysis method for certain liquid samples. It is a powerful tool in the analysis of some samples because it can keep non-volatile or inorganic material (such as salts or polymers) in the sample from entering the analytical system.

FET helps ensure your injection port, column, and detector remain as contaminant-free as possible to yield the highest degree of precision, accuracy, and sensitivity.

In the FET process, several microliters of a sample extract are injected into a sealed headspace vial. The vial is then heated to a temperature high

continued

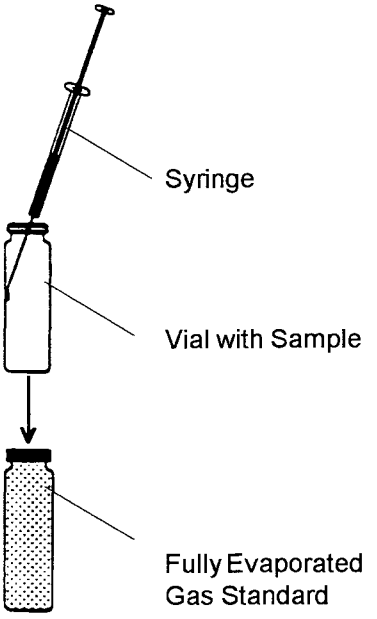


Figure 9-1 Full Evaporation Technique

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enough to fully vaporize all of the sample's volatile and semi-volatile components. (These components are normally vaporized within the gas chromatograph's injection port in direct injections.) The vapor containing only the volatile and semi-volatile compounds is then injected into the GC for final analysis. Any non-volatile material remains in the vial and does not enter the GC/MS, preventing interference with the performance of the analytical system.

FET is an ideal method for the analysis of high molecular weight compounds, such as pesticides, polychlorinated biphenyls, nitroaromatics, and polynuclear aromatics. Generally, analysis of these compounds is complicated by the complex matrices in which they are found.

FET can also eliminate the need for time consuming clean-up procedures before direct injection of wastewater and soil extractions. Because the extract is injected directly into the headspace vial, the vial serves as a disposable injection port liner/pre-column.

To perform the Full Evaporation Technique, you will have to first determine the volume and concentration requirements for the particular sample you want to run.

The FET gas phase concentration is expressed as follows:

$$[\text{FET}] = \frac{\text{Total analyte mass injected into the vial}}{\text{Total gas volume of the vial + net gas phase expansion volume}}$$

The analyte mass injected into the vial is determined from the known concentration of standard solutions or the density for pure compounds. The gas volume of the vial is dependent on the vial size chosen.

Note: Tekmar vial volumes are 22.3 ml (± 0.09% RSD), 12.6 ml (± 0.08% RSD), and 9.4 ml (± 0.06% RSD).

The gas phase expansion volume can be approximated by adding 0.2 ml to the sample loop volume. Gas phase expansion volume can be more accurately determined by using the Tekmar Parameter Optimization Kit (p/n 14-5046-000), discussed on the next page.

continued

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To determine gas phase expansion volume with the Parameter Optimization Kit:

1. Attach the bubble meter to the bulkhead fitting marked "VENT" at the back of the 7000.

2. Set up a run with a 10-minute Sample Equilibration Time.

3. Select an evaporation temperature based upon:

   - the solvent's boiling point,
   - the vapor pressure of the analytes at a given temperature, and
   - the thermal lability of the analytes at the evaporation point to ensure that heat-induced degradation does not occur.

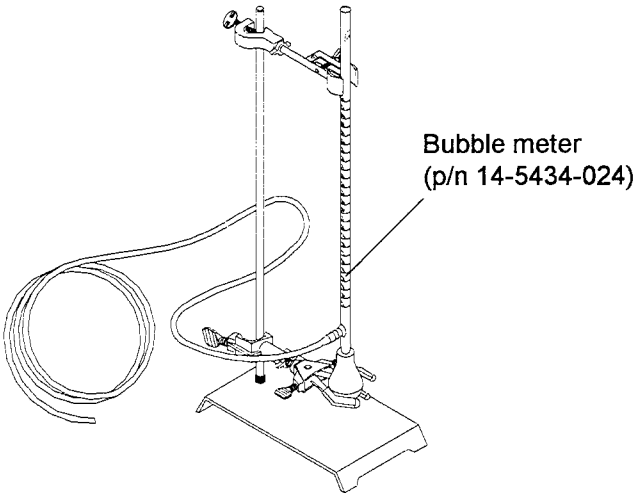


Figure 9-2 Parameter Optimization Kit

To run the Full Evaporation Technique:

1. Inject a volume of volatile or semi-volatile mixture into the vial. (Volumes are generally 1-20 µl).

2. Seal and heat the vial at a temperature that exceeds the boiling point of the solvent by at least 10°C.

3. Run the 7000 method.

4. Construct a calibration curve based on the standard results.

5. Repeat for samples, and quantitate based on the calibration curve.

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9.4 Quantitation of Headspace

Headspace analysis is a powerful quantitative tool for many samples. There are a number of procedures that can be used to quantitate samples. The most common method is to prepare a standard in the authentic matrix (e.g., a standard containing known concentrations of alcohols in water). The standard is then analyzed in the same manner as samples, and the results are used to calibrate the system. Calculations are handled using the "External Standard" method. This method is not appropriate, however, when the sample cannot be duplicated exactly.

For many liquid samples where the matrix cannot be accurately copied, standard additions are used. In this case a sample is run first, followed by an identical sample which has had a known quantity of analyte added to it. The peak area of the analyte will be greater in the second run because of the added spike. A calibration factor is calculated by dividing the increase in area by the amount of spike. This factor is used to calculate the concentration in the original sample.

The most precise quantitation technique requires a multi-step approach. The concentration of analyte in the gas phase in a headspace vial at equilibrium is described by this equation:

$$C_o = C_g\ (K + V_g/V_m)$$

where:

$\mathbf{C_o}$ = concentration of the original sample in the matrix
$\mathbf{C_g}$ = concentration of the gas
K = partition coefficient
$\mathbf{V_g}$ = volume of gas in the vial
$\mathbf{V_m}$ = volume of matrix in the vial ($\mathbf{V_m}/\mathbf{V_m}$ is known as the Phase Ratio)

All of the variables in this equation can be determined through experimental runs, to provide a complete description of the equilibrium. To achieve this, you need to establish a calibration plot of concentrations of analytes in the gas phase. This will give you the value of C_g in the above equation. Use the Full Evaporation Technique to come up with the calibration plot.

Using the method of standard addition, determine the **partition coefficient (K)** of each compound in the sample matrix:

$$\text{Partition Coefficient} = \frac{\text{Concentration in matrix } (C_m)}{\text{Concentration in gas } (C_g)}$$

continued

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Determine the **phase ratio** for your samples. The phase ratio is the ratio of the volume of headspace gas to the volume of sample in the vial (V_g/V_m). The higher the phase ratio, the lower the volume of sample and the greater the quantity of headspace.

$$\textbf{Phase ratio} = \frac{\text{Volume of Gas}}{\text{Volume of Matrix}} = \frac{V_g}{V_m} = \frac{\text{volume of vial - volume of matrix}}{\text{volume of matrix}}$$

example: $$\frac{\textbf{22.3 ml - 5.0 ml}}{\textbf{5.0 ml}} = \textbf{3.5}$$

Multiple Headspace Extraction

The Multiple Headspace Extraction technique uses multiple analyses of one sample to determine the partition coefficient. It is based on the principle that once the vial's headspace is vented (sampled), the sample will reach a new equilibrium based on the quantity of analyte left in the vial. Running the sample again will produce a peak somewhat lower than the first run. If the process is continued, the peak area will fall at an exponential rate. This rate of decay, along with the quantities determined, can be used to determine the original concentration in the sample. This method is especially useful in samples where the standard addition approach cannot be used to spike the sample. The MHE method is described in detail in **Headspace Analysis and Related Methods**, Ioffe and Vitenberg, John Wiley and Sons, 1984.

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9.5 Optimization of Headspace Sensitivity

In headspace analysis, the concentration of analyte in the gas phase (C_g) must be maximized to achieve the greatest sensitivity possible.

There are two variables in the equation $C_o = C_g (K + V_g/V_m)$ that determine C_g: the partition coefficient (K) and the phase ratio (V_g/V_m). Of these, K is the most significant factor in determining sensitivity. If K is high, the value for C_g will be low, and the response will be poor. The value of K depends on the nature of the sample (including both the analyte and the matrix) and the temperature.

Temperature is the easiest value to manipulate. Higher temperatures will shift the equilibrium to favor the headspace. However, temperature is limited by the boiling point of the sample solvent, and, possibly, the thermal lability (stability in temperature) of the components.

Generally speaking, you should work with temperatures no higher than 15° C ***below*** the boiling point of the solvent. Higher values may result in over-pressurization of the vial (a safety hazard), excessive solvent peak in the chromatogram, and carryover. If labile components are present, these will further limit the temperature value. Even if the ***target*** analytes are not labile, if ***any*** labile components exist, the temperature should be low enough to prevent component rearrangement. If compounds are being formed or degraded, equilibrium of the target analytes (and ultimately sample quantitation) will be affected.

The partition coefficient can also be changed by modifying the matrix. As a general rule, an increase in the ionic strength of a sample will shift the equilibrium towards the headspace. This is accomplished by adding salt to the sample (a procedure commonly known as "salting out"). To keep the ionic strength constant between samples, each sample is saturated with salt. This technique is very common for aqueous samples, and is particularly useful in improving the analysis of water-soluble compounds.

The phase ratio is determined by how much sample is placed in the vial. As a general rule, decreasing the phase ratio (i.e. increasing the volume of sample in the vial) will increase C_g. More sample means less gas volume, and higher gas concentrations. However, the gas volume must not be made too small. Very small phase ratios usually result in poor reproducibility. Also, there may not be enough vapor to adequately fill the sample loop. A general rule is that the gas volume should be at least twice the volume of the sample loop.

The importance of the phase ratio's contribution to sensitivity is limited by the partition coefficient (K). If K is very high, the contribution of the phase ratio is negligible. But if K is too high, sensitivity can be so poor that there is little chance of a successful analysis. If K is low, the phase

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ratio is the limiting factor. Typical K values range anywhere from less than 1 to greater than 1000. Generally, K values greater than 100 are not suited to headspace analysis. Phase ratio values range from 0.05 to 50. Samples with phase ratios in the range of 1 to 10 yield the best reproducibility.

Determining Operating Parameters

Each operating parameter can be optimized to produce the best results for your sample. Some parameters are more critical than others, and require greater attention. This section will discuss each parameter, and provide suggested values as starting points.

Sample Loop Size

Sample loop size is a major determinant in sensitivity. Larger loops will provide greater sample amounts for injection into the GC. But smaller loops generally provide more reproducible results. This is partly due to more efficient sweeping of the loop, and the better chromatographic resolution obtained when injecting smaller volumes of vapor. A large loop volume may lead to peak broadening when used with slow carrier rates.

A rule of thumb is that the loop should be fully swept in no more than 10 seconds. So if the carrier flow rate is slower than 6 ml/min., a loop smaller than 1.0 ml should be used. If you are using some form of refocusing, such as the Cryofocusing Module or on-column cold-trapping, larger loops don't present a problem.

Sample Temperature

Sample Temperature (referred to as Platen Temperature in the 7000 Method display) is extremely dependent on the type of sample. Most liquid samples are heated to temperatures up to 15° C below the boiling point of the solvent. For example, most aqueous samples are heated to 85° C. However, if this temperature causes any reactions (as in many biological samples), you will need to reduce the temperature. The best guide for choosing a sample temperature is to consult available data on samples of a similar nature.

Higher temperatures may yield higher concentrations of analytes in the headspace than is desired. Also, significant quantities of unwanted higher molecular weight compounds may appear. These can adversely affect GC time by demanding longer times for the compounds to elute (unless you are using column backflush techniques). Overheating may also lead to analyte degradation or over-pressurization of the vial.

As the temperature approaches the boiling point of the solvent, the concentration of the solvent vapor within the vial increases exponentially. This increases the potential for solvent interference (e.g. column flooding) and/or detector abnormalities. Therefore, it is recommended that platen

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temperature settings remain at least 15° C below the solvent boiling point.

> **⚠ CAUTION**
>
> **Platen temperature should never exceed 10° below the boiling point of the solvent within the vial, otherwise the cap and septum assembly may leak and/or pop off the vial due to overpressurization.**

Valve and Line Temperatures

Generally speaking, the valve and line temperatures are set to the same value -- a value identical to the platen temperature. The lowest value that prevents carryover is ideal.

Sample Equilibration Time

This time depends upon the length of time a sample takes to reach equilibrium. It is dependent on the nature of the sample matrix, the analytes, and the temperature.

Note: When a sample is in an equilibrium state, analyte concentration within the headspace is at its maximum, and does not fluctuate significantly with heating time. For this reason, Constant Heat Time Mode is not essential for acceptable precision with an equilibrated sample. But precision will be improved to some degree in all samples with Constant Heat Time.

If a sample does not equilibrate within a reasonable amount of time, the concentration of analytes in the headspace atmosphere will increase as equilibration time increases. Therefore, Constant Heat Time mode is needed to ensure precision in samples that do not reach equilibrium.

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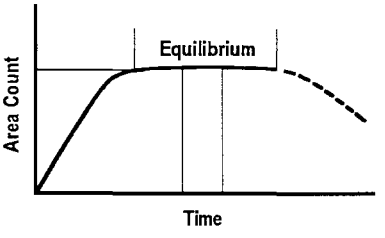


Figure 9-3 Equilibration Curve

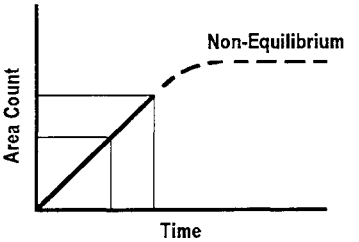


Figure 9-4 Non-Equilibrating Sample

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Liquid Samples

Low viscosity liquids reach equilibrium sooner than high viscosity fluids. Aqueous solutions take 20 to 60 minutes to reach equilibrium, but more viscous samples, such as oils, take longer -- sometimes up to several hours. Determine your optimal sample temperature experimentally:

1. Start with a value of 15 to 20 minutes. Run a series of identical samples, gradually increasing equilibration time using Method Optimization Mode. For aqueous samples, use five-minute increments. For other samples, use 10-minute increments.

2. Tabulate the area values for the peaks of interest.

3. Plot area versus time.

4. The point at which the curve flattens is the equilibrium time. Choose a time equal to or slightly greater than this time.

Solid Samples

Solids generally take significantly longer than liquids to reach equilibrium because the diffusion rate of solutes within the solid matrix is quite low. There are several methods for improving the equilibration rate. These are listed below in order of preference, based on typical performance. If a certain technique doesn't apply to your sample, move down the list until you see an appropriate method.

- Completely dissolve the solid in a solvent less volatile than the target analytes. Choose a solvent based on volatility, compatibility with the sample (completely soluble, no reactions), and detection method (a solvent not at all detected is best). If a sample is water-soluble, water is usually the best solvent.
- For non-dissolving solids (e.g. soils), use a liquid extractant to extract the solutes from the matrix. Ultrasonic disruption (after sealing in the headspace vial) can dramatically increase extraction efficiency. One drawback of this approach is that it produces a three-phase system, and equilibrium may become complicated.
- Grind solids to a small particle size. This increases the surface area and decreases the mean path length that solutes must follow to reach the gas-solid interface. However, take care when grinding to avoid losing or altering volatile components. Tekmar recommends grinding at cryogenic temperatures to prevent losses. Some samples, especially food, undergo enzymatic reactions that change the volatile profile when subjected to shear stress (cutting). These reactions may be desirable in certain applications.
- Increase the sample temperature to increase the diffusion rate. Take care not to reach temperatures that degrade the sample. (Note warnings on sampling temperatures in Section 2 of this User Manual.)

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Gas Samples
Gas samples (such as those created with the Full Evaporation Technique) require shorter heating times than liquids or solids. Times of five to 10 minutes at temperatures 10° to 15° above the solvent boiling point are generally adequate.

Constant Heat Time
Some samples will not reach equilibrium in any reasonable amount of time. For these samples, it is recommended that you use Constant Heat Time to improve reproducibility. In Constant Heat Time, each sample is heated for a precisely reproducible time before sampling. This gives each sample the same amount of time for solutes to diffuse into the headspace. However, you will see differences if samples differ in such variables as sample size, particle size, or particle shape. You can minimize these differences by grinding all samples to a uniform, small particle size. This has the added benefit of decreasing the mean path length that solutes must follow, and increases sensitivity. However, when grinding samples, be careful so as not to lose or alter any solutes.

Equilibrium time chosen in the Constant Heat Time mode will be a compromise between sensitivity, reproducibility, and practicality. As a rule, the more samples you have, the longer you can make the equilibration time (as one sample is running, the next one can be equilibrating).

Sample Mixing Parameters
Mixing liquid samples can significantly increase the speed with which equilibrium is reached. This is due to a great reduction in the mean path that solutes must follow to reach the gas-liquid interface. For samples of low viscosity (such as water), equilibrium is reached in a short period of time. More viscous samples such as oils take longer to reach equilibrium.

Mixing Time: General Guidelines
Optimal mixing time is highly dependent on the nature of the sample. Mixing time is generally 20% or less of the total Sample Equilibration time. Longer mixing times will usually lead to longer analysis times. This is because samples cannot be added to or removed from the platen, and vials cannot be sampled when other vials are mixing. With more than 20% mix time, Constant Heat Time mode is interrupted and in turn increases the time lag between samples. The time spent between samples is usually greater than what you've saved by mixing.

Mixing can be performed at the beginning, middle, or end of the sample heating period, although mixing at the beginning is usually not recommended.

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This is because the sample temperature will not yet have stabilized at the chosen value. If the temperature continues to rise after mixing, the equilibrium will shift, and you lose the benefits of mixing.

However, if you mix for the entire period of sample equilibration, the sample will reach equilibrium in the shortest period. This is because mixing also speeds the rate at which the sample reaches thermal equilibrium. So, mixing for the entire sample equilibration period is especially useful when you need to run a small number of samples in the shortest time possible.

Setting Mix Time Values

To mix continuously for the entire sample equilibration time:

1. Set the Sample Equilibrate time to 0.
2. Set the Mix time to equal the desired total Sample Equilibrate time.
3. Set the Stabilize time to 0.

To mix at the beginning of the sample heating cycle:

1. Set the Sample Equilibrate time to 0.
2. Choose Mix time for the first portion of the desired sample heating period.
3. Set the Stabilize time for the remainder of the desired heating period.

To mix in the middle of the sample heating cycle:

1. Set the Sample Equilibrate time for the first portion of the heating cycle. Most samples require 7 to 10 minutes to reach thermal equilibrium.
2. Set the desired Mix time, as discussed previously.
3. Set the Stabilize time for the last portion of the heating cycle.

To mix at the end of the sample heating cycle:

1. Set the Sample Equilibrate time for the first portion of the heating cycle.
2. Set Mix time for the remainder of the desired heating time.
3. Set Stabilize time to 0.

Mixing Power: General Guidelines

The amplitude of the mixer should be set as high as possible to provide vigorous mixing, but not so high that liquid splashes onto the septum. (Liquid on the septum may contaminate the sample needle and loop.) The power setting is generally determined by the weight of the sample. Use the following guidelines to determine an appropriate power setting:

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22 ml vials

- For 22 ml vials filled to 75% capacity, set the amplitude to 10 (the maximum).
- For low viscosity liquids (e.g. aqueous solutions) in a 22 ml vial, turn the amplitude down from 10 to 1 as the sample volume drops from 75% to 5% of volume.
- For higher viscosity fluids (e.g. oils) in a 22 ml vial, turn the amplitude down from 10 to 3 as the sample volume drops from 50% to 5% of volume.

12 ml vials

- The maximum mix power recommended for 12 ml vials filled to 75% capacity is 6.
- Turn the amplitude down from 6 to 1 as the sample volume in a 12 ml vial drops from 75% to 5% of volume.

9 ml vials

- The maximum mix power recommended for 9 ml vials filled to 75% capacity is 4.
- Turn the amplitude down from 4 to 1 as the sample volume in a 9 ml vial drops from 75% to 5% of volume.

Vial Pressurization Values

In headspace analysis, the sample vial is pressurized to enable the sample to fill the sample loop. There must be enough pressure to allow the sample vapor to completely fill the loop, but not so much that the headspace vapor dilutes, reducing sensitivity. You should avoid excessive pressure values. Some samples (e.g. most liquids) will self-pressurize to some extent via solute and solvent vapors in the headspace. The level of pressure reached is known as the "static vial pressure". The static vial pressure should be determined before deciding on a pressurization level:

To determine the static vial pressure:

1. Turn the Vial Needle Flow Controller fully off (clockwise).
2. Turn the Vial Pressurization Controller to its full open position (clockwise).
3. Build and activate a Method with a Pressurize time of 10 minutes. Use the same Platen Temperature, Equilibration Time, and Mixing parameters that you will be using for samples.
4. Prepare a sample vial with an appropriate sample, and insert it into the 7000. Start the method.
5. At the Pressurization step, note and record the pressure indicated on the Vial Pressure Setting gauge. This value represents the static vial pressure.

continued

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Note: The pressure determined by this procedure will be slightly less than the pressure in the vial before puncturing with the sample needle. This is because the vapor must fill a small volume of tubing to reach the pressure gauge. However, the value as determined here will be the correct value to use in setting the Pressurization value.

Determining the Vial Pressurization Setting

The proper value to use for vial pressurization depends on the sample headspace volume, the static vial pressure, and the sample loop size. The pressure must be great enough so that the sample loop is completely filled with sample vapor. There must be enough pressure for the loop volume plus 0.2 ml of sample vapor to exit the vial before dropping to ambient pressure. A value higher than the minimum is recommended for best reproducibility. The minimum pressure can be determined by the following equation:

$$P_{min} = \frac{14.7\,(V_g + V_l + 0.2)}{V_g} - 14.7$$

where:

$\mathbf{V_g}$ = volume of headspace in the vial, ml

$\mathbf{V_l}$ = volume of sample loop, ml

This pressure value should provide enough sample to *just* fill the loop. A higher setting is recommended for better reproducibility.

In all cases, the pressurization value must be set at least equal to the static vial pressure. If lower values are set, sample vapors will backstream into unheated supply lines, causing contamination. Tekmar recommends a value of at least 3 psig above the static vial pressure.

When using a Variable Injection Pressure Regulator (VIPR™) you will need a higher pressure setting. This is discussed in greater depth later in this section.

To set the pressurization level:

1. Open the Vial Needle Flow Controller by turning three revolutions counter-clockwise.
2. Press digit #**3** on the 7000 keypad while in the **Standby** screen.
3. Press digit #**4**, **Outputs**.
4. **PAGE UP** to **Pressure**.
5. Press **Y** for yes to turn on the pressurization valve.
6. Set the desired pressure using the Vial Pressurization Control knob.
7. The value can be adjusted by pushing **Y** to turn the valve on and **N** to turn it off while adjusting the control knob.
8. Press **F4** to exit.

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Determining the Vial Flow Rate

The vial flow rate establishes both the rate at which sample vials are pressurized, as well as how much gas sweeps the sample needle and loop between samples. This flow rate is not critical, as long as you maintain a minimum flow. A typical setting is 40 ml/min.

To set the Vial Flow Rate:

1. Place an empty, sealed vial in position 1.
2. From the Standby mode, press digit #**3** on the keypad.
3. Select #**3**, Vent Check.
4. Press **LOAD**.
5. Once the vial is on the needle, measure the flow rate at the Vent port on the rear panel with a flow meter (p/n 13-0079-000). Adjust the flow rate to the desired value using the Vial Needle Flow Control knob.
6. Press **UNLOAD** to remove the vial.
7. Press **F4** to exit.

Pressurization and Injection Time Settings

There are a number of time settings associated with moving sample vapor from the vial to the column. Each of these is set independently, for maximum operating flexibility. The settings for most of these parameters require only a minimum time value; others require more careful optimization.

Vial Pressurize Time

You can determine pressurize time by noting the time it takes for the needle on the pressurization gauge to deflect to the final pressurization value ***once the sample vial is on the needle.*** Pressurize time is dependent on the vial size, phase ratio, pressurization setting, and vial flow rate. It is recommended that you use a time equal to that determined for an empty vial (phase ratio = 0). This provides a safety factor for all samples.

To determine vial pressurization time:

1. Place a sealed, empty vial of appropriate size in carrousel position 1 (platen position 1 with 7000 only.)
2. Set up your method with the appropriate values.
3. Enter a five minute pressurization time.
4. Activate the method and allow the sample to pressurize.
5. Record the time required for the needle of the pressure gauge to deflect to the set value. (Note: a stopwatch useful for this operation is included in the Parameter Optimization Kit, p/n 14-5046-000.)
6. Add 0.1 minutes to the determined value to yield the optimal pressurization time for your method.

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Pressurize Equilibrium Time
This time allows the gas in the vial to stabilize before filling the sample loop. Typically only a short time of about 0.05 minutes is required. Longer times, up to about 0.25 min., are recommended for three-phase samples (e.g. soils).

Loop Fill Time
The time required to fill the loop is based on the total time it takes for the pressure in the sample loop to stabilize. Factors affecting this time include vial pressure, loop size, and valve oven temperature. This time can be determined by monitoring the flow out of the vent with a Parameter Optimization Kit during the Loop Fill mode. Once the flow has stopped, the pressure stabilizes. Typical values are 0.10 to 0.30 min.

To determine the appropriate loop fill time:

1. Attach the Parameter Optimization Kit (14-5046-000) to the vent line:

    1. Fill your vial with sample.
    2. Place the vial in the carrousel (or in the platen position 1 if you do not have a carrousel) and start the 7000.
    3. Allow the sample to equilibrate at a proper temperature and time (see Sample Equilibrate mode, Section 11).
    4. With the 3-prong clamp, squeeze the bulb to "pull" a bubble up to the 0 ml line.
    5. When the 7000 goes into Loop Fill mode, determine the point at which the bubble stops and, using the stop watch, record the amount of time it took the bubble to reach that point.
    6. Set the Loop Fill Parameter equal to the time that it took for the bubble to stop, plus 0.1 min.

    Note: The Loop Fill time must be at least 2.5 times the physical size of the loop to ensure a proper sweep. For example, a 2 ml loop must have a 5 ml sweep. If you find that, after following the steps above to determine the Loop Fill parameter, your value is less than 2.5 times the loop size, increase the parameter value so that it is at least 2.5 times the loop size.

Loop Equilibrium Time
This time allows the sample vapor in the loop to stabilize before injection into the GC. A time of 0.05 min. is recommended for most samples.

continued

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Inject Time

The Inject time depends on the sample loop size and the Transfer Line Flow rate (GC carrier flow). The loop should be flushed with three to five volumes of gas. Three volumes is the minimum required to ensure quantitative transfer. Volumes greater than five may lead to excessive peak broadening. The range of times can be calculated from the following equations:

$$T_{min} = \frac{\text{(Sample Loop Volume)}}{\text{(Transfer Line Flow Rate)}} \times 3$$

$$T_{max} = \frac{\text{(Sample Loop Volume)}}{\text{(Transfer Line Flow Rate)}} \times 5$$

Setting the Variable Injection Pressure Regulator (VIPR™)

The Variable Injection Pressure Regulator (VIPR™) is a 7000 accessory designed to increase sensitivity. It achieves this by establishing a backpressure on the sample loop during the Loop Fill step. This keeps the sample vapor in the loop at a pressure higher than at ambient, which would otherwise be achieved.

Higher pressure means that there is more sample vapor in the loop, and more sample injected into the GC. This leads to higher sensitivity. It also can improve reproducibility. If the ambient pressure is highly variable (i.e. the weather is changing), the pressure in the sample loop during Loop Fill will fluctuate. When using the VIPR, however, the sample loop is isolated from atmospheric pressure. This leads to better stability of responses on a day-to-day basis.

VIPR settings are generally 2-3 psi lower than the Vial Pressurization setting. Do not set the VIPR higher than this, or the sample loop may not adequately fill during Loop Fill mode.

To set the VIPR:

1. Press digit #3 in **Standby** mode.
2. Press digit #3, Vent Check.
3. Place an empty, sealed vial in Carrousel position 1 (platen position 1 when the 7050 is not installed) and press **LOAD**.
4. Set the desired pressure using the pressure knob on the front panel on the front panel of the VIPR.
5. Press **F4** to exit.

continued

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Parameter Worksheet

Below is an example of a worksheet that you can use to determine and document the parameter settings of an analysis. Each of the parameters has been previously explained in this chapter.

Headspace Analysis
Pneumatic Parameter Worksheet

Loop Size:_____ (ml) Vial Size:_____ (ml)
Sample Size:_____ (ml or gm.) Phase Ratio*:_____ (V_g/V_m)
VIPR Setting:_____(psig)

1) Static Vial Pressure:_____ psi
2) Static Vial Loop Fill Volume:_____ ml
3) Vial Pressurization Setting:_____ psi
4) Vial Pressurization Flow Rate: _____ ml/min.
5) Vial Pressurization Time:_____ min.
6) Loop Fill Volume: _____ ml
7) Loop Fill Time: _____ min.
8) Pressurization Gas Volume**:_____ ml
9) Injection Pressure Differential***:_____ psi
10) Transfer Line Flow Rate:_____ ml/min.
11) Inject Time:_____ min.
12) Sample Loop Flush Volumes****:____

* Phase Ratio = $\frac{\text{volume of vial - volume of sample}}{\text{volume of sample}}$

** Pressurization Gas Volume = Loop Fill Volume - Static Loop Fill Volume

*** Injection Pressure Differential = Vial Pressurization Setting - VIPR Setting

**** Sample Loop Flush Volumes = (Loop Volume/Inject Time)/Transfer Line Flow Rate

Figure 9-5 Parameter Worksheet

continued

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Method Optimization Mode™ (MOM)

Method Optimization Mode™ (MOM) is a unique control program on the 7000 that allows you to automatically optimize your operating parameters for best performance. In MOM, a series of samples are run while undergoing incremental changes in a pre-assigned setpoint. After the run is completed, you can review the results and determine the optimal value. This automated approach allows you to test all possible setpoints overnight. Parameters which may be selected for incremental changes include: Platen Equil, Sample Equil, Platen Temp, Mix Power, Mix, Stabilize, Pressurize, Press Equil, Loop Fill, Loop Fill Equil, and Inject.

⚠ CAUTION

* **Platen temperature should never exceed 10° below the boiling point of the solvent in the vial. Exceeding this value may create a dangerous overpressurization situation in the vial, and cause the cap and septum assembly to leak and/or pop off of the top of the vial. The maximum optimal platen temperature is usually 15° - 20° below the boiling point of the sample solvent. Do not increment the platen temperature beyond 10° below the boiling point of the sample solvent.**

When using MOM, you will need to decide which parameter you wish to optimize first. (Only one parameter can be varied at a time.) Enter lower and upper values for this parameter, then add a value to increment. For example, you may choose to vary the sample equilibration time. (Sample equilibration time is the parameter most often optimized with MOM.) An initial time of 10 minutes is common, and a final time of 90 minutes is usually more than adequate. Depending on how many samples you wish to run, an increment value of five minutes (18 samples) or 10 minutes (nine samples) may be used. To validate your data, you may choose to run each increment up to nine times.

Note: The increment value must divide evenly into the range, so that the final run ends precisely on the upper value. The 7000 will not allow you to enter values that do not divide evenly. Upon completion of the runs, you can plot peak area against equilibration time to determine the sample equilibration time.

Optimizing Time Parameters

The majority of time parameters require that you only determine the minimum value. For example, once the sample equilibration time has been determined, times longer than this value have no added benefit. Usually

continued

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longer values do not hurt the performance, but they do affect the sample throughput. Therefore, once the proper value has been established, it is unnecessary to use longer times.

Optimizing Temperature Parameters

Temperatures are usually critical to the performance of a sample method. Minimum temperatures are necessary to achieve adequate sensitivity without carryover problems. Excessive temperatures may result in sample breakdown (such as labile components) or even a hazard such as over-pressurization of a sample vial. Temperatures are optimized by starting low, and slowly raising the temperature. (Starting high and lowering the temperature is not practical due to the time required for some heated zones to cool down.) As a general rule, all heated zones should be kept at least equal to the platen temperature. This prevents any possibility of condensation.

Tracking Mode

"Tracking" is a MOM feature that allows the platen, sample loop, and transfer line temperatures to increase by the same increment during a run. This prevents condensation and reduces the chance of thermal degradation of sample components. The tracking feature can also be disabled.

Note: When tracking is enabled with platen optimization, the system will track the sample loop and transfer line temperatures. Tracking with sample loop optimized tracks the line temperature only. See pages 8-27 - 8-28 for more information on Tracking.

To program MOM, see pages 8-22 - 8-28.

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9.6 Example Application Parameters

The following pages contain examples of parameters that have been found to be effective in certain applications. You can further optimize your method by using the Method Optimization Mode™ on the 7000 (see Section 9.5 of this manual for more information).

BLOOD ALCOHOLS

Sample Loop:	0.5 ml loop
Platen Temperature:	65°C
Platen Equilibration Time:	0 min
Sample Equilibration Time:	14 min
Vial Size:	22 ml
Mixer:	Off
Mixing Time:	NO
Mix Power:	NO
Stabilize Time:	NO
Cryo Cooldown:	NI
Minutes at:	NI
Pressurize Setting:	10 psi at 90 ml/min
Pressurize Time:	0.4 min
Pressurize Equilibrium Time:	0.25 min
Loop Fill Time:	0.25 min
Loop Equilibrium Time:	0.1 min
Inject:	0.5 min
Cryo Inject:	NI
Minutes at:	NI
Valve Temperature:	120°C
Line Temperature:	120°C
Cryo Union Heater:	NI
Injections Per Vial:	1
GC Cycle Time:	4 min
Parameter Optimization:	Off
Detector:	FID
Column:	6' x 1/8" S.S., 0.2 Carbowax, 1500 on 60/80 Carbopak
Injector Temperature	200°C
Detector Temperature	200°C
Carrier Flow:	20-30 ml/min
Initial Temperature:	100°C for 2 min

Note: The Polar Kit (p/n 14-6058-002) is recommended for proper operation. Saturate the blood sample with NaCl. High gas purity is required (99.999%).

⚠ CAUTION

Keep the end of the transfer line short and insulated to prevent cold spots between the end of the transfer line and the injection port.

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DIOXANE IN WATER

Sample Loop:	1 ml loop
Platen Temperature:	85°C
Platen Equilibration Time:	0 min
Sample Equilibration Time:	60 min
Vial Size:	22 ml
Mixer:	On
Mixing Time:	5 min
Mix Power:	2
Stabilize Time:	1 min
Cryo Cooldown:	NI
Minutes at:	NI
Pressurize Setting:	12 psi at 40 ml/min
VIPR Setting:	8 psi
Pressurize Time:	0.25 min
Pressurize Equilibrium Time:	0.05 min
Loop Fill Time:	0.25 min
Loop Equilibrium Time:	0.05 min
Inject:	1 min
Cryo Inject:	NI
Minutes at:	NI
Valve Temperature:	85°C
Line Temperature:	85°C
Cryo Union Heater:	NI
Injections Per Vial:	1
GC Cycle Time:	30 min
Parameter Optimization:	Off
Detector:	FID
Column:	DB-5, 60M, 0.32 um
Carrier Flow:	4cc/min
Initial Temperature:	40°C for 5 min
Rate:	6°C/min to 100°C
Hold Time:	1 min

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USEPA 502 ANALYTES IN SOILS

Sample Loop:	1 ml loop
Platen Temperature:	85°C
Platen Equilibration Time:	0 min
Sample Equilibration Time:	50 min
Vial Size:	22 ml
Mixer:	On
Mixing Time:	10 min
Mix Power:	8
Stabilize Time:	0.1 min
Cryo Cooldown:	NI
Minutes at:	NI
Pressurize Setting:	15 psi at 40 ml/min
Pressurize Time:	0.20 min
Pressurize Equilibrium Time:	0.08 min
Loop Fill Time:	0.10 min
Loop Equilibrium Time:	0.08 min
Inject:	1 min
Cryo Inject:	NI
Minutes at:	NI
Valve Temperature:	85°C
Line Temperature:	85°C
Cryo Union Heater:	NI
Injections Per Vial:	1
Detector:	Ion Trap or Mass Spec
Column:	DB-VRX, 60M, 0.32 um, 1.8 u md_f
Initial Temperature:	10°C for 5 min
Ramp 1 Temperature:	5°C/min to 100°C
Ramp 2 Temperature:	15°C/min to 180°C
Ramp 3 Temperature:	10°C/min to 200°C
Final Temperature:	200°C, hold for 2 min
Carrier Flow:	1 ml/min

Note: E-Form Option Kit (p/n 14-5531-000) is recommended for proper operation.

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POLYMERS

Sample Loop:	1 ml loop
Platen Temperature*:	200°C
Platen Equilibration Time:	0 min
Sample Equilibration Time:	30 min
Vial Size:	22 ml
Mixer:	Off
Mixing Time:	NI
Mix Power:	NI
Stabilize Time:	NI
Cryo Cooldown:	NI
Minutes at:	NI
Pressurize Setting:	12 psi at 40 ml/min
VIPR Setting:	9 psi
Pressurize Time:	0.3 min
Pressurize Equilibrium Time:	0.05 min
Loop Fill Time:	0.3 min
Loop Equilibrium Time:	0.05 min
Inject:	1 min
Cryo Inject:	NI
Minutes at:	NI
Valve Temperature:	200°C
Line Temperature:	200°C
Cryo Union Heater:	NI
Injections Per Vial:	1
Detector:	FID
Column:	DB-1, 30M, 0.53 um
Carrier Flow:	8cc/min
Initial Temperature:	35°C for 10 min
Rate:	6°C/min to 200°C
Hold Time:	20 min

*The sample temperature of polymers is highly dependent upon the nature of the sample and the goal of the analysis. If analyzing for residual solvents, you should not exceed the decomposition temperature of the polymer (which may be as low as 80°C). Higher values may be used to study decomposition behavior.

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502 ANALYTES IN DRINKING WATERS

Sample Loop:	1 ml loop
Platen Temperature:	85°C
Platen Equilibration Time:	0 min
Sample Equilibration Time:	30 min
Vial Size:	22 ml
Mixer:	On
Mixing Time:	2 min
Mix Power:	5
Stabilize Time:	2 min
Cryo Cooldown:	NI
Minutes at:	NI
Pressurize Setting:	6 psi at 40 ml/min
Pressurize Time:	0.3 min
Pressurize Equilibrium Time:	0.05 min
Loop Fill Time:	0.3 min
Loop Equilibrium Time:	0.05 min
Inject:	1 min
Cryo Inject:	NI
Minutes at:	NI
Valve Temperature:	85°C
Line Temperature:	85°C
Cryo Union Heater:	NI
Injections Per Vial:	1
GC Cycle Time:	42 min
Parameter Optimization:	Off
Detector:	FID
Column:	DB-624, 60M, 0.53 um
Carrier Flow:	6cc/min
Initial Temperature:	35°C for 5 min
Rate:	3°C/min to 100°C
Hold Time:	1 min

Note: E-Form Option Kit (p/n 14-5531-000) is recommended for proper operation.

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FLAVORS IN FOODS

Sample Loop:	1 ml loop
Platen Temperature:	65°C
Platen Equilibration Time:	0 min
Sample Equilibration Time:	30 min
Vial Size:	22 ml
Mixer:	On
Mixing Time:	5 min
Mix Power:	6
Stabilize Time:	1 min
Cryo Cooldown:	-175°C
Minutes at:	15 min
Pressurize Setting:	8 psi at 50 ml/min
Pressurize Time:	0.25 min
Pressurize Equilibrium Time:	0.1 min
Loop Fill Time:	0.1 min
Loop Equilibrium Time:	0.1 min
Inject:	1 min
Cryo Inject:	200°C
Minutes at:	0.75 min
Valve Temperature:	65°C
Line Temperature:	65°C
Cryo Union Heater:	65°C
Injections Per Vial:	1
Transfer Line	0.53 mm I.D. fused silica
Detector:	FID at 300°C
Column:	DB-5, 30M, 0.53 um, 1.5 u md_f
Carrier Flow:	8 ml/min
Initial Temperature:	35°C for 5 min
Rate:	5°C/min to 100°C
Hold Time:	1 min

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FULL EVAPORATION TECHNIQUE

Sample Loop:	1 ml loop
Platen Temperature*:	115°C
Platen Equilibration Time:	0 min
Sample Equilibration Time:	5 min
Vial Size:	22 ml
Mixer:	Off
Mixing Time:	NI
Mix Power:	NI
Stabilize Time:	NI
Cryo Cooldown:	NI
Minutes at:	NI
Pressurize Setting:	15 psi at 40 ml/min
VIPR Setting:	12 psi
Pressurize Time:	0.25 min
Pressurize Equilibrium Time:	0.05 min
Loop Fill Time:	0.25 min
Loop Equilibrium Time:	0.05 min
Inject:	1 min
Cryo Inject:	NI
Minutes at:	NI
Valve Temperature:	115°C
Line Temperature:	115°C
Cryo Union Heater:	NI
Injections Per Vial:	1
GC Cycle Time:	30 min
Parameter Optimization:	Off
Detector:	FID
Column:	DB-1, 30M, 0.53 um
Carrier Flow:	8cc/min
Initial Temperature:	35°C for 10 min
Rate:	6°C/min to 200°C
Hold Time:	1 min

*The sample temperature of flavors is highly dependent upon the nature of the sample. Many flavor components are labile, and excessive temperature may change the nature of the sample. For example, temperatures in excess of 60° are not recommended for dairy products.

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RESIDUAL SOLVENTS IN PHARMACEUTICALS

Sample Loop:	0.5 ml loop
Platen Temperature:	85°C
Platen Equilibration Time:	0 min
Sample Equilibration Time:	10 min
Vial Size:	22 ml
Mixer:	On
Mixing Time:	2 min
Mix Power:	5
Stabilize Time:	0.1 min
Cryo Cooldown:	NI
Minutes at:	NI
Pressurize Setting:	15 psi at 50 ml/min
Pressurize Time:	0.35 min
Pressurize Equilibrium Time:	0.1 min
Loop Fill Time:	0.2 min
Loop Equilibrium Time:	0.1 min
Inject:	1 min
Cryo Inject:	NI
Minutes at:	NI
Valve Temperature:	85°C
Line Temperature:	85°C
Cryo Union Heater:	NI
Injections Per Vial:	1
GC Cycle Time:	45 min
Transfer Line:	0.53mm I.D. fused silica
Detector:	FID at 260°C
Column:	Rtx 1301, 30m, 0.53mm, 3 u d_f
Carrier Flow:	4.5 ml/min
Initial Temperature:	40°C for 20 min
Rate:	35°C/min to 240°C
Hold Time:	5 min

Note: E-Form Option Kit (p/n 14-5531-000) is recommended for proper operation.

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10.1 Overview of the Section

This section of the manual includes instructions for scheduling methods, aborting runs, and running priority samples.

10.2 Scheduling Methods (7050 Only)

Note: To perform method scheduling, the 7050 must be installed and acknowledged by the 7000 microprocessor.

When Method Scheduling is turned on, all runs must be scheduled or the 7000 will not acknowledge them. If you are using only one method for your automated run, turn Method Scheduling off.

Note: Method Scheduling allows you to run a single vial or sets of vials within up to four different methods. For instance, you could run one vial at different sample temperatures. Here is an example:

Vial 1, Method 1 - 80°
Vial 1, Method 2 - 90°
Vial 1, Method 3 - 120°
Vial 1, Method 4 - 150°

⚠ CAUTION

*** Platen temperature should never exceed 10° below the boiling point of the solvent within the vial. If you exceed this value, the cap and septum assembly may leak and/or pop off of the top of the vial. The maximum optimal platen temperature is usually 15 - 20° below the boiling point of the sample solvent. Do not over-increment the platen temperature beyond 10° below the boiling point of the solvent in the vial.**

Note: Full Evaporation Technique is an exception to the above due to the limited volume (<20 ul).

To enable the 7000 to acknowledge the 7050:

1. In the Standby screen press **F4 (Conf)** for the Current Configuration screen.

2. Press **F3 (Inst)** to view the Instrument Configuration screen.

3. If the configuration for the Automatic Sampler reads "**N**", press **"Y"** on the keypad and press **F4 (Exit)** to return to the Current Configuration screen.

Note: The configuration will not allow a "Y" unless the 7000 has been powered up and allowed to self-test.

4. Press **F4 (OK)** to return to the Standby screen.

continued

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To access the method scheduling mode:

1. Press **F2 (A/S)** in the Standby screen for the Automatic Sampler Control screen:

```
Automatic Sampler Control
Current position: 1          Platen: 1
Start:  1          Stop:  1
Enable method schedule:  N
Help      Sched       ->       Exit
```

2. If you are using only one method to analyze a batch of samples, press **F3 (->)** to move the cursor to highlight "Enable Method Schedule". Press **"N"** for no.

   Note: If you choose not to schedule a method (Enable Method Schedule: N), the 7000/7050 will run vials using the existing Method 1-4 with the start-to-stop positions as shown in the above screen.

3. For multiple method runs, select Enable Method Schedule: **Y** (press **F3 (->)** to move the cursor to highlight the Method Schedule option in the Automatic Sampler Screen).

4. Press **Y** on the keypad (Method Scheduling now activated) to create a schedule.

5. Press **F2 (Sched)** to access the Method Scheduling screen:

```
          Enable method schedule:  Y
Start:       Stop:      Method:
Start:       Stop:      Method:
Start:       Stop:      Method:
Start:       Stop:      Method:
Help      Clear       ->      Exit
```

Note: If you want to delete a previous method schedule, press F2 (Clear).

6. Press **F3 (->)** to position the highlighted box at the first start position on the screen.

continued

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7. Press **ENTER** on the keypad to access the Start Position screen:

```
Current:  1
Minimum:  1       Maximum:  50
Start:


        <Press ENTER for no change>
```

8. Enter a designated start position for your first method schedule and press **ENTER** again.

9. Press **F3 (->)** to position the highlighted box at the first stop position in the screen.

10. Press **ENTER** to access the Stop Position screen:

```
Current:  1
Minimum:  1       Maximum:  50
Stop:


        <Press ENTER for no change>
```

11. Enter a designated stop position for your first method schedule and press **ENTER** again.

12. Press **F3 (->)** to position the highlighted box at the first Method position in the screen.

13. Press **ENTER** to access the Method screen:

```
Current:  1
Minimum:  1       Maximum:  4
Method:


        <Press ENTER for no change>
```

continued

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14. Assign a method number to your first schedule of runs and press **ENTER** again.

15. Repeat these steps to compete all your scheduled runs.

16. Press **F4 (Exit)** to return to the Autosampler screen.

17. Press **F4 (Exit)** to return to the Standby screen.

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10.3 Aborting Runs/ Priority Sampling

The abort function lets you stop a run in progress or interrupt an automated sample batch to run a priority sample. **Note: The abort option is only available while a run is in progress.**

To access the Abort Mode:

1. Press **F2 (A/S)** from any operation mode screen during a run and the **F3 (->)** will be replaced with **F3 (Abort)** in the Autosampler screen:

```
Automatic Sampler Control
Current position:  1       Platen: 1
Start:  1         Stop:  3
Enable method schedule:  N

Help    Sched     Abort     Exit
```

2. Press **F3 (Abort)** to access the Abort screen:

```
F1 - Finish this one, then abort
F2 - Finish all in platen
F3 - Abort immediately
F4 - Don't abort

One      Plat      Now      Exit
```

3. ***Choose one of the following options:***

- **F1 (One) to finish the vial in progress and abort the rest of the scheduled run.** When you press **F1 (One)**, the 7000 returns to the interrupted mode of operation, and finishes the vial in process. Then it will enter the Auto Unload mode and unload the rest of the vials from the platen to their original position in the carrousel.

- **F2 (Plat) to finish all the vials in the platen.** When you press **F2 (Plat)**, the 7000 returns to the interrupted mode of operation, finishes all the vials in the platen and returns to the Standby screen.

- **F3 (Now) to immediately abort the run in process.** When you press **F3 (Now)**, the 7000 enters the Auto Unload mode to retrieve the vial and return it to the platen. The 7000 empties the platen and returns to the Standby mode.

- **F4 (Exit) to escape the abort option and return to the interrupted mode of operation.** When you press **F4 (Exit)**, the 7000 operation picks up the run where it left off when you selected **(Abort)**.

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10.4 Running an Unscheduled Automated Method

The 7000 has four default methods that are based on typical samples. To run a sample using the default parameter values (already configured into the system), see pages 12-9 - 12-10 for instructions.

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11.1 Overview of the Section

This section of the manual covers the sequence of operation in a typical 7000 Headspace Autosampler method.

The 7000 has 11 different program modes. This section describes these modes in their sequential order:

- Standby
- Platen Equilibrate
- Sample Equilibrate
- Mixing
- Stabilize
- Pressurize
- Pressure Equilibrate
- Loop Fill
- Loop Equilibrate
- Inject
- Re-Equilibrate*

*If using Multiple Headspace Extractions (see Section 8 for more information on Re-Equil Mode).

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11.2 Sequence of Operation-Summary

The 7000 Headspace Autosampler is based on a valve and loop sampling approach. The 7000 has separate, independent gas flow systems for vial pressurization and carrier gas functions. Separating pressurization and carrier gases allows you to optimize each flow system individually for the best possible analytical results.

The flow diagram (Figure 11-1) illustrates the gas flow system of the 7000. Gases enter through the two inlets -- **carrier** and **pressurize.**

From the carrier inlet on the back of the 7000, carrier gas is directed through a flow controller (**F2**). A pressure gauge (**G2**) measures pressure in this line. The flow passes into the heated valve oven section (**H1**) through the 6-port valve (**V6**), then through the heated transfer line (**H2**), and into the GC. This provides the column with carrier gas at a rate set at **F2**. The back pressure of the column can be read at **G2**.

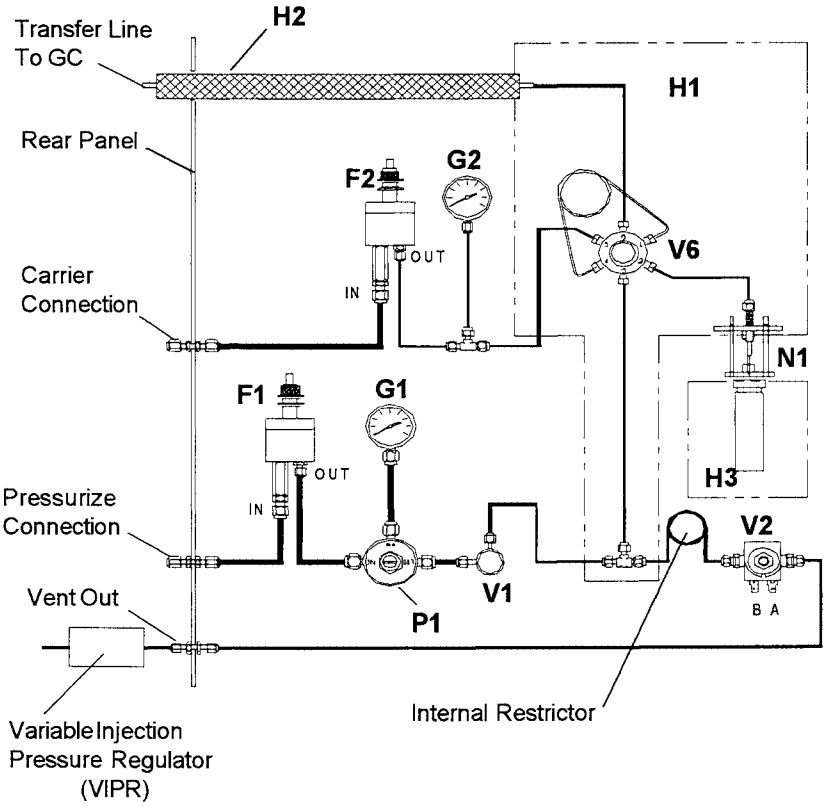


Figure 11-1 7000 Flow Diagram

continued

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The following steps show the sequence of operation in a 7000 run. Refer to the diagram on the previous page.

Step	*Function*	*Mode*
Step 1	Carrier gas flows through the flow controller **(F2)**, eventually reaching the GC column.	Standby
Step 2	Additional gas, regulated to a selected flow rate at **F1**, sweeps the lines, loop, and needle.	Standby
Step 3	Samples are introduced to the platen heated zone and allowed to equilibrate.	Sample Equil.
Step 4	Samples are mixed and heated for a programmed period of time.	Mixing (optional)
Step 5	Samples continue to heat for a specified amount of time after mixing.	Stabilize
Step 6	The sample vial is raised onto the needle **(N1)**, puncturing the septum. Pressurization gas fills the vial, pressurizing it to the pressure regulator **(P1)** setpoint. The vial pressurization value is displayed by **(G1)** when the vial is on the needle.	Pressurize
Step 7	The pressurize valve **(V1)** is closed. The vial may be allowed to pressure equilibrate for a brief period to ensure complete diffusion of the pressurization gas with the vial's atmosphere.	Pressure Equil.
Step 8	The vent valve **(V2)** is opened. The gas in the sample vial expands through the sample loop to the atmosphere, as the loop is filled with sample vapor.	Loop Fill
Step 9	The vent valve **(V2)** closes. This allows the sample vapor to equilibrate to the higher loop temperature, and the pressure and flows in the loop to stabilize.	Loop Equil.
Step 10	The 6-port valve **(V6)** switches, placing the sample loop in line with the carrier gas flow. Carrier gas backflushes the loop, sweeping the sample through the heated transfer line into the GC.	Inject
Step 11	The sample vial comes down off the needle and **V1** opens to resume pressurization gas sweep flow. **V2** opens for approximately 15 seconds to sweep the vent line with clean pressurization gas. **V2** closes for the next sampling cycle. Once injection is complete, **V6** is switched back to its original position as the 7000 completes its sequence of operation.	Inject

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Standby Mode

The 7000 system has four default methods that are programmed at the factory. The following pages give an explanation of the method parameters and the 7000 sequence of operation as shown in the summary on page 11-2. After you select your desired method, the introductory screen appears followed by the self test screens.

Once all of the self test screens are viewed (or skipped), the **Standby Mode** screen appears and remains until parameter setpoints are met:

```
Standby          Method 1
Platen Temp:   40° -> 40°
Sample Loop Temp:   40° -> 40°
Line Temp:   40° -> 40°

Meth       A/S       Temp       Conf
```

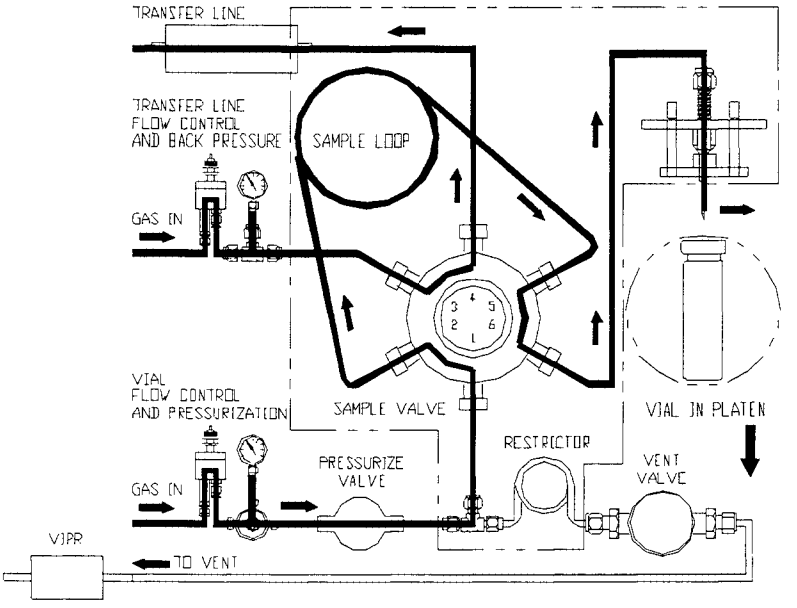


Figure 11-2 7000 Screen and Flow in Standby Mode

continued

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In Standby mode, carrier gas flows through the flow controller, eventually reaching the GC column. This flow is always on, constantly providing carrier gas to the GC. Flow to the column may be regulated by the 7000 or GC inlet flow controls. Optimal carrier flow settings will depend upon the column length, ID, and packing material (if applicable). An additional vial pressurization gas is regulated to selected pressure and flow rate values (Figure 11-2). This gas is used to pressurize the vial before injection and to reduce carryover by sweeping the sample path between injections.

Note: Optimum Standby flow ranges are determined by the length of time between injections - see Section 9, Developing Methods.

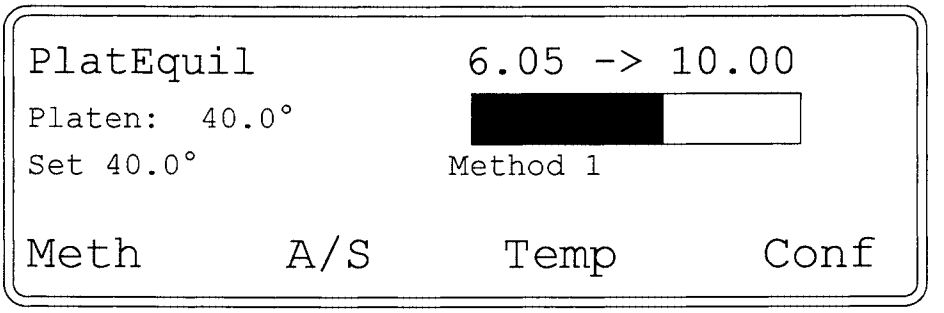
When the unit has met the parameter values for Method 1, it pauses at Standby until you press **STEP** on the keypad.

Anytime you enter a new platen setpoint, the Platen Equilibrate screen will appear, as shown on the next page.

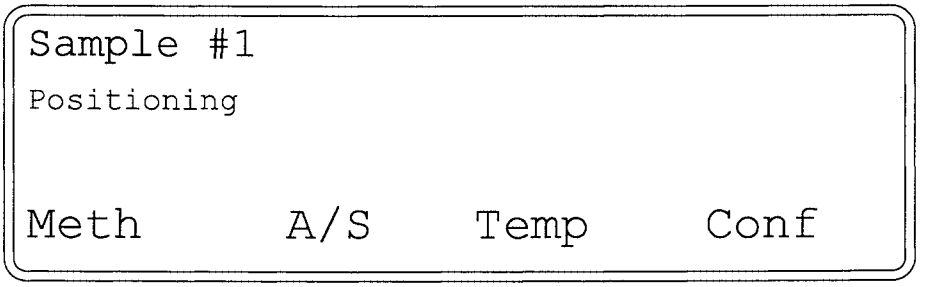
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Platen Equilibrate Mode

The **Platen Equilibrate** function allows the platen to equilibrate (stabilize) for a programmable time (0-60 minutes) after the setpoint temperature has been reached. A setpoint of 0-10 minutes is usually adequate.



Then the system automatically advances to the next screen:



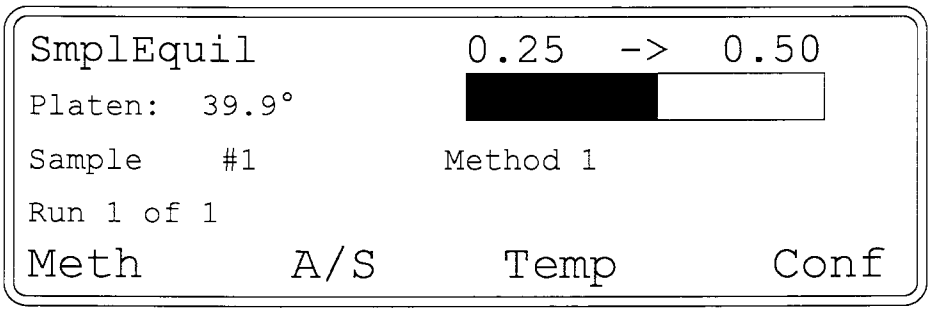
At this point the unit loads the first vial from the 7050 to the platen. Then the Sample Equilibration mode begins.

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Sample Equilibrate Mode

During **Sample Equilibrate Mode**, sample vials are heated in the platen for a specified length of time. This time value is equal to the time necessary for partitioned equilibrium to occur. If equilibration time is long enough, the 7000 will load multiple samples into the heated platen to maximize sample throughput.

During Sample Equilibrate mode, the following screen and valve configurations are activated:



Minimum and maximum programmable values for Sample Equil are 0-999.9 minutes.

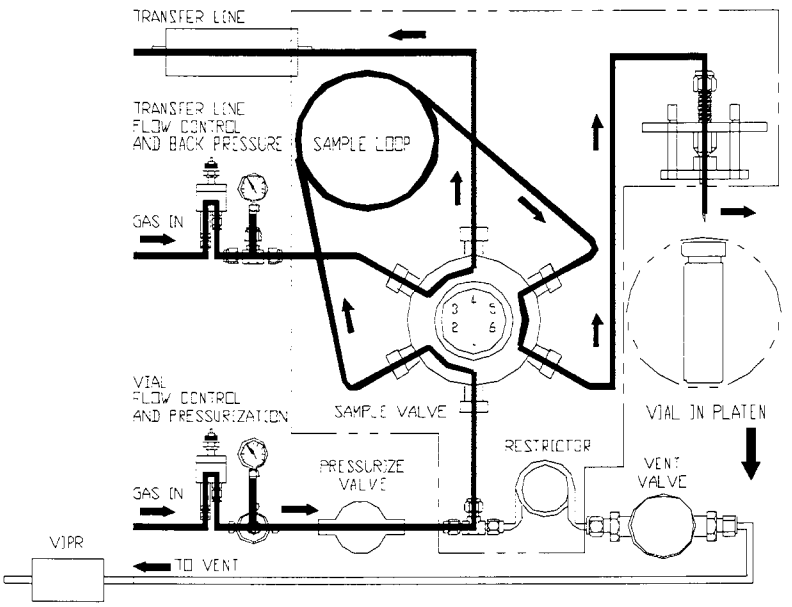


Figure 11-3 7000 Screen and Flow in Sample Equilibrate Mode

continued

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The vial's atmosphere or gas phase is referred to as the "headspace." The concentration of analytes increases in the headspace during Sample Equilibrate time. After sufficient time has elapsed, the concentration of analytes in the headspace may reach a steady state condition known as **equilibrium** (see Figure 11-4). Keep in mind that a sample may develop increasing concentrations of analyte over time ***without*** ever reaching equilibrium (see Figure 11-5). An example of non-equilibrating samples are some polymers that have an extremely slow partitioning rate.

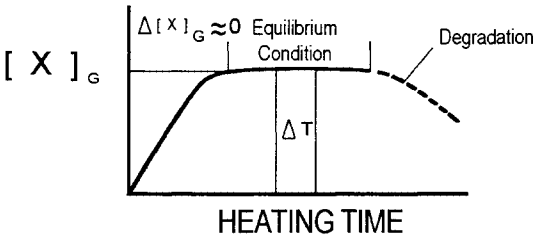


Figure 11-4 Equilibration Curve

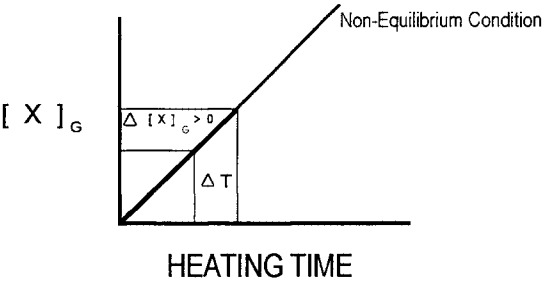


Figure 11-5 Non-Equilibrating

$[X]_G$ = Concentration of Analyte in Gas Phase of Headspace

$\triangle T$ = Change in Heating Time

$\triangle [X]_G$ = Change in Concentration of $[X]_G$

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Mixing Time and Power Modes

Mixing is a process in the 7000 that speeds equilibration and reduces sample thermal exposure time of the sample. The agitating action increases the surface area and ensures that the greatest amount of analytes possible will leave the liquid (or solid) phase and enter the gas phase in the vial. Mixing is effective for liquid samples, and for solid samples that have had a liquid extraction substance added to them.

```
Mixing
Raising Mixer

Meth      A/S      Temp      Conf
```

```
Mixing                    0.25 -> 0.50
Platen:  40.0°
Sample #1                 Method 1
Run 1 of 1
Mix Power: 1
Meth      A/S      Temp      Conf
```

Minimum and maximum programmable values for mixing are 0-999.9 minutes. Mix power minimum and maximum values are 1-10.

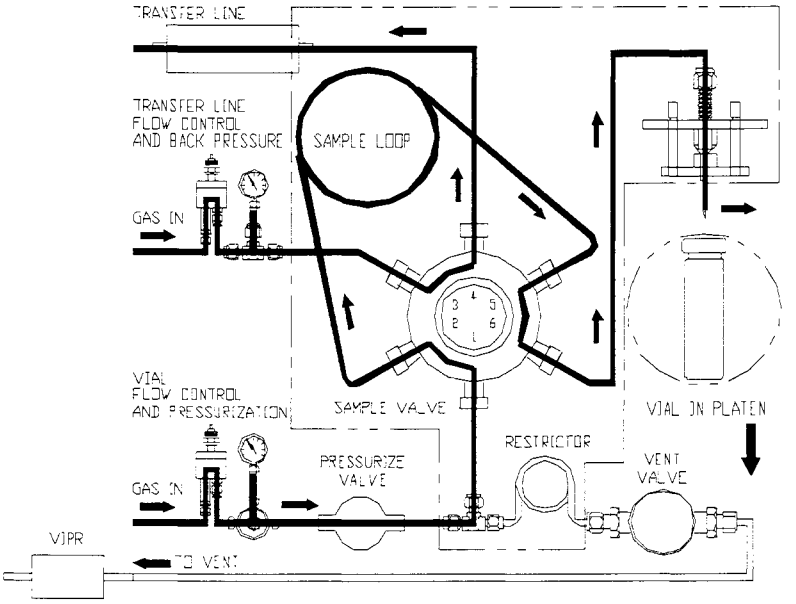
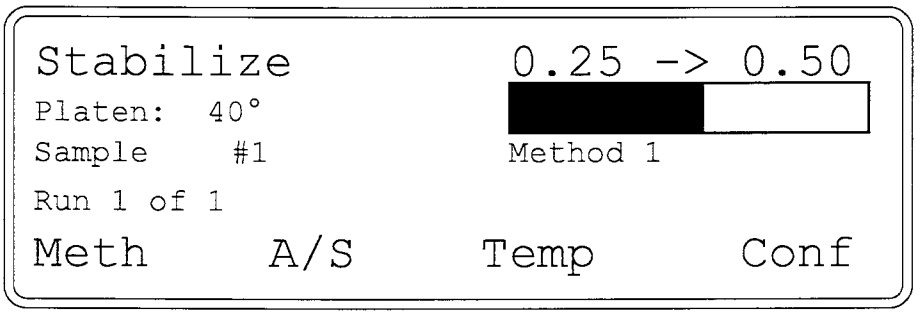


Figure 11-6 Screens and Flow in Mixing Mode

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Stabilize Mode

During **Stabilize Mode**, the vial equilibrates at rest after mixing. For samples that foam, aerosolize, or emit dust particles during mixing, Stabilize lets the sample settle before it is injected. Generally speaking, Stabilize time should not be greater than 2 - 5 minutes long.



Minimum and maximum programmable values are 0-60 minutes.

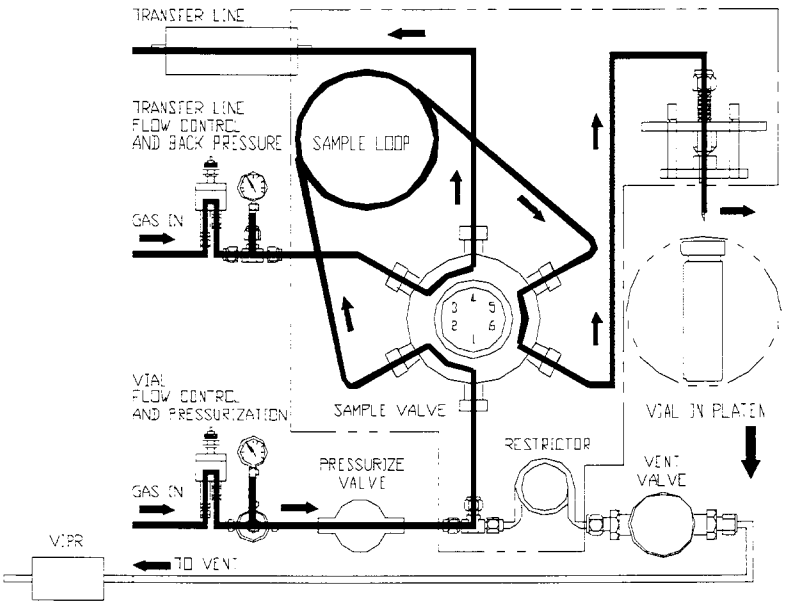


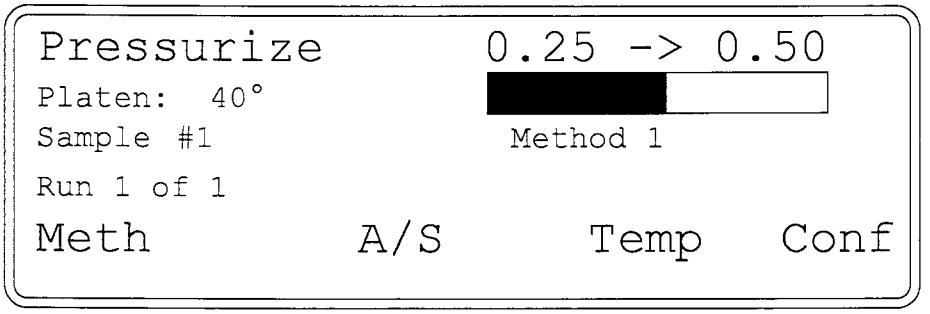
Figure 11-7 Screen and Flow in Stabilize Mode

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Pressurize Mode

In **Pressurize Mode**, the sample vial raises up onto the needle, which punctures the septum. Pressurization gas fills the vial, bringing it to the set vial pressurization value (3 - 27 psi).

Note: Pressurization time must be long enough for the vial to reach the maximum pressure as set on the vial pressurization regulator.



Minimum and maximum programmable values for Pressurize time are 0-60 min.

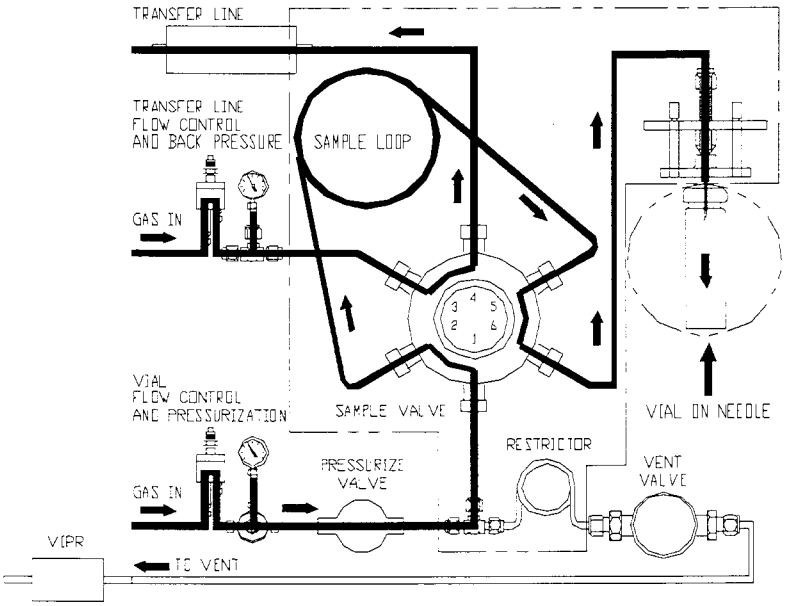


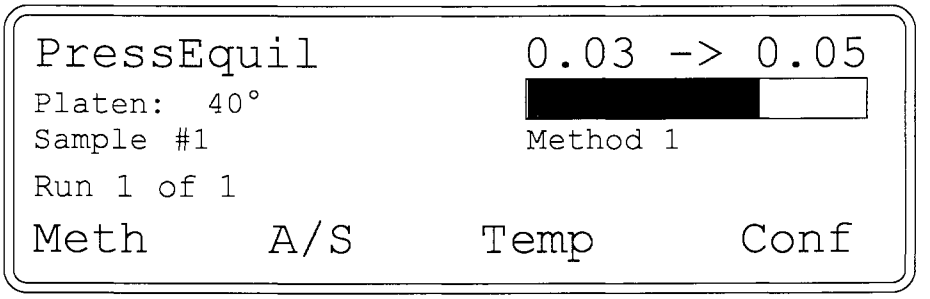
Figure 11-8 Screen and Flow in Pressurize Mode

continued

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Pressure Equilibrate Time Mode

During **Pressure Equilibrate Time Mode**, the pressurize valve and vent valve are closed, isolating the vial and allowing the newly-introduced pressurization gas to diffuse into the headspace atmosphere. Typical Pressure Equilibrate Time settings range from 0.05 to 0.25 minutes.



Minimum and maximum programmable values are 0-60 minutes.

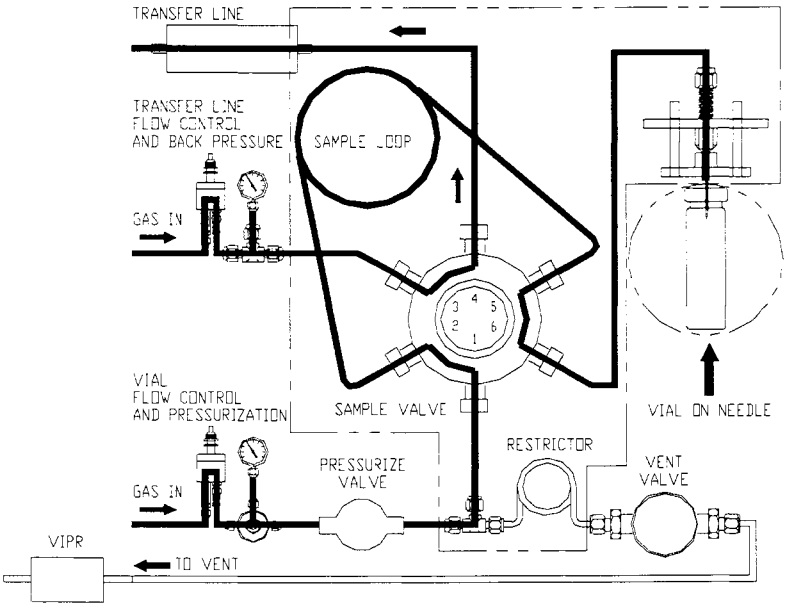
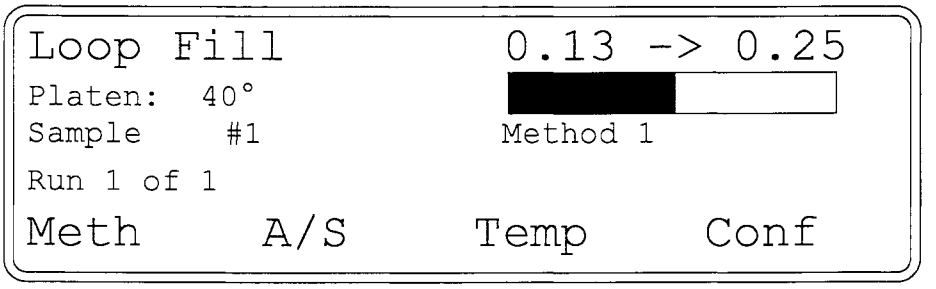


Figure 11-9 Screen and Flow in Pressure Equilibrate Time Mode

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Loop Fill Mode

In **Loop Fill Mode**, the vent valve opens. Pressure from the vial displaces the headspace through the sample loop and to the vent, filling the loop with the headspace contents. Typical Loop Fill times are 0.1 to 0.4 minutes (long enough to allow the vial contents to exhaust completely). See Section 9 for information on setting Loop Fill time.



Minimum and maximum programmable values are 0-60 minutes.

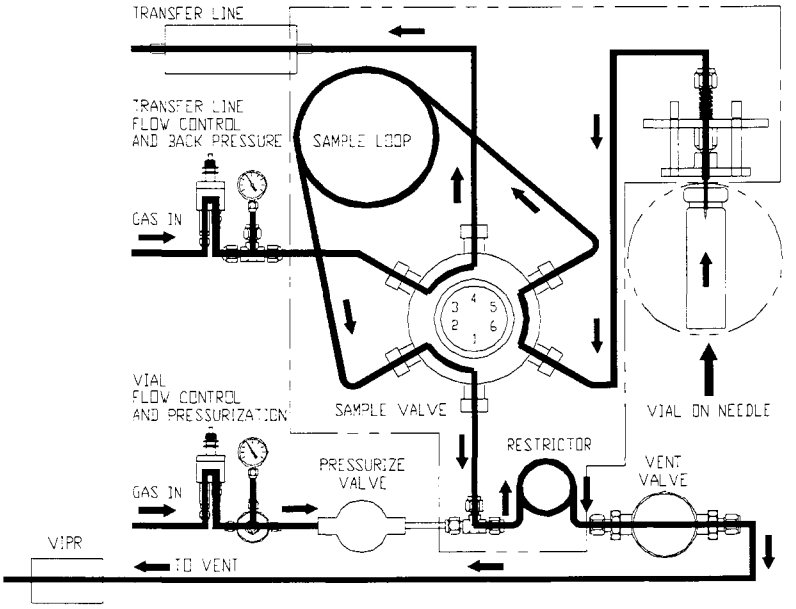
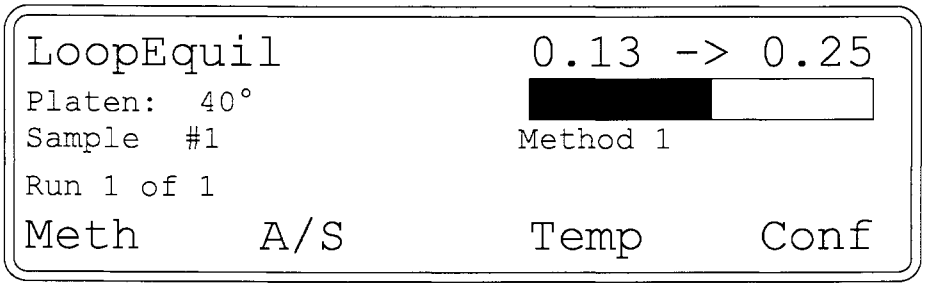


Figure 11-10 Screen and Flow in Loop Fill Mode

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Loop Equilibrate Time Mode

After the loop fills with sample, the vent and pressure valves close. The sample vapor allows the flow in the loop to stabilize. The following screen appears during **Loop Equilibrate Time** mode:



Minimum and maximum programmable values are 0-60 minutes.

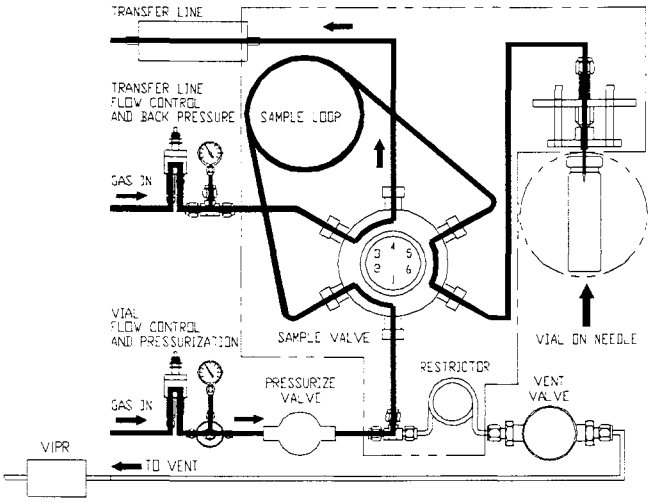
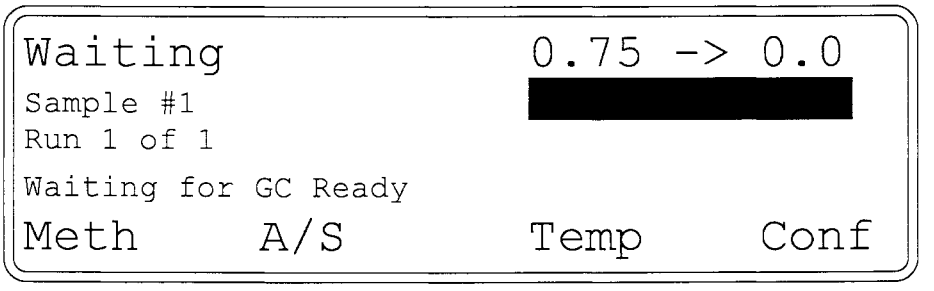


Figure 11-11 Screen and Flow in Loop Equilibrate Time Mode

At this stage in the sequence, if a conflict in operation occurs (e.g. the GC is not ready) and you have selected the "Constant Heat Time Mandatory: No" option, the sample will remain in the vial to minimize sample degradation. The following screen will appear:



When the 7000 receives the GC Ready signal, it will advance to Inject.

continued

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If you have selected the "Constant Heat Time Mandatory: Yes" option (Section 8), the following screen will appear to alert you that the GC is not ready for inject:

```
Error: Constant Heat Time Error


                                Mute
```

If you press **F4 (Mute)** to silence the alarm. The following screen will appear:

```
Error: Constant Heat Time Conflict
Will complete vials in Platen,
then halt processing
Refer to Manual for
more information.
                                Exit
```

Note: If the GC is not ready for injection, the 7000 will abort the run to avoid wasting vials.

If the GC is not ready, the problem could be:

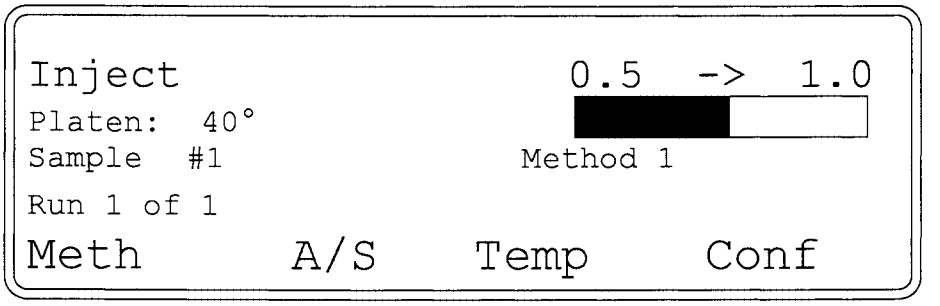
- With the GC,
- With the cycle time of the GC not being synchronized with the 7000/GC run time.

Should you encounter the above error screen, refer to Section 13 for more information and action steps.

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Inject Mode

When the 7000 gets the GC ready signal, the 6-port valve rotates, placing the sample loop contents in line with the column carrier gas. Both the GC and the data system are started automatically by the 7000. Carrier gas backflushes the loop, sweeping the sample through the heated transfer line and injecting it into the GC.



Upon injection, the sample loop should be swept with a volume of carrier gas at least two times the volume of the loop. To calculate the injection time needed to flush the loop with the appropriate volume of carrier gas, refer to the following calculations:

$$\frac{\text{Loop Size (ml) x 2}}{\text{Column Carrier Flow Rate ml/min}} = \text{Inject Time}$$

$$\textit{Example:}\ \frac{\text{2 ml x 2}}{\text{8 ml/min}} = \frac{4}{8} = \text{0.5 min. Inject Time}$$

(Minimum and maximum programmable inject times are 0-60 min).

Note: An Inject time of zero yields a Vial Atmosphere Purge.

Vial Atmosphere Purge

In Vial Atmosphere Purge, the 7000 vents (displaces) the headspace out of the vial with inert pressurization gas. This is especially helpful when you are running samples that show reactivity when exposed to air during equilibration. You can program Vial Atmosphere Purge when setting method parameters by assigning an Inject time of zero (0) to the first run and allowing the second run to proceed according to normal parameters while using Method Scheduling.

continued

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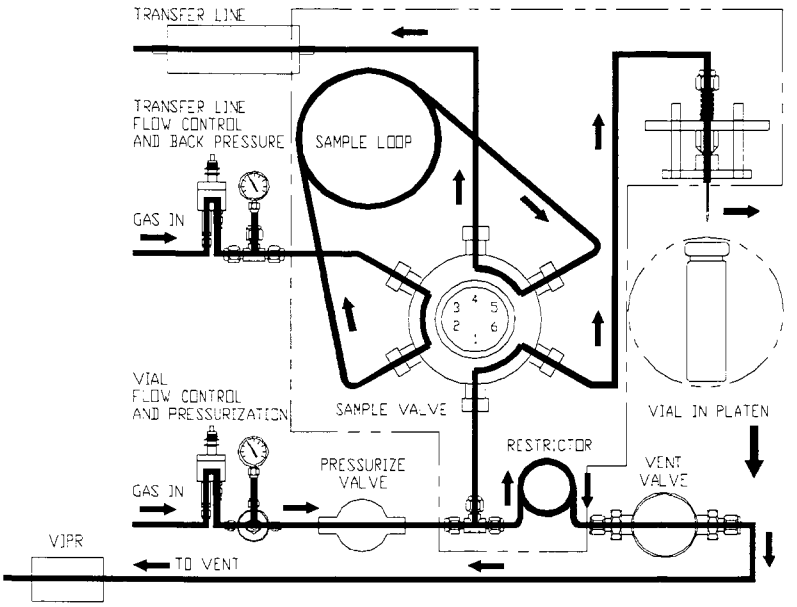


Figure 11-12 Flow in Inject Mode

Completion of the Operation Sequence

Once injection is complete, the six-port valve switches back to its Standby Mode position. The sample vial comes down off the needle and the pressurize and vent valves activate for about 15 seconds to sweep the vent valve path and sample needle lines. This minimizes the potential of sample-to-sample contamination and carryover from the vent exhaust line.

Once the injection is complete, the vial returns to its original position in the Carrousel deck (if the Model 7050 is installed) which completes the 7000 operation sequence.

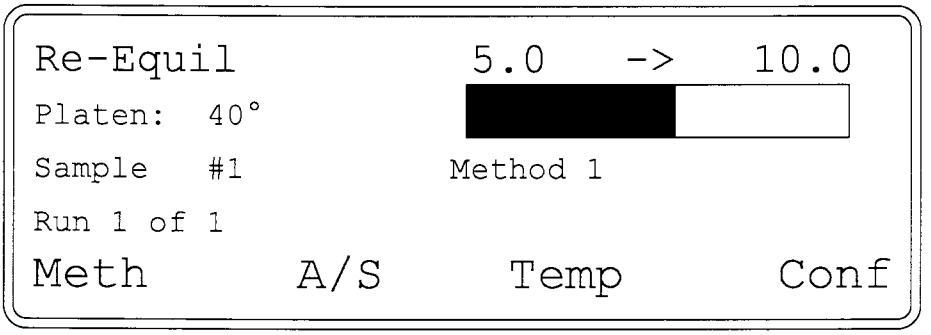
Re-Equil Mode

If you are running multiple injections*, the same vial will be sampled the number of times indicated. In **Re-Equil Mode**, the sample is given time to re-distribute the headspace after each injection. Since the vial has already been in the heated zone (platen), Re-Equil time usually does not need to be more than 25% of the Sample Equilibrate time. The following screen and valve configurations are activated in Re-Equil mode.

*See Section 8 for more information on Multiple Headspace Extraction.

continued

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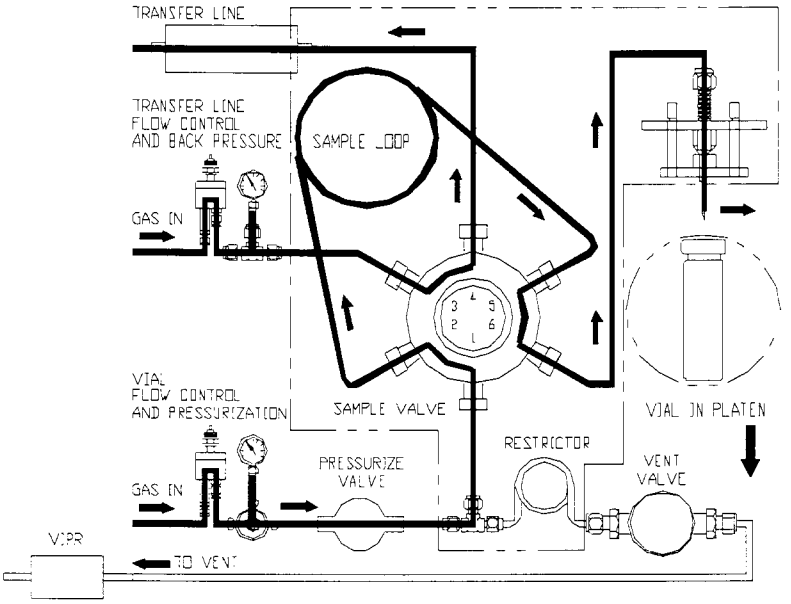
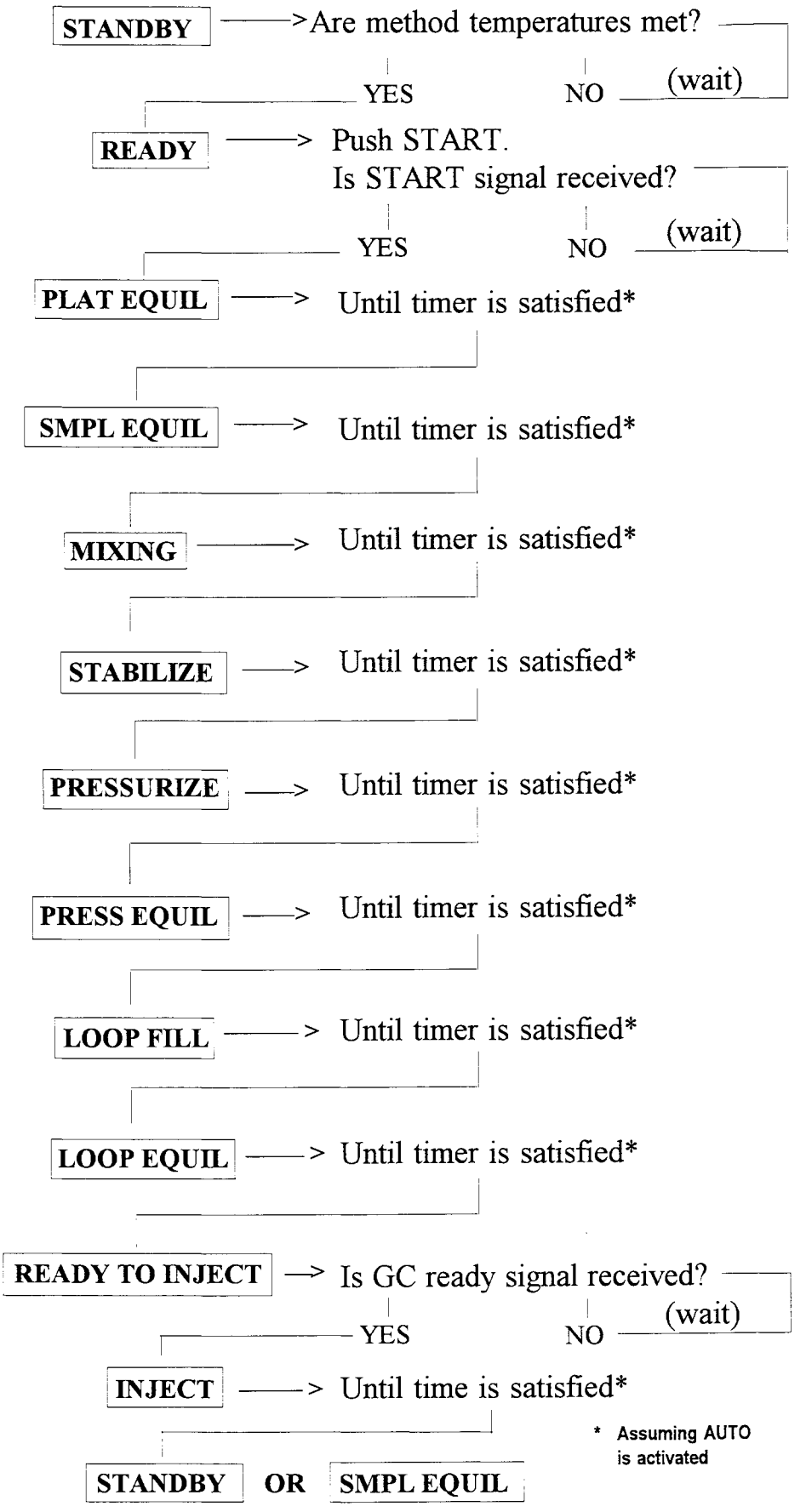


Figure 11-13 Screen and Flow in Re-Equil Mode

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11.3 Sequence of Operation Flowchart



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11.4 Valve Output Chart

Operation Mode/Step	*6-Port Valve*	*Pressurize Valve*	*Vent Valve*
Standby/Ready			
Smpl Equil			
Mixing			
Stabilize			
Raising vial to needle		*	
Pressurize			
Press Equil		*	
Loop Fill		*	*
Loop Equil		*	
Inject	*		
Inject+	*		*
Re-Equil			

+As soon as the sample elevator reaches the down sensor
*Valve is activated

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12.1 Overview of the Section

This section of the manual covers these topics:

- Steps Before Running a Method
- Running Self Tests
- Aborting Runs/Priority Sampling
- Running an Unscheduled Automated Method

The Tekmar™ 7000/7050 system can be operated from the keypad on the front of the unit or through TekLink™, a Windows® based software program that allows you to control the 7000 operations by remote PC.

If you have TekLink, refer to your TekLink User's Manual for information on installing TekLink, configuring your 7000 unit(s) and operating your 7000 with TekLink. To order TekLink or to receive a demonstration disk, please call Tekmar Sales Support at (800) 543-4461 or (513) 247-7000 locally and outside the U.S.

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12.2 Steps Before Running a Method

Note: Do not begin a run until you have determined the appropriate method parameters for your particular samples.

The default method values may not give you the optimum results available with the 7000. To use the instrument to its highest capabilities, refer to Section 9 for more background on developing methods before beginning a sample run.

Four methods can be stored in the 7000's memory. Default values for the methods are automatically loaded. When powered up, the 7000 will hold (Standby Mode) in Method 1.

Before you begin:
You should complete the following steps before beginning a run. These preparatory steps are briefly reviewed in this section. For detailed operating instructions, go to the section in the manual noted.

1) Power Up
2) Change the Viewing Angle
3) Leak Check
4) Set Pneumatics
5) Set the Clock
6) Program the 7000 to Acknowledge Accessories*
7) Enable Method Scheduling**
8) Set Autosampler Start and Stop Positions
9) Verify the System is Set for Constant Heat Time**
10) Prepare the 7000 for Vials
11) Load Vials into the 7000 or 7050

* Optional
** Available with 7050 only

continued

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- ❑ **Power Up**
  If this is an initial power up (or a power up after a power failure resulting in memory loss), the "Parameters Invalid" screen will appear. Press **(Run)** or **(Edit)** to cue the system to load the default values into Random Access Memory (RAM). Press **F4 (Exit).**

- ❑ **Changing the Viewing Angle**
  To adjust the screen angle for your comfort, refer to Section 14.2

- ❑ **Leak Check**
  To do a quick check for a leak tight system, refer to Section 4.7.

- ❑ **Set Pneumatics (flows, pressures)**
  For proper pneumatic settings, refer to Section 5.

- ❑ **Set the Clock**
  Refer to Section 14.3.

- ❑ **Acknowledge Accessories**
  To enable the 7000 to acknowledge the 7050 Carrousel and/or the Cryofocusing Module, refer to Section 14.

- ❑ **Enable or Disable Method Scheduling**
  Refer to Section 10.

- ❑ **Verify Start and Stop Positions in the 7050**
  Refer to Section 10.

- ❑ **Verify the System is Set for Constant Heat Time**
  Refer to Section 8.

- ❑ **Choose Vial Sizes**
  Before operating the 7000 by itself or with the 7050, you need to determine the vial sizes you are going to use and how to prepare the unit for those sizes. Refer to Section 6.

- ❑ **Load Vials in the 7000 or the 7050**
  Refer to Section 6.

continued

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OPTIONAL STEPS:

- ❑ **Configure MOM™ for Optimum Parameter Settings**
  Use Method Optimization Mode™ (MOM) to determine your optimum parameter settings. Refer to Section 8.

- ❑ **Develop Methods**
  Once you've calculated the optimum parameter settings, you will need to use these settings to develop your methods. Section 8 gives you the step-by-step instructions to enter the optimum parameter settings. Section 9 explains some of the theory and applications involved in method development.

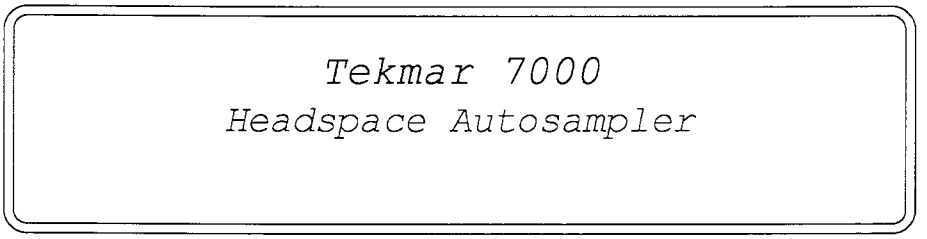
- ❑ **Schedule Methods**
  Use the Method Scheduling mode to assign groups of samples to the methods you have developed. Refer to Section 10.

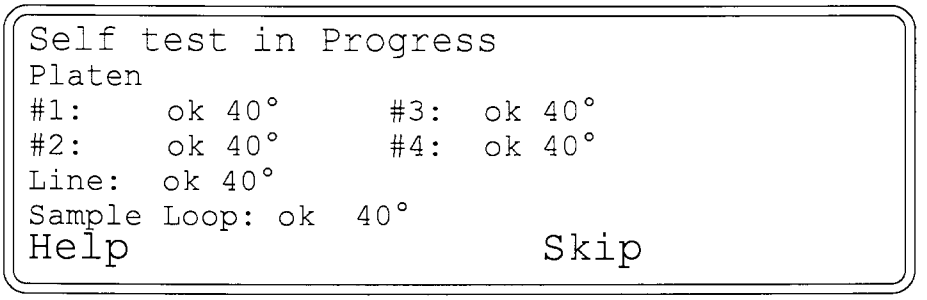
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12.3 Running Self Tests

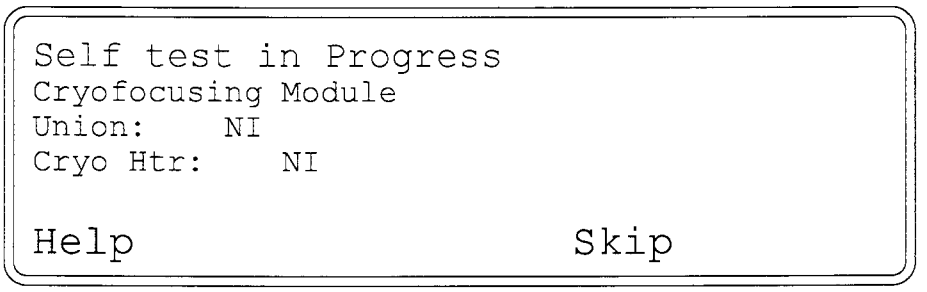
When you turn the 7000 on, the system conducts self tests to confirm that all of its heated components are working properly.

To conduct self tests, the 7000 briefly turns on each heater in succession. When the thermocouple for a particular heater registers a temperature increase (so the 7000 knows that heater is working), the system advances to the next heater.



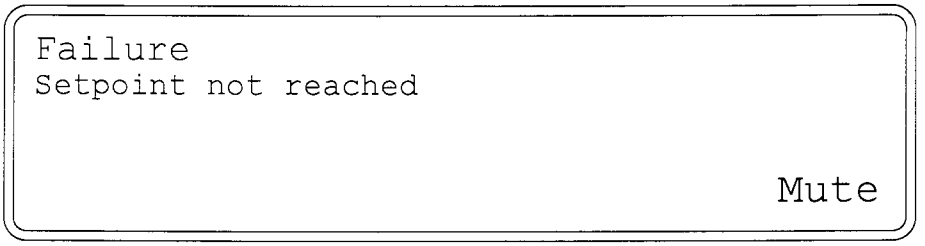


Note: "OK" indicates that the particular heater is operating properly -- it hasn't fallen short of or gone beyond its setpoint.



Note: "NI" indicates that the particular accessory listed is not installed.

An error screen will appear if there are heater faults (if the heater fails to register an increase in temperature).



continued

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Skipping Self Tests

You can skip self tests any time by pressing **F4 (Skip)**. The 7000 will advance to the next heater test. The 7000 treats a skipped self test as a failure. Here is what you will need to do:

1. Press **F3 (Ignore)** to acknowledge that the test was intentionally skipped.

2. If you get an error message on a heater that was not skipped, press **F4 (Retest)** to rerun the self tests on that set of heaters.

```
Failure
Platen
#1:    skipped     #3:  skipped
#2:    skipped     #4:  skipped
Line:  skipped
Sample Loop: skipped
Help              Ignore     Retest
```

If a heater continues to fail the self test, please contact the Tekmar Service Department for assistance at (800) 874-2004 or (513) 247-7000 locally or outside the U.S.

When the self tests are complete, the system goes to the Current Configuration screen:

```
Current Configuration          v. x.xx
Date:  1/05/99      Time:  21: 36: 47
A/S:   YES           Cryo:  NO    Aux: NO

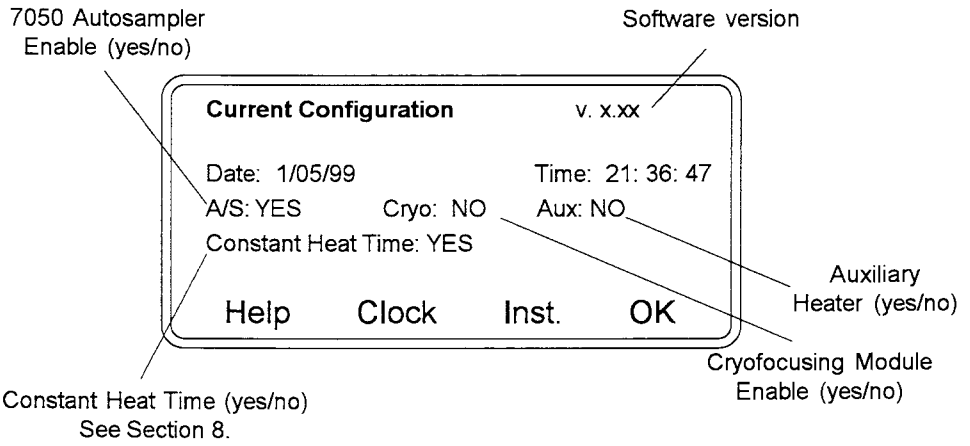
Constant Heat Time: Yes

Help     Clock     Inst.     OK
```

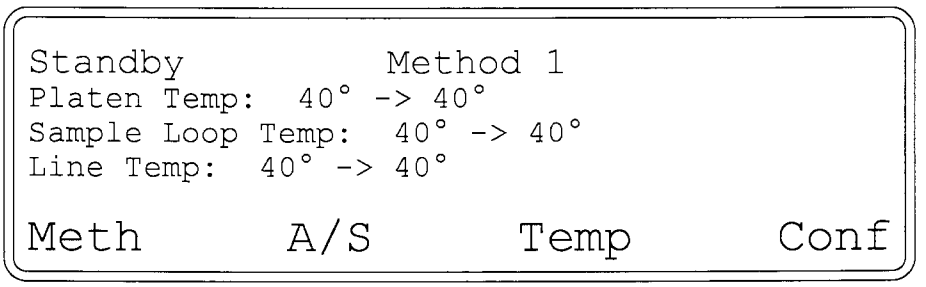
continued

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Following is an explanation of the Current Configuration screen:



1. From the Current Configuration screen, press OK to advance to the Standby screen:



From the Standby screen, you can activate the following functions:

- F1 (Meth) - to edit or run 1 of 4 methods.
- F2 (A/S) - to set up start and stop positions in the 7050 autosampler and to schedule methods if desired.
- F3 (Temp) - displays the setpoint and actual temperatures of the platen, sample loop, and line.
- F4 (Config) - displays the current configuration of your system, including:
  - version of software
  - date and time stored in microprocessor
  - configuration of 7050 carrousel, cryofocusing module and auxiliary heater accessories.
- GC cycle time

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12.4 Aborting Runs/ Priority Sampling

See Section 10.2 for information on aborting runs and running a priority sample.

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12.5 Running an Unscheduled Automated Method

The 7000 system has four default methods that are based upon typical samples. To run a sample using the default values (already configured into the system), start with Method 1. The introductory screen appears followed by the self test screens. Once you've viewed (or skipped) all the self test screens, the Standby screen appears and remains until the 7000 meets parameter setpoints:

```
Standby                          Method 1
Platen Temp:  40° -> 40°
Valve Temp:   40° -> 40°
Line Temp:    40° -> 40°

Meth        A/S        Temp        Conf
```

When the 7000 has met the parameter values for Method 1, it pauses until you press **STEP** on the keypad. The following screen will appear. (If the 7000 is in AUTO mode, it will automatically advance to this screen):

```
Ready                            Method 1
Platen:  40°
Ready for samples to be loaded
Press START to begin

Meth        A/S        Temp        Conf
```

1. Press **F2 (A/S)** to confirm the desired start and stop positions for your automated run:

```
Automatic Sampler Control
Current position: 1        Platen: 1
Start:  1          Stop:  10
Enable method schedule:  Y

Help       Sched       ->       Exit
```

continued

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2. Press **F4 (Exit)**. The following screen will appear:

```
Ready                          Method 1
Platen:   40°
Ready for samples to be loaded
Press START to begin

Meth        A/S        Temp       Conf
```

3. Press **F4 (Conf)** to verify that all accessories are configured. The Configuration screen will come up:

```
Current Configuration              v. x.xx

Date:   1/05/99          Time:  21: 36: 47
A/S: YES           Cryo:  YES       Aux: NO
Constant Heat Time Mandatory?:   YES

Lock      Clock      Inst.       OK
```

4. If everything is correct, press **F4 (OK)**.

5. Press **F1 (Meth)** and the desired method number. Press **F2 (Run)** then press **START** on the keypad and the following screen will appear:

```
Are applicable GC times correct?

Method       1:     15          2:     15
             3:     15          4:     15

YES        NO
```

If you select "YES", the 7000 will advance in your selected method. If you select "NO" the system will go back to Standby to allow you to edit the GC times through your method parameter settings.

Note: If you want the system to begin a run without having to press the START key, set BIAS switch 4 up and INPUT switch 3 down on the 7000 I/O Board. These settings yield a constant start signal. When the system is configured in this way, you press AUTO to start the system when it has reached its setpoint. If you press HOLD, the method won't begin.

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13.1 Overview of the Section

This section of the manual describes failure and error messages you may encounter when operating the 7000/7050.

Note: This section does not necessarily show ALL of the screens that may occur during a particular error or failure. For additional help, call the Tekmar Service Department at (800) 874-2004. Outside the U.S., please call (513) 247-7000.

13.2 Error - Vials in Platen

This screen appears when there is a power failure or when powering up after the instrument was turned off during an operating mode:

```
ERROR - Possibly vials in Platen




                                    Mute
```

1. Press **F4 (Mute)** and the Vial Location screen will appear:

```
ERROR - Possibly vials in Platen
Vials must be removed
Vials:  1  2  3  4  5  6  7  8  9  10  11  12

          Auto      Manual      Exit
```

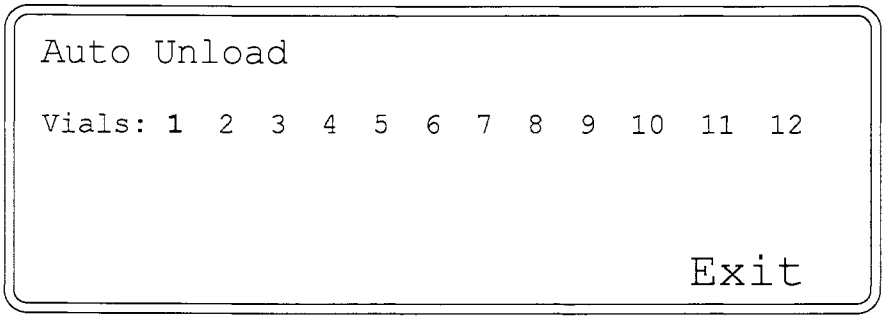
2. When you see this error screen, you have three options:

   - Press **F2 (Auto)** to enable the 7000 to automatically unload the specified chambers in the platen.
   - Press **F3 (Manual)** to unload the platen manually by using the **STEP** and the **LOAD/UNLOAD** keys.
   - Press **F4 (Exit)** to skip this step and return the system to the Standby mode.

3. Press **F2 (Auto)** to bring up this screen:

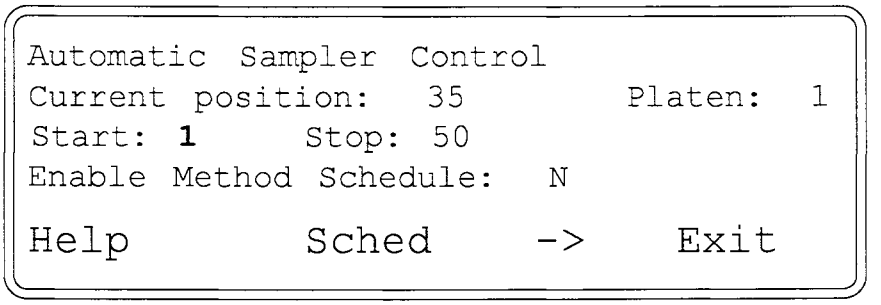
continued

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4. Press **F3 (Manual)** to bring up the Automatic Sampler Control screen which allows you to use the platen parameter to unload the specified platen positions. Don't pay attention to the "start", "stop", and "enable method schedule" prompts on the screen. Just use the "platen" prompt to key in the number for the platen position(s) you want to unload.

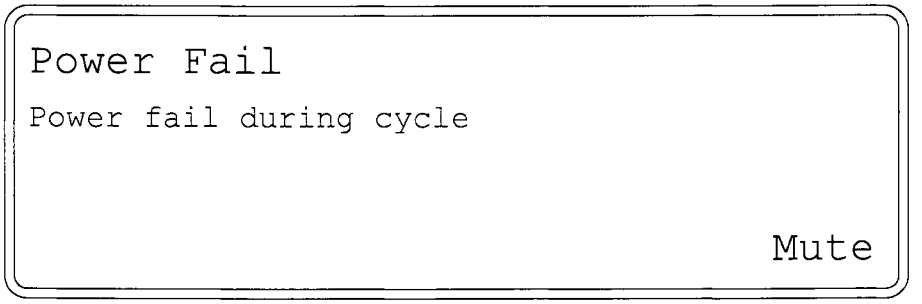
5. Use the **STEP** key to advance the platen to a desired position. You may use the "±" key to go in reverse. Use the **LOAD/ UNLOAD** key to unload the vials from the platen.



6. Press **F4 (Exit)** to skip the unload step and return the 7000 to the Standby mode.

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13.3 Power Fail During Cycle - Kept Memory



This screen lets you know that a power failure has occurred, but that the 7000's battery back-up system has saved the current method parameters. Press **F4 (Mute)** to enable the unit to proceed with the Self Test mode.

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13.4 Power Fail - Memory Lost-Reset Clock

```
POWER FAIL
Memory lost - Reset Clock


                              Mute
```

Note: The 7000 has a memory-protect function which will save parameters for up to 90 days after a memory loss occurs.

During this memory fail, all parameters revert to default values. You will need to re-enter all values to modified methods, including date and time.

1. If there were vials in the platen at the time of the power failure, the following screen would appear:

```
ERROR - Possibly vials in Platen


                              Mute
```

2. Check for vials in the platen. To do this press **F4 (Mute)** and the following screen will appear:

```
ERROR - Possibly vials in Platen
Vials must be removed
Specific vials unknown
all positions in question

       Auto      Manual      Exit
```

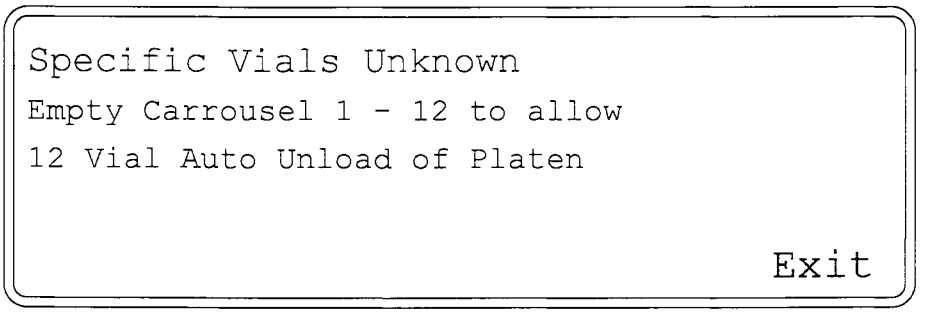
3. You have some options when you see this error screen:

   - Press **F2 (Auto)** to enable the 7000 to automatically unload all 12 platen chambers.
   - Press **F3 (Manual)** to unload the platen manually by using the **STEP** and the **LOAD/UNLOAD** keys.

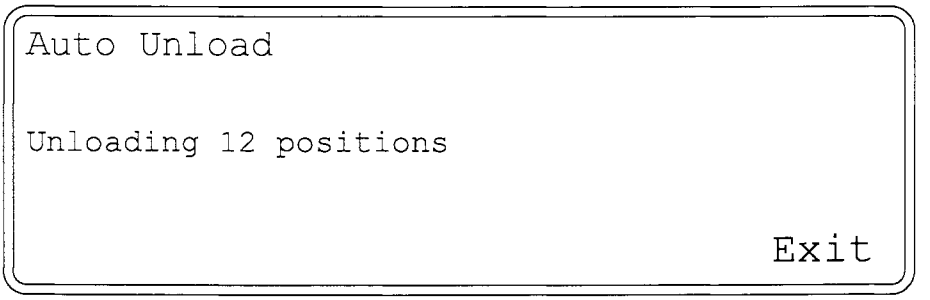
continued

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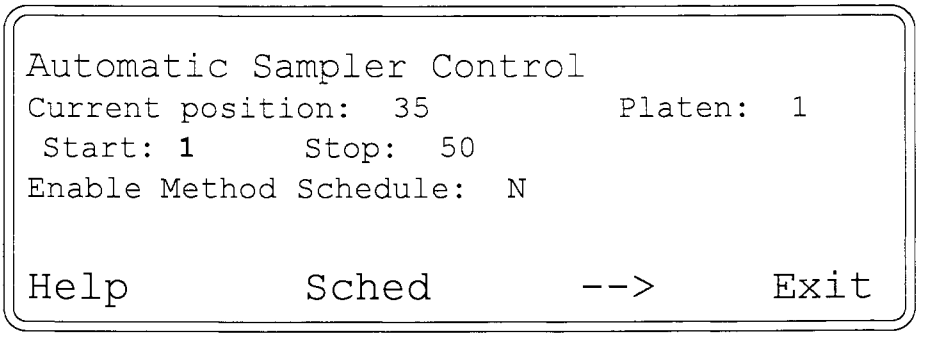
4. Press **F2 (Auto)** to bring up this screen:



5. Press **F4 (Exit)** and the 7000 will automatically unload the 12 platen positions. The following screen will come up:



6. Press **F3 (Manual)** to bring up the Automatic Sampler Control screen. This allows you to choose the platen positions to unload. Push **STEP** to advance to a desired position or "±" to go in reverse. Press **LOAD/UNLOAD** to unload the vials from the platen.



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13.5 Error - GC Cycle Time Conflict

If you have selected the **Constant Heat Time Mandatory: Yes** option (Section 8.2), the 7000 will not ignore the GC cycle time error and will sound an alarm. The following screen will appear to alert you that the GC is not ready for an injection:

```
ERROR:
Constant Heat Time Error

                              Mute
```

Press **F4 (Mute)**. One of the following screens will appear:

```
ERROR: Constant Heat Time Error
Will complete vials in Platen,
then halt processing.
Refer to Manual for
more information.
                              Exit
```

```
ERROR: Constant Heat Time Error
(Vial #s)
1 to 3 results in doubt.
Remaining vials need to be
reprogrammed and restrained.
Refer to Manual for
more information.
                              Exit
```

If the GC is not ready it may be due to:

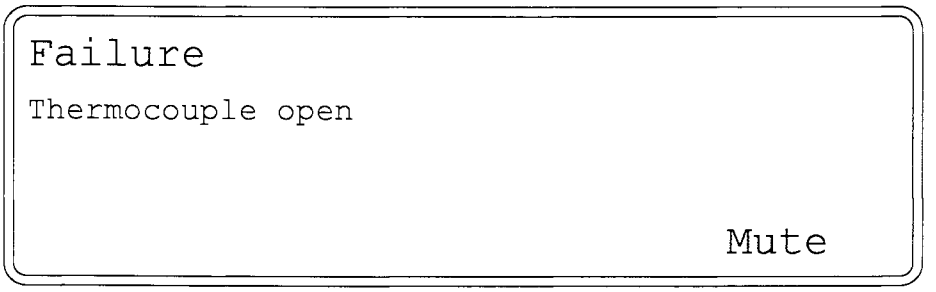
- A failure or problem with the GC.
- The cycle time of your GC is not synchronized with the 7000/GC cycle time.

If you encounter this error screen, you will need to refer to Section 8.4 for more information and action steps.

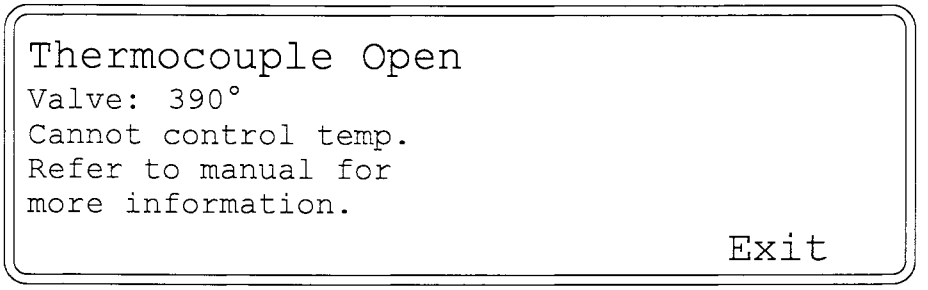
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13.6 Failure - Thermocouple Open

This screen appears when a valve, platen, or line heater sensor is not responding:



Press **F4 (Mute)** and the following screen appears to show you the type of heater in question:



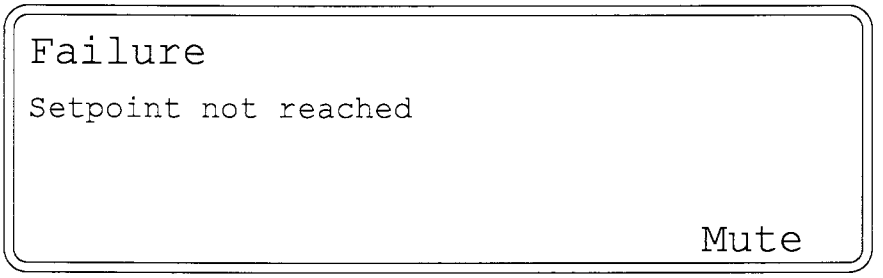
This is a warning screen. You will need to take action to solve the problem. The unit will automatically cut off power to the heaters in the base unit so that there is no damage to the unit. Call the Tekmar Service Department at (800) 874-2004 or (513) 247-7000 locally and outside the U.S. before proceeding.

Once the problem is solved, you can press **F4 (Exit)** to return to the current mode of operation.

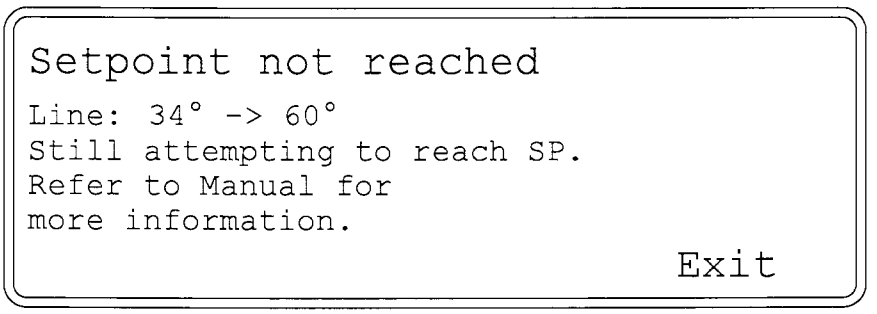
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13.7 Failure - Setpoint Not Reached

This screen appears when a valve, platen, or line heater has not heated to its assigned setpoint:



Press **F4 (Mute)** and the following screen appears designating the type of heater in question:



Note: This failure message could indicate that the thermocouple is not responding properly (see the previous page).

This screen will appear if a heater setpoint has not been reached. This is a warning screen. You will need to take action to solve the problem. If you press **F4 (Exit),** you will override the setpoint and the run will begin. If this screen appeared even after the 7000 ran its Self Tests, please call the Tekmar Service Department at (800) 874-2004 or (513) 247-7000 locally and outside the U.S. before proceeding. Once the problem is solved, you can press **F4 (Exit)** to return to the current mode of operation.

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13.8 Fatal Failure - Platen Motor

```
Fatal Failure

Motor:        Platen
Position:   L1  M4  S7
Positioning Failure

                                    Mute
```

This screen appears when the optical encoder board finds that the platen is not positioned properly. This is a warning screen and action should be taken to alleviate the problem. The unit will automatically cut off power to the platen motor to prevent damage to the unit.

1. Press **F4 (Mute)** to silence the alarm.

Although the 7000 indicates "fatal failure", the microprocessor has a current draw sensing function on all mechanical systems. When a motor drive system encounters resistance (a jam), it will draw more current to overcome the resistance. Each motor drive assembly has a not-to-exceed current draw to protect motors from burning up and gears from stripping.

2. Press **F4 (Exit)** to enable the 7000 to correct itself. If, after trying this step once or twice the problem is not corrected, call the Tekmar Service Department at (800) 874-2004 or (513) 247-7000 locally and outside the U.S.

```
Fatal Failure
Motor: Platen
Position:   L1  M4  S7
Positioning Failure
Refer to Manual

          Sensors                   Exit
```

3. Press **F2 (Sensors)** to open the Sensor screen for troubleshooting.

```
   Smp - Vial - - Index - Mixer -
E  U   D E  L M   D R  H  F 9   1  2   D
r  p   o j  o a   o e  o  o     2  2   o
r      w e  a n   w v  m  r m   m  m   w
o      n c  d L   n    e  w l   l  l   n
r        t    d             d

Car 1  Plat L10  M1   S4☼          Exit
```

continued

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Note: After a platen failure, if the asterisk (*) after the S (S4*) is not shown on the sensor screen, you will need to manually position the platen by disengaging the platen drive motor, and turning the platen by hand until the * appears on the screen.

⚠ WARNING



Electrical shock hazard inside this instrument.
Unplug power cord before servicing.

⚠ WARNING



Hot surfaces inside. Use caution when servicing the instrument.

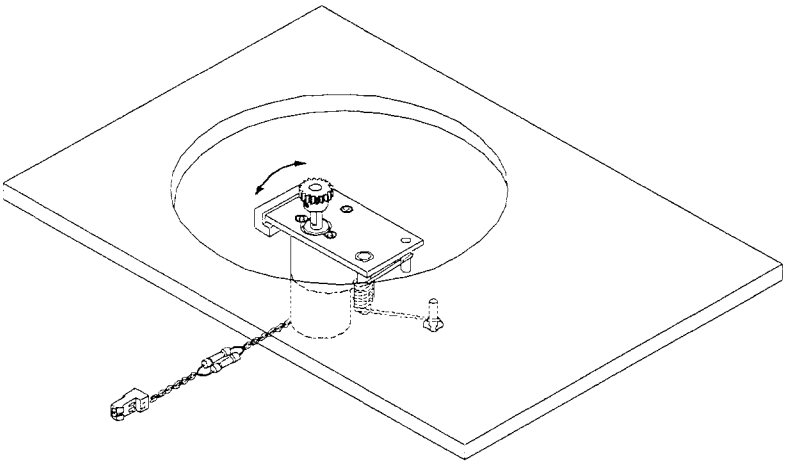


Figure 13-1 Platen Drive Motor Location

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13.9 Fatal Failure - Carrousel Motor

This screen appears when the encoder board finds that the 7050 carrousel is not positioned correctly. This is a warning screen. You will need to take action to solve the problem. The unit will automatically cut off power to the motor in question to prevent instrument damage.

```
Fatal Failure
Motor: Carrousel
Position:   25
Positioning Failure


                                    Mute
```

1. Press **F4 (Mute)** to silence the alarm. The following screen will appear:

```
Fatal Failure
Motor: Carrousel
Position:   25
Positioning Failure
Refer to Manual

          Sensors                   Exit
```

2. Press **F4 (Exit)** to enable the 7000 to correct itself. If, after trying this step once or twice the problem is not corrected, call the Tekmar Service Department at (800) 874-2004 or (513) 247-7000 before proceeding. Pressing **F2 (Sensors)** opens the Sensor screen for troubleshooting.

```
   Smp - Vial - - Index - Mixer -
E U   D E  L M   D R  H  F 9   1  2   D
r p   o j  o a   o e  o  o     2  2   o
r     w e  a n   w v  m  r m   m  m   w
o     n c  d L   n    e  w l   l  l   n
r       t    d             d

Car 1  Plat L10  M1   S4✡             Exit
```


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13.10 Fatal Failure - Vial Loader

This screen appears when there is a positioning conflict and the motors are restricted. This is a warning screen. You will need to take action to solve the problem. The unit will automatically cut off power to the motor in question to prevent instrument damage.

```
Fatal Failure
Motor: Vial Loader
Carrousel not properly positioned

                                  Mute
```

1. Press **F4 (Mute)** to silence the alarm. The following screen will appear:

```
Fatal Failure
Motor: Vial Loader
Carrousel not properly positioned
Refer to Manual

              Sensors              Exit
```

2. Press **F4 (Exit)** to enable the 7000 to correct itself. If, after trying this step once or twice the problem is not corrected, call the Tekmar Service Department at (800) 874-2004 or (513) 247-7000 locally and outside the U.S. before proceeding. Pressing **F2 (Sensors)** opens the Sensor screen for troubleshooting.

```
   Smp - Vial - - Index - Mixer -
E U   D E  L M  D R  H  F 9   1  2   D
r p   o j  o a  o e  o  o     2  2   o
r     w e  a n  w v  m  r m   m  m   w
o     n c  d L  n    e  w l   l  l   n
r       t    d          d

Car 1  Plat L10  M1   S4✡              Exit
```


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13.11 Fatal Failure - Sample Loader

This screen appears when the encoder board detects an error in the positioning of the sample loader. This is a warning screen. You will need to take action to solve the problem. The unit will automatically cut off power to the motor in question to prevent instrument damage.

```
Fatal Failure
Motor:  Sample Loader
Sensor:  Up (or Down)
Sensor didn't close
                                  Mute
```

1. Press **F4 (Mute)** to silence the alarm.

```
Fatal Failure
Motor:  Sample Loader
Sensor:  Up (or Down)
Sensor didn't close
Refer to Manual
          Sensors                 Exit
```

2. Press **F4 (Exit)** to enable the 7000 to correct itself. If, after trying this step once or twice the problem is not corrected, call the Tekmar Service Department at (800) 874-2004 or (513) 247-7000 locally and outside the U.S. before proceeding. Pressing **F2 (Sensors)** opens the Sensor screen for troubleshooting.

```
   Smp - Vial - - Index - Mixer -
E  U   D E   L M   D R   H   F 9   1  2   D
r  p   o j   o a   o e   o   o     2  2   o
r      w e   a n   w v   m   r m   m  m   w
o      n c   d L   n     e   w l   l  l   n
r        t     d               d
Car 1  Plat L10  M1   S4✡              Exit
```


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13.12 Fatal Failure - Vial Loader

This screen appears when the encoder board detects incorrect positioning of the vial loader. This is a warning screen. You will need to take action to solve the problem. The unit will automatically cut off power to the motor in question to prevent instrument damage.

```
Fatal Failure
Motor:  Vial Loader
Sensor: Eject (or Load or ManLd or Down)
Sensor didn't close
                                    Mute
```

1. Press **F4 (Mute)** to silence the alarm. One of the following screens will appear:

```
Fatal Failure
Motor:  Vial Loader
Sensor: Eject (or Load or ManLd or Down)
Sensor didn't close
Refer to Manual
          Sensors                   Exit
```

```
Fatal Failure
Motor:  Vial Loader
Platen not properly positioned
Refer to Manual
          Sensors                   Exit
```

2. Press **F4 (Exit)** to enable the 7000 to correct itself. If, after trying this step once or twice the problem is not corrected, call the Tekmar Service Department at (800) 874-2004 or (513) 247-7000 locally and outside the U.S. before proceeding. Pressing **F2 (Sensors)** opens the Sensor screen for troubleshooting.

```
   Smp - Vial - - Index - Mixer -
 E U   D E  L M  D R  H  F 9   1  2   D
 r p   o j  o a  o e  o  o     2  2   o
 r     w e  a n  w v  m  r m   m  m   w
 o     n c  d L  n    e  w l   l  l   n
 r       t    d          d
Car 1  Plat L10  M1   S4✡             Exit
```


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13.13 Fatal Failure - Mixer Sensor

This screen appears when the encoder board detects incorrect positioning of the mixer. This is a warning screen. You will need to take action to solve the problem. The unit will automatically cut off power to the motor in question to prevent instrument damage.

```
Fatal Failure
Motor: Mixer
Sensor: Mixer Down (or 9 ml, 12 ml or 22 ml)
Sensor didn't close
                                        Mute
```

1. Press **F4 (Mute)** to silence the alarm. One of the following screens will appear:

```
Fatal Failure
Motor: Mixer
Sensor: Mixer Down (or 9 ml, 12 ml or 22 ml)
Sensor didn't close
Refer to Manual
      Sensors                           Exit
```

```
Fatal Failure
Motor: Mixer
Platen
Not properly positioned
Refer to Manual
      Sensors                           Exit
```

2. Press **F4 (Exit)** to enable the 7000 to correct itself. If, after trying this step once or twice the problem is not corrected, call the Tekmar Service Department at (800) 874-2004 or (513) 247-7000 locally and outside the U.S. before proceeding. Pressing **F2 (Sensors)** opens the Sensor screen for troubleshooting.

```
   Smp - Vial - - Index - Mixer -
E U   D E   L M   D R   H   F 9    1   2    D
r p   o j   o a   o e   o   o      2   2    o
r     w e   a n   w v   m   r m    m   m    w
o     n c   d L   n     e   w l    l   l    n
r       t     d               d

Car 1  Plat L10  M1   S4✡                Exit
```


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13.14 Fatal Failure - Carrousel Index

This screen appears when the encoder board detects incorrect positioning (the carrousel was not positioned correctly). This is a warning screen and action should be taken at this point to alleviate the problem. The unit will automatically cut off power to the motor in question to prevent damage to the unit.

```
Fatal Failure
Motor:       Carrousel Index
Sensor: Home (or Rev or Forwd)
Sensor didn't close

                                    Mute
```

1. Press **F4 (Mute)** to silence the alarm.

```
Fatal Failure
Motor:       Carrousel Index
Sensor: Home (or Rev or Forwd)
Sensor didn't close
Refer to Manual

      Sensors                       Exit
```

2. Press **F4 (Exit)** to enable the 7000 to correct itself. If, after trying this step once or twice the problem is not corrected, call our Service Department at (800) 874-2004 or (513) 247-7000 locally and outside the U.S. before proceeding. Pressing **F2 (Sensors)** opens the Sensor screen for troubleshooting.

```
   Smp - Vial - - Index - Mixer -
 E U   D E  L M   D R  H  F 9   1  2   D
 r p   o j  o a   o e  o  o     2  2   o
 r     w e  a n   w v  m  r m   m  m   w
 o     n c  d L   n    e  w l   l  l   n
 r       t    d          d

Car 1  Plat L10  M1   S4✡              Exit
```


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13.15 Fatal Failure - Platen (or Carrousel) Motor

This screen appears when a motor jams. This is a warning screen. You will need to take action to solve the problem. The unit will automatically cut off power to the motor in question to prevent instrument damage.

```
Fatal Failure
Motor: Platen (or Carrousel)
Motor Failure

                                    Mute
```

1. Press **F4 (Mute)** to silence the alarm.

```
Fatal Failure
Motor:  Platen (or Carrousel)
Motor Failure
Refer to Manual

              Sensors                Exit
```

If you have just set up the 7000 for the first time and this screen appears, it may mean you have forgotten to remove the shipping inserts in the platen. If so, proceed as follows:

If the 7050 is installed, disconnect the 7050 I/O cable and remove the 7050 from the unit. Follow the instructions in Section 4 to remove the shipping inserts.

If the inserts are not the problem or the screen reads **Motor: Carrousel**, press **F4 (Exit)** to allow the 7000 to correct itself. If, after trying this step once or twice the problem is not corrected, call the Tekmar Service Department at (800) 874-2004 or (513) 247-7000 locally and outside the U.S. before proceeding. Pressing **F2 (Sensors)** opens the Sensor screen for troubleshooting. Pressing **F4 (Exit)** again will return you to your current mode of operation.

```
   Smp - Vial - - Index - Mixer -
E  U   D E  L  M  D R  H  F 9   1  2  D
r  p   o j  o  a  o e  o  o     2  2  o
r      w e  a  n  w v  m  r m   m  m  w
o      n c  d  L  n    e  w l   l  l  n
r        t     d           d

Car 1  Plat L10  M1   S4✡              Exit
```


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14.1 Overview of the Section

This section of the manual includes instructions for reviewing and changing instrument and accessory configurations, changing the viewing angle of the screen, reviewing and resetting the clock, locking the keypad, and cleaning the system.

14.2 Reviewing and Changing Instrument and Accessory Configurations

1. To review installed accessories, press **F4 (Conf)** from the Standby screen.

```
Current Configuration                v. x.xx
Date:  1/05/99          Time:  21: 36: 47
A/S: YES        Cryo:  NO          Aux: NO
Constant Heat Time Mandatory: YES

Help     Clock     Inst.     OK
```

2. Press **F3 (Inst)** to view the Instrument Configuration listing:

```
Automatic Sampler:   Y
Cryofocusing:   not installed
Aux Heater: not installed
Constant Heat Time Mandatory: Y
Help     <-      ->        Exit
```

To change an accessory's configuration (turn it on or off):

1. Press **F2 (<-)** or **F3 (->)** to move the highlighted box to the desired accessory.

2. With the desired accessory highlighted, press **Y (digit 7)** or **N (digit 9)** to re-configure the system for that accessory.

Note: If an accessory is not installed or its self test has been skipped, it cannot be turned on (activated).

3. When all accessories are configured as desired, press **F4 (Exit)** to return to the Current Configuration screen.

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14.3 Changing the Viewing Angle of the Screen

You can adjust the viewing angle of the LCD screen for readability. In Standby mode, press **PAGE UP** to increase the angle of the screen and **PAGE DOWN** to decrease the angle. Press the key down firmly for a continuous change in the angle or press and release the key for an incremental change.

Note: You can't adjust the viewing angle while the unit is in a mode where PAGE UP and PAGE DOWN provide other functions (for example, while editing the Method Parameters Listing).

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14.4 Reviewing and Resetting the Clock

The clock controls the date and time acknowledged by the 7000.

1. To view the clock, press **F4 (Conf)** during execution of any program mode. For example, pressing **F4 (Conf)** from the Standby screen will display the Current Configuration screen.

```
Current Configuration                v. x.xx
Date:  1/05/99          Time:  21: 36: 47
A/S: YES        Cryo:  NO          Aux: NO
Constant Heat Time Mandatory: YES

Help      Clock      Inst.      OK
```

2. If the time and date are correct, press **F4 (OK)**. To change them, press **F2 (Clock)**:

```
Set Clock Time & Date
Date (mm/dd/yy) :          01/05/99
Time (24 hr format) :       12:30

          <-            ->       Exit
```

3. Press **F2 (<-)** or **F3 (->)** to select the digit that needs to be changed. Pressing the desired digit on the keypad instantly enters it into system memory.

4. When all values are correct, press **F4 (Exit)**, to return to the Current Configuration screen.

5. From the Current Configuration screen, press **F4 (OK)** for the Standby screen.

Note: If you press an invalid key when attempting to enter new time and date values, the message "-> INVALID DIGIT/KEY <- " appears on the screen and the system beeps. When the message disappears you can try again to enter the new values.

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14.5 Locking the Keypad

The Current Configuration screen includes an option that allows you to assign a password to your system to lock the keypad and prevent an inadvertent change in parameters. To do this, bring up the Current Configuration screen from the Standby screen by pressing **F4 (Conf)**:

```
Current Configuration                VX.XX
Date:      08/18/99 Time:   12:30:00
A/S: YES      Cryo: YES      Aux: NO
Constant Heat Time Mandatory? YES

Lock       Clock        Inst      OK
```

1. From the Current Configuration screen, press **F1 (Lock)** to bring up the next screen:

```
Parameter Lock
F2 (Lock/Unlock) to toggle lock
F3 (Pass) to change password

          Lock     Pass      Exit
```

2. Press **F3 (Pass)** to change the password. The following screen will come up. You will need to enter both the old and new codes. The initial password assigned at the factory is "0" (zero).

```
Enter Password Code

Old Code:

<Press Enter alone to exit>
```

4. Press **ENTER**. The following screen will come up. Enter the new code.

continued

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```
Enter Password Code

New Code:

<Press Enter alone to exit>
```

5. If you press **F2 (Lock)** from the Parameter Lock screen (on previous page) the following screen will come up.

```
Enter Password Code

Code:

<Press Enter alone to exit>
```

6. Enter your new code and press **ENTER**. The keypad will lock.

7. To unlock the keypad, go to the Parameter Lock screen from the Current Configuration Screen. When the system is locked, the Parameter Lock screen will have an "unlock" prompt at the bottom of the screen:

```
Parameter Lock
F2 (Lock/Unlock) to toggle lock
F3 (Pass) to change password

        Unlock      Pass      Exit
```

8. Press **F2 (Unlock)**. The following screen will come up:

```
Enter Password Code

Code:

<Press Enter alone to exit>
```

9. Use the keypad to enter your code. Press **F4** to exit. The system will unlock.

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14.6 Cleaning the 7000

The 7000 platen chambers can be cleaned periodically. To do this:

1. Shut power off.
2. Remove the four screws from the platen cover panel and remove the panel.
3. Remove the front panel of the unit.
4. Use a clean, damp cloth to swab the inside of the platen chambers.
5. Pull back gently on the platen drive motor to gain access to the remainder of the platen chambers. Swab the chambers with the clean, damp cloth.
6. Reinstall the platen cover and front panel.

⚠ WARNING



Hot surface. Allow the platen to cool before cleaning.

⚠ CAUTION

Do not allow water or any other liquid to enter the electronics portion of the 7000.

14.7 Cleaning the 7050

The 7050 may be cleaned periodically to ensure smooth loading and unloading. To do this:

1. Lift the dust cover.
2. Unscrew the thumb nut and remove the carrousel from the deck.
3. Wrap a clean, dry cloth loosely wrap around your index finger.
4. Carefully wipe out each opening in the 50-position carrousel.
5. Wipe the deck with a clean dry cloth and replace the carrousel.

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15.1 Overview of the Section

This section of the manual provides information you may need if you encounter a problem while operating your 7000/7050 system. Topics covered include:

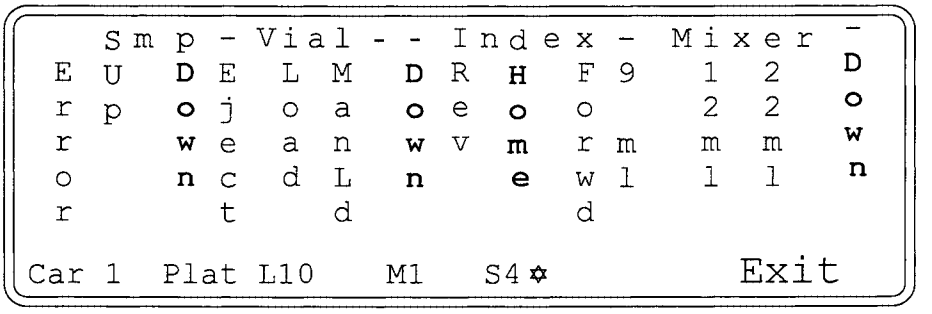
- Viewing the Sensor Screen
- Using the Motor Test Mode Screen
- Verifying Outputs
- Troubleshooting Flowcharts
- Expediting the Troubleshooting Process (forms)

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15.2 Viewing the Sensor Screen

This screen lists the sensors which detect all motor movement and positioning in the 7000. It is used to verify and locate a problem with a motor or position. Refer to the sensor screen when calling the Tekmar Service Department at (800) 874-2004 or (513) 247-7000 to discuss any Fatal Failure or Error screen that comes up.

To access the sensor screen:
Press **1** on the keypad anytime during any mode and the sensor screen will appear. Positions shown on the screen are described below.



Motors and their positions:

Sample Elevator Motor

Smp	Up
	Down

Carrousel Indexing Motor

-Index-	Rev
	Home
	Forwd

Vial Elevator Motor

-Vial-	Eject
	Load
	ManLd
	Down

Mixer Motor

-Mixer-	9 ml
	12 ml
	22 ml
	Down

Highlighted areas on the screen show the current position of the motors.

Platen and 7050 positions are displayed along the bottom of the screen:

Example:

Car	7050 position #1 (position 1 is aligned with the aperture)
Plat	Platen positioning information
L	Platen position #10 is at the Loader Elevator position
M	Platen position #1 is at the Mixer Elevator position
S	Platen position #4 is at the Sample Elevator position
✡	Positioning is correct

If question marks (???) instead of vial locations appear along the bottom of the sensor screen, either the platen or the 7050 is not positioned correctly.

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15.3 Using the Motor Test Mode Screen

This screen lists the motors in the 7000 used for sample processing. It is used to verify if each mechanism is functioning properly. Refer to this screen when calling the Tekmar Service Department at (800) 874-2004 or (513) 247-7000 or use it to perform a routine check of the motors.

Accessing the Motor Test Mode:
Press **2** on the keypad during the Standby mode and the motor test screen will appear. The table below shows the number and name of each of the motors and what the corresponding test will verify.

```
1 - Platen          4 - Carrousel
2 - Smpl Loader     5 - Car Index
3 - Vial Loader     6 - Mixer

          Sensors                    Exit
```

#	*Motor*	*Designates*
1	Platen	verifies rotation and sensors
2	Smpl Loader	verifies movement and sensors
3	Vial Loader	verifies movement and sensors
4	Carrousel	verifies rotation and sensors
5	Car Index	verifies movement and sensors
6	Mixer	verifies movement and sensors

Press **1** through **6** on the keypad to activate the designated mechanism. The 7000 will run through a self test and then return to the Motor Test screen.

Here is an example of how the Motor Test Mode operates:
If you press **6** (Mixer), for example, the mixer begins vibrating and the elevator moves up to the position where a 22 ml vial would sit. It will stay there indefinitely until you press another key. If you press any key, it will go to the 12 ml position. Press another key and the mixer will go to the ml position. Press another key and it will go to the down position, then it will quit and the motor test screen will appear again.

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15.4 Verifying Outputs

This mode allows you to toggle all valve and oven fan outputs on and off to check for proper functioning. Refer to this screen when calling Tekmar's Service Department at (800) 874-2004 or (513) 247-7000 for troubleshooting assistance.

To access the output verification screen:

1. While in Standby mode, press **HOLD**, then press **3** on the keypad. The following screen will appear:

```
1 - Leak Check
2 - Set Flow
3 - Vent Check
4 - Outputs

      Sensors                    Exit
```

2. Press **4** and the Output Verification screen will appear:

```
Outputs
--> Press

(PAGE DOWN & PAGE UP to change)
     (Y/N for On/Off)
                                Exit
```

3. Press **PAGE UP** or **PAGE DOWN** to select the different outputs.

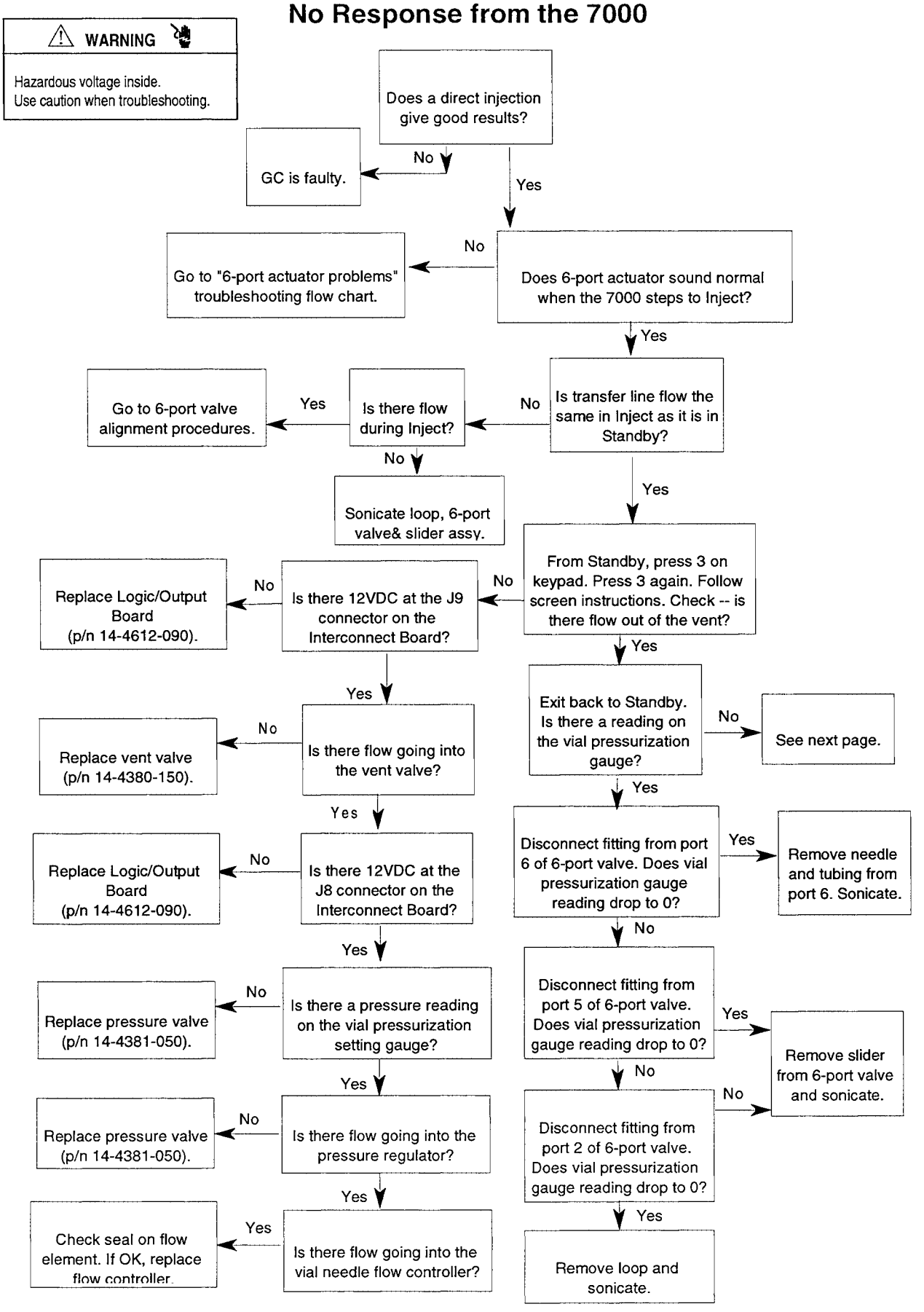
4. Press **Y** and **N** to toggle the selected output on and off. The 6-port valve is the loudest. The others may not be as easy to hear.

15.5 Troubleshooting Flow Charts

The following pages contain flow charts that explain procedures for solving specific operation problems.

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No Response from the 7000



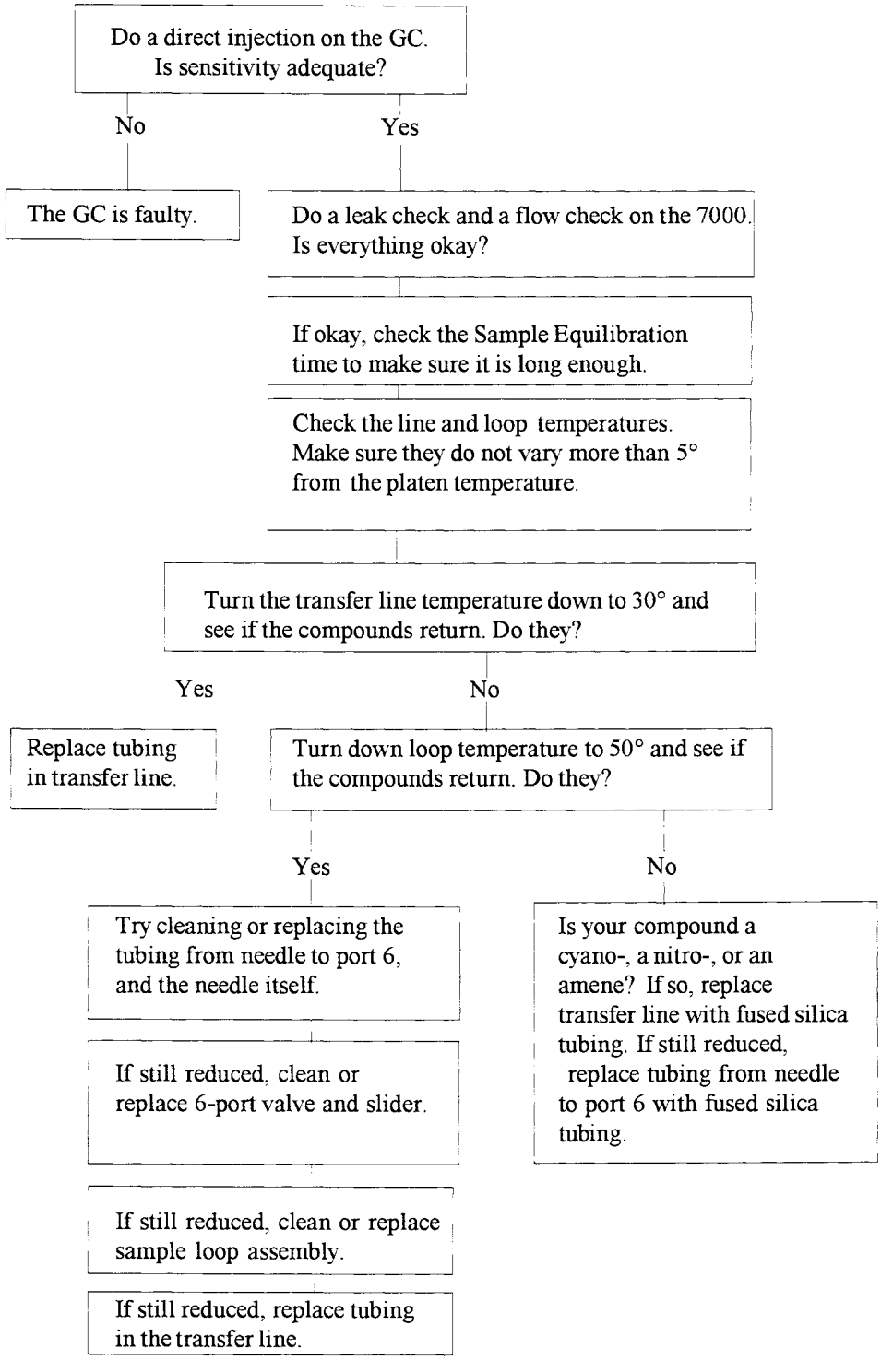
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Reduced Response from the 7000

⚠ WARNING

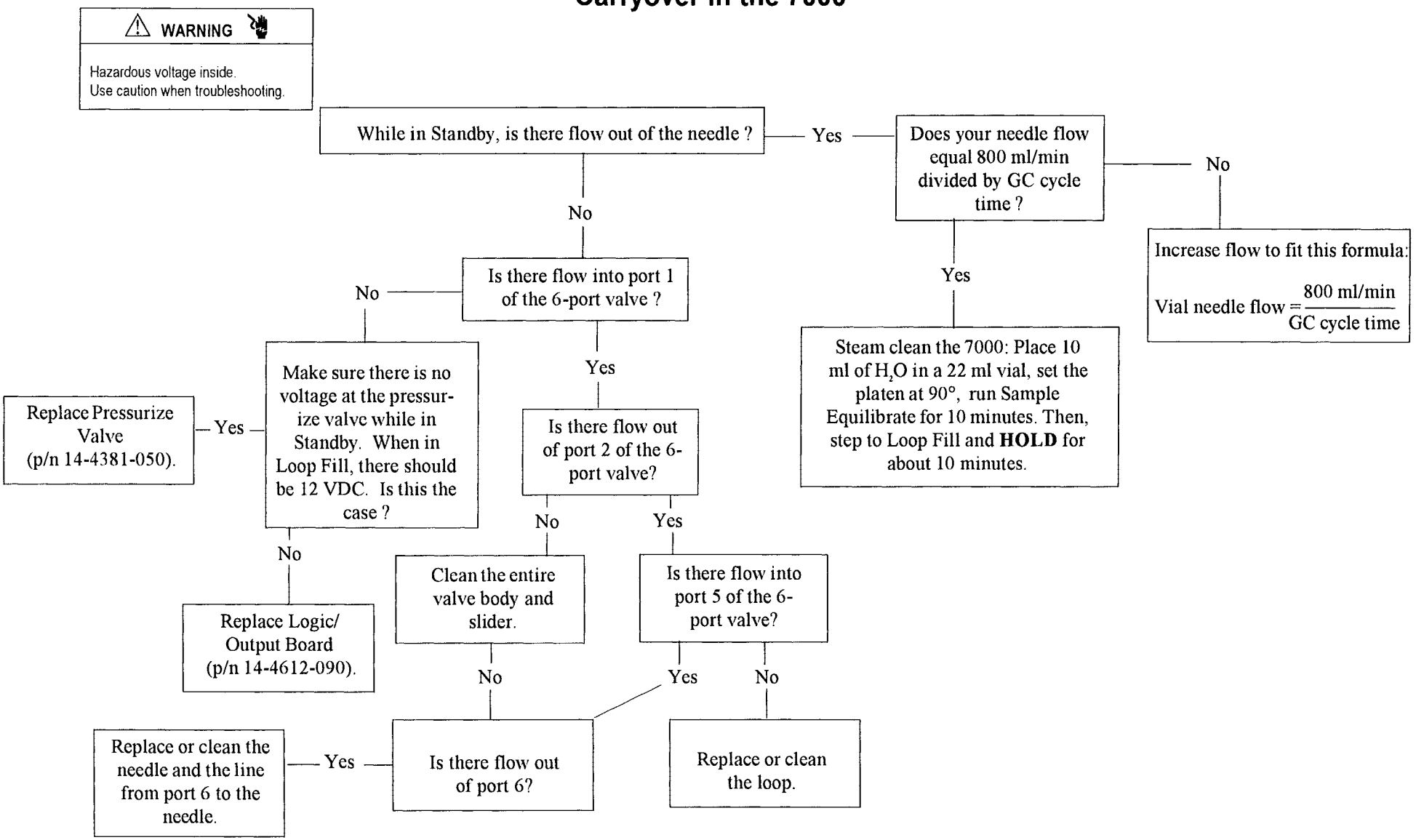
Hazardous voltage inside.
Use caution when troubleshooting.

Note: Reduced response means you were seeing peaks until this point and now the peaks are reduced; it does not refer to initial runs not providing good results.



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Carryover in the 7000



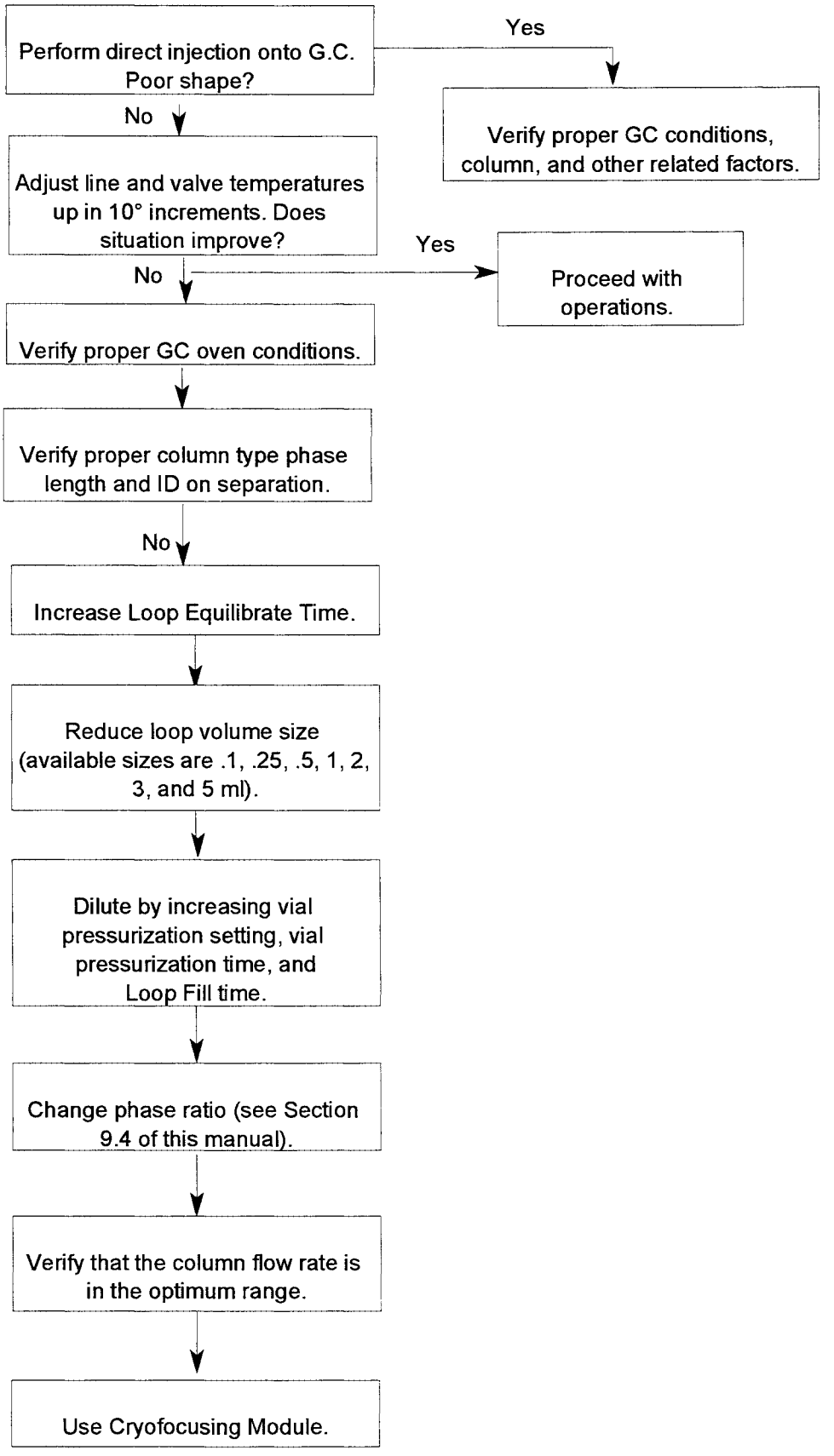
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Poor Peak Shape and/or Tailing

⚠ WARNING

Hazardous voltage inside.
Use caution when troubleshooting.

Note: The Method Optimization Mode™ (MOM) may automate these troubleshooting procedures. See also Section 9 of this manual - **Developing Methods.**



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⚠ WARNING

Hazardous voltage inside.
Use caution when troubleshooting.

No Peaks and/or Sensitivity

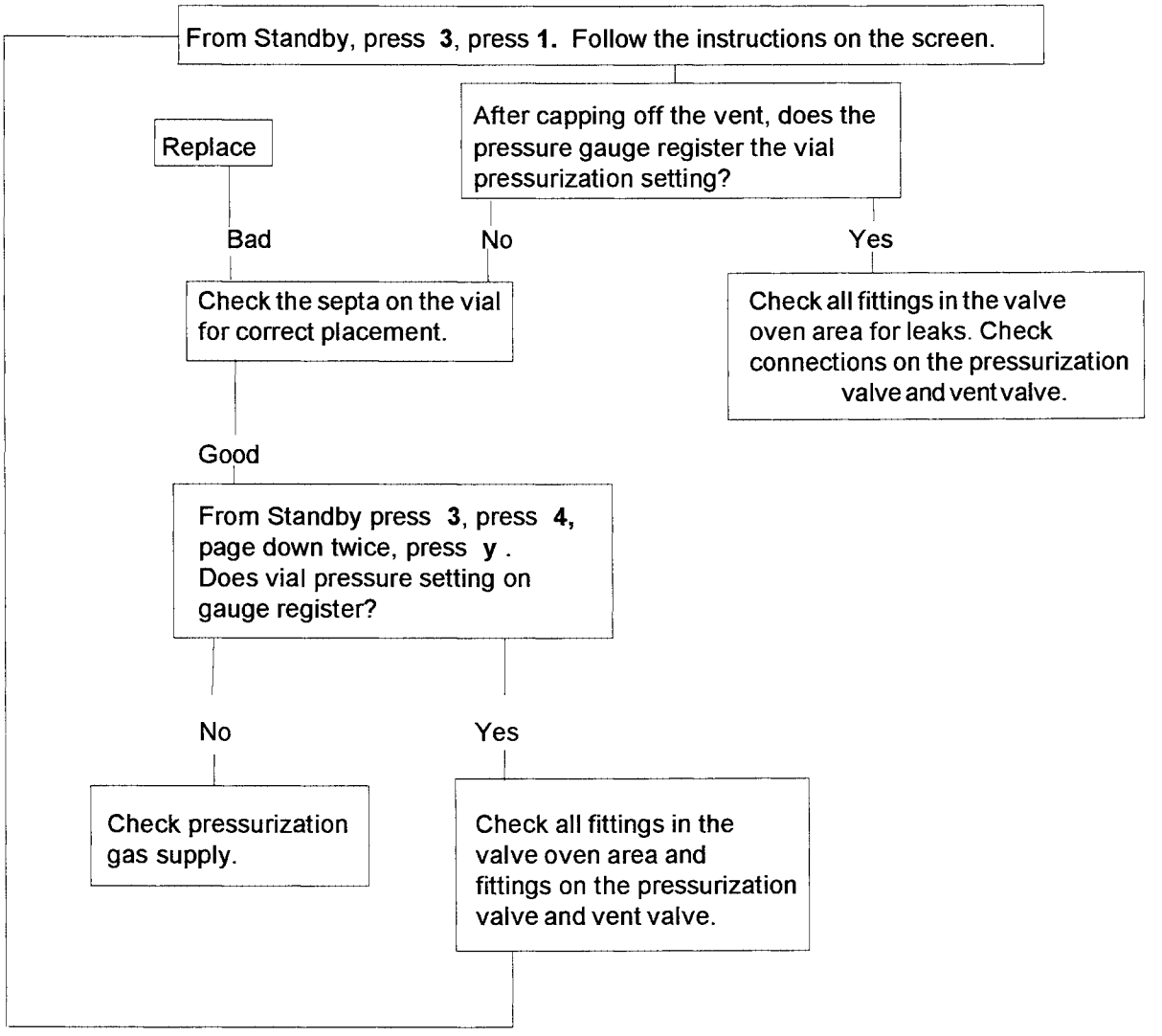


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7000 Leak Check

⚠ WARNING

Hazardous voltage inside.
Use caution when troubleshooting.

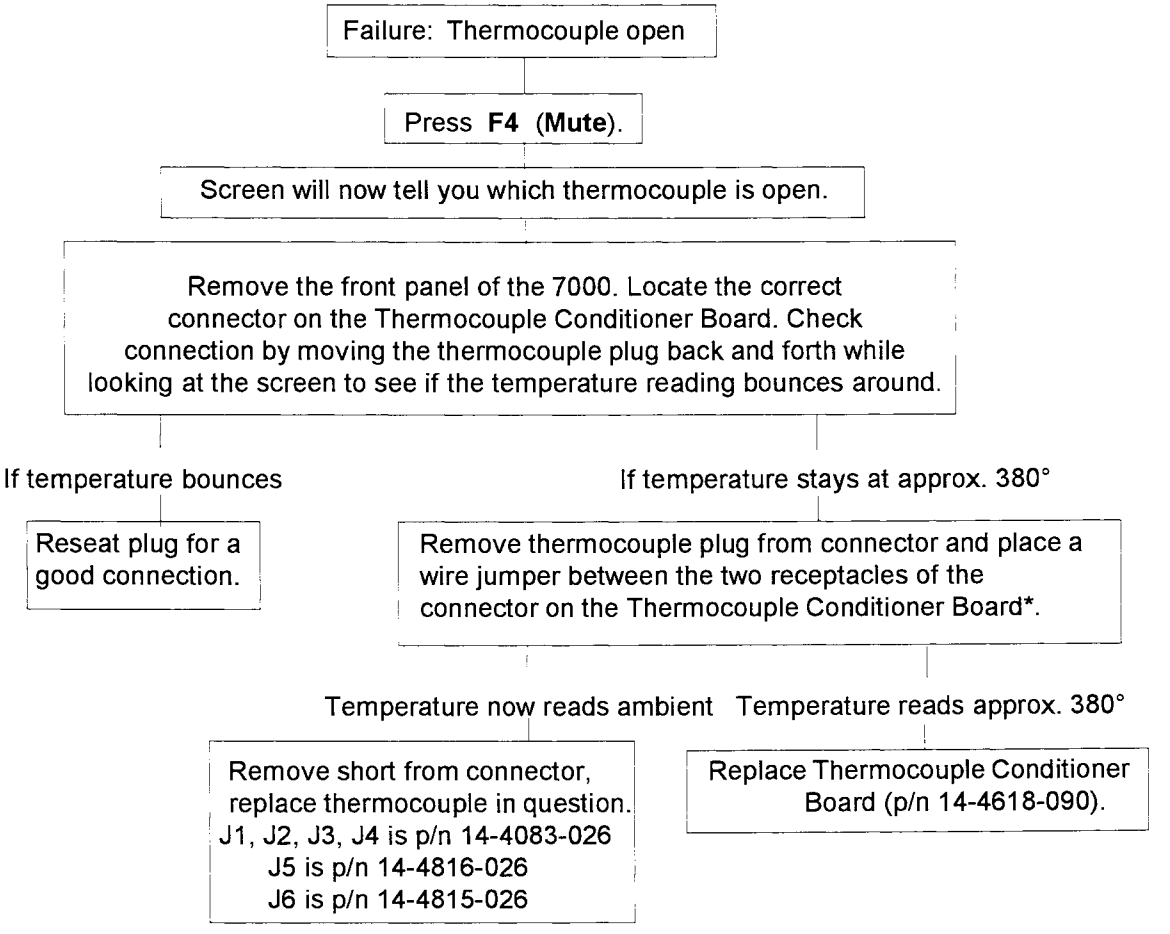


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7000 Heater Malfunctions

⚠ WARNING

Hazardous voltage inside.
Use caution when troubleshooting.



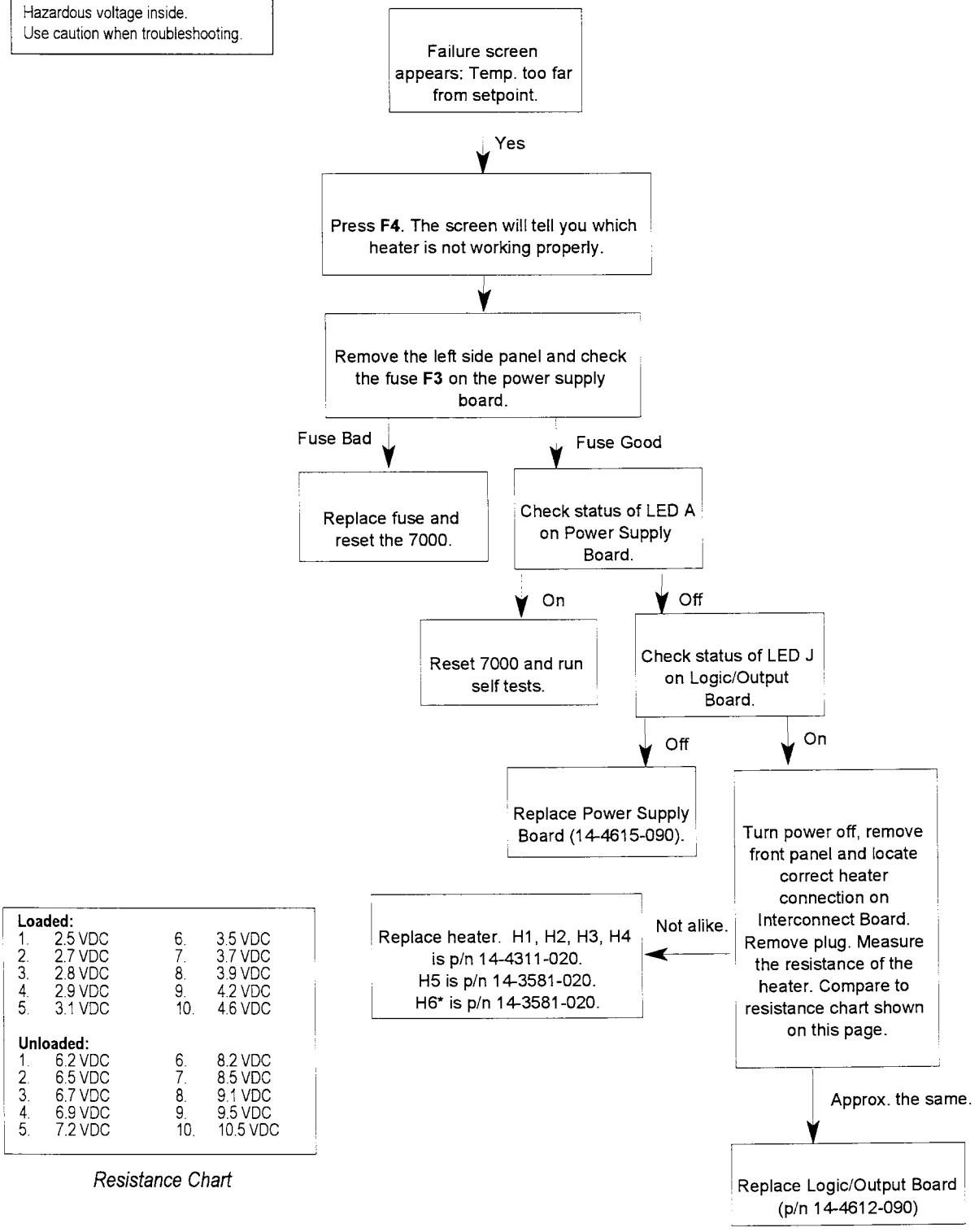
* If the temperature reads ambient, do not leave the wire jumper between the two receptacles for longer than one minute, or the heater temperature may "run away."

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7000 Heater Malfunctions

⚠ WARNING

Hazardous voltage inside.
Use caution when troubleshooting.



Loaded:			
1.	2.5 VDC	6.	3.5 VDC
2.	2.7 VDC	7.	3.7 VDC
3.	2.8 VDC	8.	3.9 VDC
4.	2.9 VDC	9.	4.2 VDC
5.	3.1 VDC	10.	4.6 VDC
Unloaded:			
1.	6.2 VDC	6.	8.2 VDC
2.	6.5 VDC	7.	8.5 VDC
3.	6.7 VDC	8.	9.1 VDC
4.	6.9 VDC	9.	9.5 VDC
5.	7.2 VDC	10.	10.5 VDC

Resistance Chart

H6, the valve oven heater, is three heaters connected in parallel. You will have to disconnect wires from the terminal strip to isolate the faulty heater. Each heater has a resistance of approximately 550 Ω

.

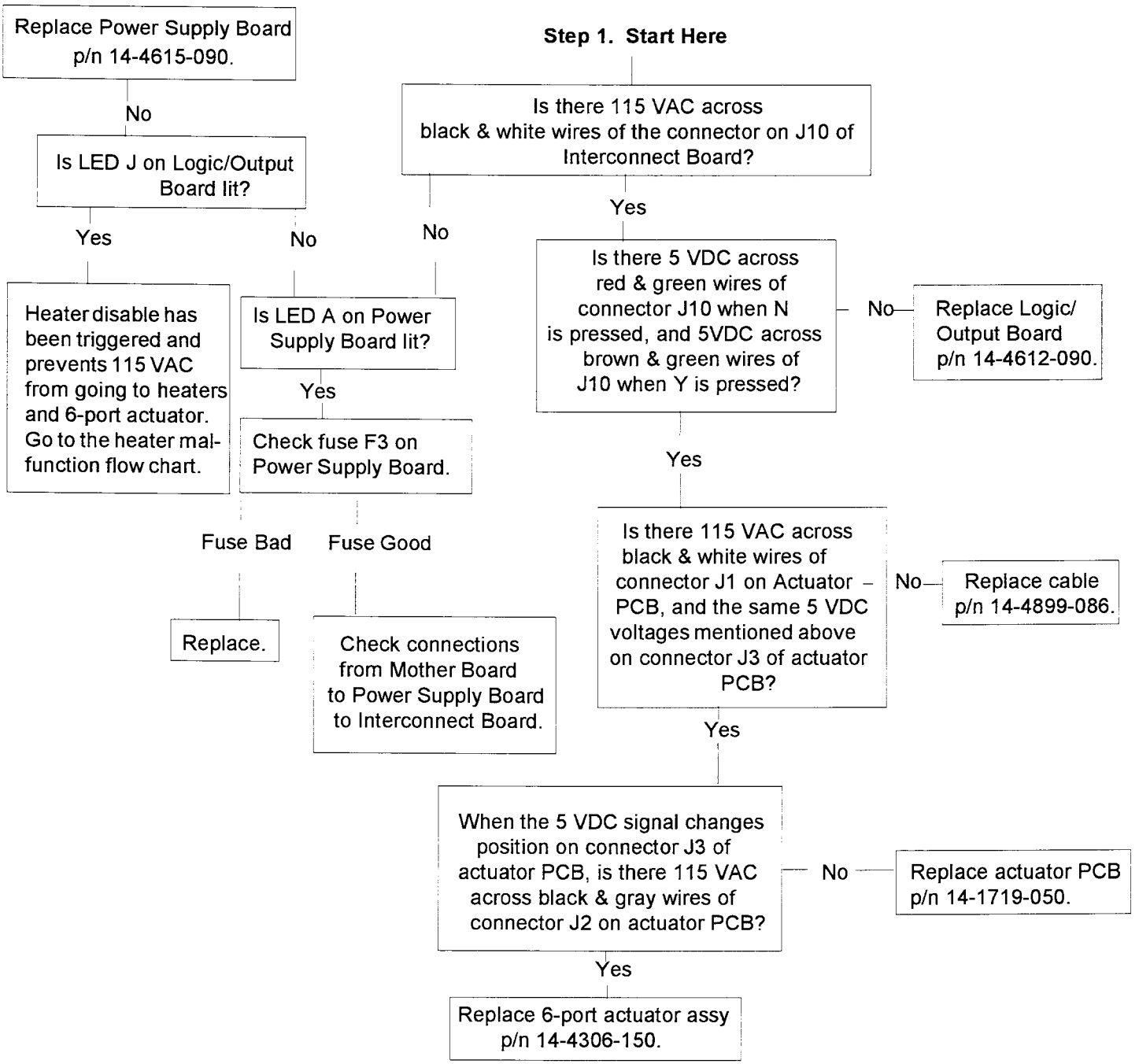
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6-Port Actuator Problems

⚠ WARNING

Hazardous voltage inside.
Use caution when troubleshooting.

From Standby press **3**, press **4**, and **PAGE DOWN** three times. Press **Y** at "6 Port" and then **N**. If you do not hear any sound, go to **Step 1**, if there is a sound repeat **Y** and **N** several times and see if you get a failure. If the sound repeats itself on one movement, try cleaning off the sensors on the Actuator PCB with canned air.



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Elevator Assembly Failures

⚠ WARNING

Hazardous voltage inside.
Use caution when troubleshooting.

Situation #1: Sensor didn't open. Screen:

```
Fatal Failure
Motor: __*__ Loader
Sensor: __*__ down
Sensor didn't open                    MUTE
```

* = Smpl, Mixer, or Vial

A. Remove the front, right side, and/or rear panels to gain access to the elevator assembly in question. Are both the ribbon cable and two-pin Molex connectors properly seated?

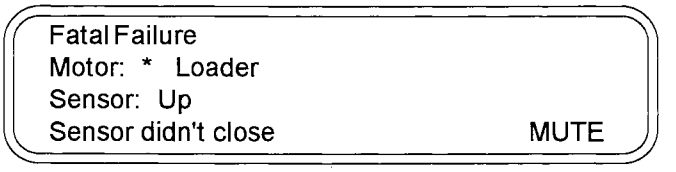
- No → Re-seat them.
- Yes → Remove the left side panel to see if the correct LED on the Motor Control Board turns on when **F4 (Exit)** is pressed from within the fatal failure screen. ***Note: The LED will only turn on for a couple of seconds before another failure is identified.*** Did the correct LED come on?
  - No → Replace the Motor Control Board (p/n 14-4613-090).
  - Yes → Remove the two-pin Molex connector from the PCB assembly and check the resistance of the motor.
    - 0 or infinite ohmage → Replace elevator assy.
      Sample 14-5167-000
      Mixer 14-5162-000
      Vial 14-5175-000
    - Good resistance reading → Replace PCB
      Sample 14-4622-090
      Mixer 14-4621-090
      Vial 14-4620-090

* The Sample Elevator assembly is p/n 14-5167-000; the Mixer Elevator assembly is p/n 14-5162-000; the Vial Elevator assembly is p/n 14-5175-000.

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Elevator Assembly Failures, cont.

Situation #2: Sensor didn't close. Screen:

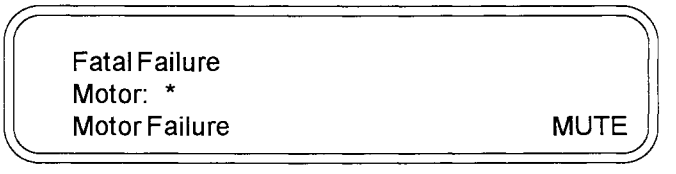


* = Smpl, mixer, or vial

Go to step A on the previous page.
Note: LED will stay on longer than a few seconds before another failure is identified

* The Sample Elevator assembly is p/n 14-5167-000; the Mixer Elevator assembly is p/n 14-5162-000; the Vial Elevator assembly is p/n 14-5175-000.

Situation #3: Sensor didn't close. Screen:



* = Smpl, Mixer, or Vial

Check sensor screen and make sure the platen has a clock bit (denoted by an asterisk at the bottom of the screen). Does it?

No

Pull the platen motor toward the front of the unit and adjust its position.

Exit sensor screen and retest motor.

Yes

Turn the instrument off, remove anything that covers path of elevator assembly in question, and check for clearance. Is anything blocking or jamming the path?

Yes

Remove and retest motor.

No

Remove elevator assembly from instrument base but leave plugged in and test.

Passes

Recheck for pathway clearance.

Fails

Replace elevator assembly.

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⚠ WARNING

Hazardous voltage inside.
Use caution when troubleshooting.

Carrousel & Platen Failures

Error: Sensor Conflict
Motor: Carrousel Index

A. Turn power off. Remove the 7050 Carrousel assembly. Remove the bottom cover of Carrousel via 6 Phillips screws.

Check the Carrousel Index Board transmissive assembly (U2, U3, and U4) for dust or dirt. Clean with canned air. Hook up Carrousel and run self test on Carrousel Indexer Board.

- Passes → Put cover back on. Proceed with operations.
- Does not pass → Replace Carrousel Indexer Board (p/n 14-4625-090).

Fatal Failure
Motor: Carrousel Position:____ Positioning Failure

Does Carrousel move?

- No → Press **F4 (Exit).** Does LED K on Motor Control Board come on?
  - No → Replace Motor Control Board. (p/n 14-4613-090).
  - Yes → Go to Step A above.
- Yes → Check continuity of 16-pin cable from Indexer Board to Encoder Board.
  - Bad → Replace cable (p/n 14-4884-086).
  - Good → Replace Encoder Board (p/n 14-4623-090).

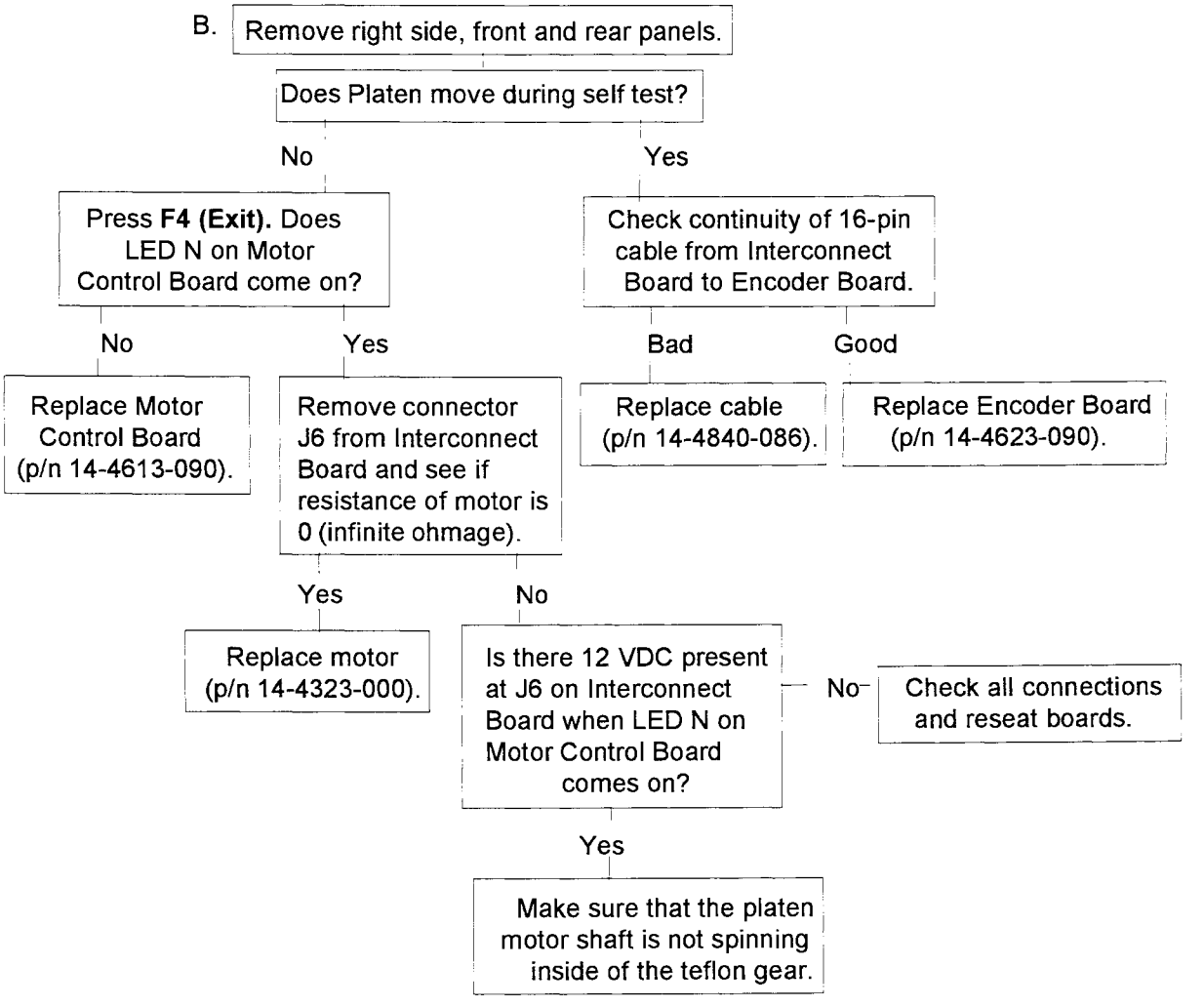
Remove connector from J4 of Indexer Board. Is resistance of motor 0 (infinite ohmage)?

- No → Check continuity of 25-pin cable from Motor Control Board to 7050 Carrousel.
  - Bad → Replace cable (p/n 14-4793-029).
  - Good → Replace Carrousel Indexer Board (p/n 14-4625-090).
- Yes → Replace motor (p/n 14-4426-000).

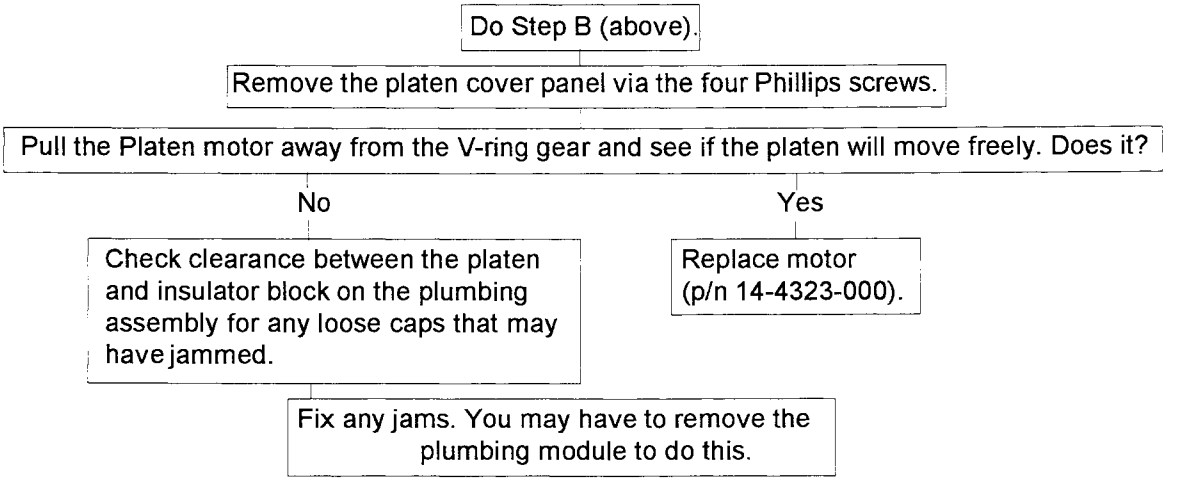
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Carrousel & Platen Failures, cont.

Fatal Failure
Motor: Platen **Position:_____** **Positioning Failure**



Fatal Failure
Motor: Platen **Position:_____** **Motor Failure**



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15.6 Expediting the Troubleshooting Process

There are two forms inserted into the manual following this page that will help Tekmar respond more efficiently to a problem or concern that may come up during your operation of the 7000/7050. They are:

- **Sample Submission Form** - This form is for your use anytime you would like help from Tekmar in analyzing and compiling chromatographic data on a particular sample. Specific instructions are included on the form.

- **Priority Resolution Form** - This form will help Tekmar to more efficiently address a specific service or application problem that you encounter when setting up or running the 7000/7050. Instructions are included on the form.

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Cincinnati, Ohio 45242-9576
Tel (800) 543-4461 • (513) 247-7000
Fax (513) 247-7050
Service (800) 874-2004

Tekmar 7000 Sample Submission Form

Client Information:

Name: _____

Phone: _____

Fax: _____

Company/Institution: _____

Address: City: _____ State: _____ Country: _____ Zip: _____

Optional: Manager: _____ Phone: (_____) _____

Alternate client contacts: _____

_____

The Purpose of This Documentation:

This documentation is to assist our efforts in providing you with the best possible results in the shortest period of time. This information may prove to be essential in producing representative results for use in your decision making process. **The sections of most importance are marked with an * . Please try to answer as many of these as possible and if time permits answer all others that apply.**

* *A. May Tekmar use the results from our analysis on your sample in technical publications?* ❒YES ❒NO

* *B. Would you be interested in a joint technical publication?* ❒YES ❒NO

* *C. Are the results from this analysis considered proprietary?* ❒YES ❒NO

Sample Information:

* *1. Due to regulatory requirements and potential hazard exposure we cannot accept samples which fall into the following categories:*

A. Biohazards including blood, serum, cultures, or any materials which may pose an infectious hazard.

B. Radioactive materials including radio labeled isotopes which fall under NRC regulations.

C. Teratogens

D. Toxic materials requiring EPA, NIOSH, or OSHA special permits for transportation, handling and disposal.

E. Regulated substances requiring special permits such as narcotics.

If your samples fall into any of these catagories, please contact your Tekmar representative or call us toll free at (800) 543-4461 for an onsite evaluation.

* *2. Do you have material safety data (MSDS information or equivalent) on the sample that you are submitting?*

❒YES ❒NO If the answer to 2 is YES, please submit this information with your sample.
If the answer to 2 is NO, please list the components that you anticipate are present in your sample.

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Analysis Detail:

* 1. Analyte Information:

Compound Name	Approximate Concentration	M.W.	M.P.	B.P.	Functional Groups	Solubility Data	
						Sol. In.	Insol. In.

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Analysis Detail Cont.:

* 2. *Matrix Information*

Component Name	M.W.	M.P.	B.P.	Functional Groups	Solubility Data Sol. In.	Insol. In.

* 3. *Does a method of analysis exist for this sample?* ❒YES ❒NO

If **YES,** please enclose a copy of this method, including sample collection\preparation and standardization.

* 4. *Method of analysis*: (i.e. Federal EPA method #, FDA, NIOSH, OSHA/method #)

Please specify: _____

* 5. *Sample size:* (ml, grams) _____

* 6. *Vial size/supplier:*

❒	9ml Tekmar	9ml other supplier: _____
❒	12ml Tekmar	12ml other supplier: _____
❒	22ml Tekmar	22ml other supplier: _____
❒	Other vial size: ___	Supplier: _____

* 7. *Septa type/supplier:*

❒	Butyl rubber Tekmar (grey)	Butyl rubber other supplier: _____
❒	Silicone rubber/Teflon face Tekmar(tan)	Silicone/Teflon face other supplier: _____
❒	Other septa type	Supplier: _____

* 8. *Cap type/supplier:*

❒	Regular aluminum Tekmar	Regular aluminum other supplier: _____
❒	Other cap type	Supplier: _____

9. *Crimper type/supplier:*

❒	Hand crimper Tekmar	Hand crimper other supplier: _____
❒	Crimp station Tekmar	Crimp station other supplier: _____
❒	Other	Supplier: _____

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Analysis Detail Cont.:

* 10. Carrier type: Purity: _____

* 11. Vial pressurization gas type: Purity: _____

* 12. Hydrocarbon trap: *(i.e. green canister/Tekmar)* Age: _____

13. Other supply gas purger traps: Age: _____

* 14. ROM version: (Upper right corner of Configuration Screen) _____

15. Transfer line back pressure on 7000: _____ (psi)

* 16. Column back pressure value on GC: _____ (psi)

* 17. Column flow rate: (measured at the exit of column or detector) _____

* 18. Transfer line controller flow element range: (circle appropriate range/type)

❒0-10cc (Blue) ❒0-25cc (Red w/ Silver Dot) ❒0-60cc (Red) ❒0-400cc (Black) Other: _____

* 19. Loop size: ❒100ul ❒250ul ❒500ul ❒1ml ❒2ml ❒3ml ❒5ml

20. Vial static pressure after being heated at the platen temperature for the total thermal exposure time: _____

* 21. Vial pressurization setting: _____ (psi)

* 22. Vial needle flow rate: _____ (cc/min)

23. Method number: _____

* 24. Platen temperature _____ (°C)

* 25. Platen equilibration time: _____ (min)

* 26. Sample equilibration time: _____ (min)

* 27. Vial size: ❒22ml ❒12ml ❒9ml

* 28. Mixer: ❒On ❒Off

* 29. Mixing time: _____ (min)

* 30. Mixing power: ❒1 ❒2 ❒3 ❒4 ❒5 ❒6 ❒7 ❒8 ❒9 ❒10

* 31. Stabilizing time: _____ (min)

* 32. Cryofocusing Module cooldown time: _____ (min)

* 33. Pressurization time: _____ (min)

* 34. Pressurize equilibrate time: _____ (min)

* 35. Loop fill time: _____ (min)

* 36. Loop equilibrate time: _____ (min)

* 37. Injection time: _____ (min)

* 38. Cryofocusing Module injection time: _____ (min)

* 39. Valve temperature: _____ (°C)

* 40. Line temperature: _____ (°C)

* 41. Cryofocusing Module union heater temperature: _____ (°C)

* 42. Injections per vial: ❒1 ❒2 ❒3 ❒4 ❒5 ❒6 ❒7 ❒8 ❒9

* 43. Septum puncture ❒With ❒Without

* 44. Re-Equil ❒Multiple (min) ❒Single

45. GC cycle time: _____ (min)

46. Method Optimization Mode: (M.O.M.™) ❒On ❒Off

47. Parameter being optimized: _____

48. Incremented by: ❒°C ❒min ❒Power Setting

Analysis Detail Cont.:

49. I/O dip switch settings:

Bias				Output				Input			
open	open	open	open	open	open	open	open	open	open	open	open
close	close	close	close	close	close	close	close	close	close	close	close
1	2	3	4	1	2	3	4	1	2	3	4

* *50. Heated transfer line type:* (line from 7000 to GC) ❒Fused Silica ❒Nickel Other: _____

* *51. Heated transfer line ID:.* ❒0.32mm (fused silica) ❒0.53mm (fused silica) ❒.034 "(nickel) Other: _____

* *52. HTL GC injection port interface:* ❒ Standard ❒Cryofocusing Module
❒Direct column connect ❒Septum needle adapter

* *53. GC:* Make: _____ Model: _____ Age/SN:_____

* *54. Injection port type:* ❒Packed ❒Capillary Split/Splitless ❒Capillary Splitless
❒Capillary on-column ❒ other:_____

* *55. Split ratio:*_____

56. Injection port cryofocus: ❒yes ❒no

57. Injection port temperature: _____ (°C)

* *58. Column type:* ❒Packed 1/4"(glass) ❒Packed 1/8"(metal) ❒Capillary (including megabore)

* *59. Column manufacturer:* _____

* *60. Column length:*_____ (m/ft)

* *61. Column I.D:* _____ (mm/in)

* *62. Column phase material:*_____

* *63. Column film thickness:* ❒Capillary _____ microns
❒Packed _____ loading and mesh size

* *64. Column flow rate:* _____ (cc/min)

65. Oven cryo: ❒yes ❒no Type: ❒LN_2 ❒$C0_2$

Oven Temperature Profile:

66. *Isothermal run profile:* Temperature: _____ (°C) Run time: _____ (min)

67. *Temperature programmed profile:*

Initial temperature: _____ (°C) Initial hold time: _____ (min)

RAMP 1: _____ (°C/min) RAMP 1 final temperature: _____ (°C) RAMP 1 hold time: _____ (min)

RAMP 2: _____ (°C/min) RAMP 2 final temperature: _____ (°C) RAMP 2 hold time: _____ (min)

RAMP 3: _____ (°C/min) RAMP 3 final temperature: _____ (°C) RAMP 3 hold time: _____ (min)

Final temperature: _____ (°C) Final hold time: _____ (min)

Recycle time to initial temperature (cooldown time): _____ (min)

68. *Actual G.C. cycle time:* (Total times from 68) _____ (min)

69. *Detector type(s):* ❒PID ❒ELCD ❒FID ❒ECD ❒MSD ❒NPD
❒TCD ❒FPD ❒IRD ❒Other:

70. *Detector:* Mftr: _____ Model: _____

71. *Detector temperature:* _____ (°C)

72. *Make up gas type:* Flow: _____ (cc/min) Pressure: _____ (psi)

73. *Aux gas type(s)* (1): _____ Flow: _____ (cc/min) Pressure: _____ (psi)
(2): _____ Flow: _____ (cc/min) Pressure: _____ (psi)
(3): _____ Flow: _____ (cc/min) Pressure: _____ (psi)

74. *M.S. interface type:* Capillary direct: _____ Open split: _____ Other: _____

75. *M.S. manufacturer:* Type: ❒QUAD ❒ITD ❒Other: _____
Model: _____ Age/SN: _____

76. *Pump type:* Rough pump: ❒E2M1 ❒E2M2 ❒Turbomolecular pump ❒Diffusion pump
❒multistage or differential pump ❒Other: _____

77. *Jet separator manufacturer:* _____

78. *Jet separator conditions:* _____ Temp: _____ (°C)

79. *M.S. pumping capacity:* _____ (cc/min)

80. *Typical operating vacuum:* _____ (Torr)

81. *Ionization mode:* ❒70EV electron impact (EI) ❒Chemical ionization (CI)
❒Fast atom bombardment (FAB) ❒Other: _____

82. *Data system:* Mftr: _____ Model: _____
Software: _____ Rev: _____

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83. Standards information:

Standard Name/ Concentration	Internal Standard	External Standard	Surrogate	Other	MW	MP	BP	Solubility Data Sol. In Insol.In.

* *84. Standard dissolved in:* _____

* *85. Standard injection volume:* _____

* *86. Quantitative method (% area, area, height, area/height, etc.):* _____

* *87. Integration parameters:* Attenuation: _____ Threshold: _____

Peak Width: _____ Other: _____

88. Calibration range: _____

89. Calibration type: (# of points, slope, intersept, etc.) _____

90. Are you using SIM? ❒Yes ❒No If no, what is the scan range? Low mass: _____

High mass: _____

* *91. Data enclosed:* ❒Chromatogram(s) ❒Mass spectra ❒Integration reports ❒Calibration table

❒Other: _____

_____

(Please attach the above data to this form)

* *92. Expected detection limits for analysis* _____ (%, part per million, etc.)

* *93. Acceptable signal to noise ratio, at lower limits of detection:* (ie 5:1 for 1NG or 5:1 for 1PPM,etc.-give range if applicable)

* *94. Acceptable limits for sample to sample carryover:* _____ (%, part per billion, etc.)

* *95. Anticipated sample load:*_____ (samples/day)

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Tekmar
7143 East Kemper Road • P.O. Box 429576
Cincinnati, Ohio 45242-9576
Tel (800) 543-4461 • (513) 247-7000
Fax (513) 247-7050
Service (800) 874-2004

Priority Resolution Form

Tekmar 7000 Headspace Autosampler Systems

FOR TEKMAR USE ONLY
Contact Date: ____/____/9____
Initial Tekmar Contact: _____
PRF Owner(s): _____

Client Information:

Name: _____

Phone: _____

Fax: _____

Company/Institution: _____

Address: City: _____ State: _____ Country: _____ Zip: _____

Optional: Manager: _____ Phone: (_____) _____

Alternate client contacts: _____

_____

Equipment in Question (check all that apply):

❒7000 ❒7050 ❒7000/7050 ❒Cryofocusing Module Other: _____

Equipment Installer (Name): _____

Company: _____

Installation Date: ____/____/9____

Serial Number(s): _____

Problem: _____

_____

_____

_____

_____

_____

_____

_____

_____

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When Problem First Occurred:

Changes Made Coincidental With Problem:

If the solution to this problem is not well documented, please supply all conditions on next page under 'Analysis Detail'.

Tekmar Suggested Solutions/Actions:

Follow-up Dates:

Results of Suggested Solutions/Actions:

Resolution Date:

Suggestions To Tekmar:

Analysis Detail:

1. Analyte Information:

Compound Name	Approximate Concentration	M.W.	M.P.	B.P.	Functional Groups	Solubility Data	
						Sol. In.	Insol. In.

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Analysis Detail Cont.:

* 2. *Matrix Information*

Component Name	M.W.	M.P.	B.P.	Functional Groups	Solubility Data Sol. In.	Insol. In.

* 3. *Does a method of analysis exist for this sample?* ❒YES ❒NO

If **YES,** please enclose a copy of this method, including sample collection\preparation and standardization.

* 4. *Method of analysis*: (i.e. Federal EPA method #, FDA, NIOSH, OSHA/method #)

Please specify: _____

* 5. *Sample size:* (ml, grams) _____

* 6. *Vial size/supplier:*

❒	9ml Tekmar	9ml other supplier: _____
❒	12ml Tekmar	12ml other supplier: _____
❒	22ml Tekmar	22ml other supplier: _____
❒	Other vial size: ___	Supplier: _____

* 7. *Septa type/supplier:*

❒	Butyl rubber Tekmar (grey)	Butyl rubber other supplier: _____
❒	Silicone rubber/Teflon face Tekmar(tan)	Silicone/Teflon face other supplier: _____
❒	Other septa type	Supplier: _____

* 8. *Cap type/supplier:*

❒	Regular aluminum Tekmar	Regular aluminum other supplier: _____
❒	Other cap type	Supplier: _____

9. *Crimper type/supplier:*

❒	Hand crimper Tekmar	Hand crimper other supplier: _____
❒	Crimp station Tekmar	Crimp station other supplier: _____
❒	Other	Supplier: _____

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Analysis Detail Cont.:

* 10. Carrier type: Purity: _____

* 11. Vial pressurization gas type: Purity: _____

* 12. Hydrocarbon trap: (i.e. green canister/Tekmar) Age: _____

13. Other supply gas purger traps: Age: _____

* 14. ROM version: (Upper right corner of Configuration Screen) _____

15. Transfer line back pressure on 7000: _____ (psi)

* 16. Column back pressure value on GC: _____ (psi)

* 17. Column flow rate: (measured at the exit of column or detector) _____

* 18. Transfer line controller flow element range: (circle appropriate range/type)

❒0-10cc (Blue) ❒0-25cc (Red w/ Silver Dot) ❒0-60cc (Red) ❒0-400cc (Black) Other: _____

* 19. Loop size: ❒100ul ❒250ul ❒500ul ❒1ml ❒2ml ❒3ml ❒5ml

20. Vial static pressure after being heated at the platen temperature for the total thermal exposure time: _____

* 21. Vial pressurization setting: _____ (psi)

* 22. Vial needle flow rate: _____ (cc/min)

23. Method number: _____

* 24. Platen temperature _____ (°C)

* 25. Platen equilibration time: _____ (min)

* 26. Sample equilibration time: _____ (min)

* 27. Vial size: ❒22ml ❒12ml ❒9ml

* 28. Mixer: ❒On ❒Off

* 29. Mixing time: _____ (min)

* 30. Mixing power: ❒1 ❒2 ❒3 ❒4 ❒5 ❒6 ❒7 ❒8 ❒9 ❒10

* 31. Stabilizing time: _____ (min)

* 32. Cryofocusing Module cooldown time: _____ (min)

* 33. Pressurization time: _____ (min)

* 34. Pressurize equilibrate time: _____ (min)

* 35. Loop fill time: _____ (min)

* 36. Loop equilibrate time: _____ (min)

* 37. Injection time: _____ (min)

* 38. Cryofocusing Module injection time: _____ (min)

* 39. Valve temperature: _____ (°C)

* 40. Line temperature: _____ (°C)

* 41. Cryofocusing Module union heater temperature: _____ (°C)

* 42. Injections per vial: ❒1 ❒2 ❒3 ❒4 ❒5 ❒6 ❒7 ❒8 ❒9

* 43. Septum puncture ❒With ❒Without

* 44. Re-Equil ❒Multiple (min) ❒Single

45. GC cycle time: _____ (min)

46. Method Optimization Mode: (M.O.M.™) ❒On ❒Off

47. Parameter being optimized: _____

48. Incremented by: ❒°C ❒min ❒Power Setting

Analysis Detail Cont.:

49. I/O dip switch settings:

Bias				Output				Input			
open	open	open	open	open	open	open	open	open	open	open	open
close	close	close	close	close	close	close	close	close	close	close	close
1	2	3	4	1	2	3	4	1	2	3	4

* *50. Heated transfer line type:* (line from 7000 to GC) ❒Fused Silica ❒Nickel Other: _____

* *51. Heated transfer line ID:.* ❒0.32mm (fused silica) ❒0.53mm (fused silica) ❒.034 "(nickel) Other: _____

* *52. HTL GC injection port interface:* ❒ Standard ❒Cryofocusing Module
❒Direct column connect ❒Septum needle adapter

* *53. GC:* Make: _____ Model: _____ Age/SN:_____

* *54. Injection port type:* ❒Packed ❒Capillary Split/Splitless ❒Capillary Splitless
❒Capillary on-column ❒ other:_____

* *55. Split ratio:*_____

56. Injection port cryofocus: ❒yes ❒no

57. Injection port temperature: _____ (°C)

* *58. Column type:* ❒Packed 1/4"(glass) ❒Packed 1/8"(metal) ❒Capillary (including megabore)

* *59. Column manufacturer:* _____

* *60. Column length:*_____ (m/ft)

* *61. Column I.D:* _____ (mm/in)

* *62. Column phase material:*_____

* *63. Column film thickness:* ❒Capillary _____ microns
❒Packed _____ loading and mesh size

* *64. Column flow rate:* _____ (cc/min)

65. Oven cryo: ❒yes ❒no Type: ❒LN_2 ❒$C0_2$

Oven Temperature Profile:

66. *Isothermal run profile:* Temperature: _____ (°C) Run time: _____ (min)

* 67. *Temperature programmed profile:*

Initial temperature: _____ (°C) Initial hold time: _____ (min)

RAMP 1: _____ (°C/min) RAMP 1 final temperature: _____ (°C) RAMP 1 hold time: _____ (min)

RAMP 2: _____ (°C/min) RAMP 2 final temperature: _____ (°C) RAMP 2 hold time: _____ (min)

RAMP 3: _____ (°C/min) RAMP 3 final temperature: _____ (°C) RAMP 3 hold time: _____ (min)

Final temperature: _____ (°C) Final hold time: _____ (min)

Recycle time to initial temperature (cooldown time): _____ (min)

68. *Actual G.C. cycle time:* (Total times from 68) _____ (min)

* 69. *Detector type(s):* ❒PID ❒ELCD ❒FID ❒ECD ❒MSD ❒NPD

❒TCD ❒FPD ❒IRD ❒Other:

* 70. *Detector:* Mftr: _____ Model: _____

71. *Detector temperature:* _____ (°C)

72. *Make up gas type:* Flow: _____ (cc/min) Pressure: _____ (psi)

73. *Aux gas type(s)* (1): _____ Flow: _____ (cc/min) Pressure: _____ (psi)

(2): _____ Flow: _____ (cc/min) Pressure: _____ (psi)

(3): _____ Flow: _____ (cc/min) Pressure: _____ (psi)

74. *M.S. interface type:* Capillary direct: _____ Open split: _____ Other: _____

75. *M.S. manufacturer:* Type: ❒QUAD ❒ITD ❒Other: _____

Model: _____ Age/SN: _____

76. *Pump type:* Rough pump: ❒E2M1 ❒E2M2 ❒Turbomolecular pump ❒Diffusion pump

❒multistage or differential pump ❒Other: _____

77. *Jet separator manufacturer:* _____

78. *Jet separator conditions:* _____ Temp: _____ (°C)

79. *M.S. pumping capacity:* _____ (cc/min)

80. *Typical operating vacuum:* _____ (Torr)

81. *Ionization mode:* ❒70EV electron impact (EI) ❒Chemical ionization (CI)

❒Fast atom bombardment (FAB) ❒Other: _____

82. *Data system:* Mftr: _____ Model: _____

Software: _____ Rev: _____

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83. Standards information:

Standard Name/ Concentration	Internal Standard	External Standard	Surrogate	Other	MW	MP	BP	Solubility Data Sol. In Insol.In.

** 84. Standard dissolved in:* _____

** 85. Standard injection volume:* _____

** 86. Quantitative method (% area, area, height, area/height, etc.):* _____

** 87. Integration parameters:* Attenuation: _____ Threshold: _____

Peak Width: _____ Other: _____

88. Calibration range: _____

89. Calibration type: (# of points, slope, intersept, etc.) _____

90. Are you using SIM? ❒Yes ❒No If no, what is the scan range? Low mass: _____

High mass: _____

** 91. Data enclosed:* ❒Chromatogram(s) ❒Mass spectra ❒Integration reports ❒Calibration table

❒Other: _____

_____

(Please attach the above data to this form)

** 92. Expected detection limits for analysis* _____ (%, part per million, etc.)

** 93. Acceptable signal to noise ratio, at lower limits of detection:* (ie 5:1 for 1NG or 5:1 for 1PPM,etc.-give range if applicable)

** 94. Acceptable limits for sample to sample carryover:* _____ (%, part per billion, etc.)

** 95. Anticipated sample load:*_____ (samples/day)

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16.1 Contacting Tekmar Service

Our Service Department can help locate the cause of a problem and can determine the best way to expedite repair. All replacement parts for the Tekmar 7000 and the 7050 Carrousel are described in this section. Tekmar's factory service facilities are located in Cincinnati, Ohio. For factory or on-site assistance, please call:

Toll Free Service
Assistance and Technical Support
8 am - 6 pm EST
(800) 874-2004
Outside the USA and Canada call
(513) 247-7000

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16.2 Replacement Parts and Accessories

Instrument Interface Options (Input/ Output Cables are Required for Operation)

Please include the model and serial number of your instrument when ordering spare parts.

14-2991-000	Interface, Hewlett-Packard (HP) 5890 GC
14-4830-086	Interface, two Tekmar 7000's on one HP 5890 (7000s must hook to separate columns)
14-4188-086*	Interface, HP 5890 w/5970 MSD and Unix or Pascal based software
14-4652-086	Interface, HP 5890 w/5970 MSD and Unix-B or MS-DOS software, HP 5890/5971 MSD and Unix-B or MS-DOS software, and HP 5890/5989 MS Engine
14-2993-000	Interface, HP 5995/96/85/87/88/92 GC/MS with HP-1000/ RTE GC/MS Software, HP 5890 w/5970 MSD and RTE (RTE-A, RTE-6, or Rev F**)
14-2974-000	Interface, HP 5700 Series (except 5710/30/90)
14-2976-000	Interface, HP 5710/30/90 GC w/5790 MSD with ChemStation using Quicksilver Software
14-2990-000	Interface, HP 5880A, 5840A
14-3318-000	Interface, HP 5995/96/87/85/92 with ChemStation-Quicksilver Software
14-3010-000	Interface Kit, HP 5995/85/93/92 GC/MS (includes I/O box and requires HP's BATCH or AQUARIUS software and an external events relay board to operate w/SIDS Data System)
14-2970-000	Interface, Perkin Elmer Sigma Series
14-3233-000	Interface, Perkin Elmer 8000 Series and 9000 Autosystem Series GC
14-2968-000	Interface, Varian 3300/3400/3500/3600
14-5044-086	Interface, two Tekmar 7000s to one Varian 3400 GC (7000s must hook to separate columns)
14-2969-000	Interface, Varian 3700
14-2966-000	Interface Kit, Varian Vista (includes I/O box for switching 2000A to 2000B) and Varian 6000
14-3052-000	Interface, A&B to Varian Vista I/O Box
14-2972-000	Interface, Tracor 560, 565, 570
14-2992-000	Interface, Tracor 540 and Waters Dimension I
14-4655-086	Interface, two Tekmar 7000s to one Tracor 540 (7000s must hook to separate columns)

* This cable requires the HP A111 (HP p/n 05990-60111) or A211 (HP p/n 05990-60211) accessory card and internal accessory cable (HP p/n 05987-60158) if using the Pascal software.

**Revision F uses both master and slave cables. Use pins 27 and 28 instead of 25 and 26 on the MS Molex plug.

continued

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	14-3430-000	Interface, Tracor 585/9000 and Waters Dimension II
	14-2973-000	Interface, Shimadzu GC 9A
	14-4610-086	Interface, Shimadzu GC 14A/15A, GC 14A w/QP 1000 EX MSD and GC 14A w/QP 2000 MSD
	14-4009-000	Interface Splicer Cable, Finnigan 5100/4000/4500 and OWA
	14-4938-086	Interface, Carlo Erba Mega /Vega Series and Fisons 8000
	14-3147-000	Interface, General Purpose/HNU 321
	14-4966-000	BCD Interface Kit for transmission of vial number to data system. Requires BCD Interface Cable
	14-4129-000	BCD Interface Cable
	14-5106-186	RS232 Interface Cable, 12 ft. (9 pin to 25 pin)
	14-5106-286	RS232 Interface Cable, 20 ft. (9 pin to 25 pin)
	14-5107-186	RS232 Interface Cable, 12 ft. (9 pin to 9 pin)
	14-5107-286	RS232 Interface Cable, 20 ft. (9 pin to 9 pin)
TekLink 7000™ (for remote control of units from a PC)	14-5731-076	TekLink 7000 Instrument Control Software Includes TEKLink 7000 software, one 3.5" diskette and one 5.25" floppy, 12 ft. (9-pin to 9-pin) RS232 cable, 9 pin to 25 pin cable adapter and instruction manual.
	14-5732-076	TekLink 7000 Instrument Control Software with ROM upgrade Same as 14-5731-076 with the addition of the 1.1 version ROM
		TekLink 7000 requires: • 80386 or higher computer • Microsoft™ Windows® 3.1 or greater • 1.1 or greater version 7000 ROM • An available serial port expansion board for TekLink cable
	14-4872-075	ROM with program 7000, Version 1.1
	14-5745-090	Serial port expansion board for PC
Loops	14-4943-067	Sample Loop Kit 100ul, 250ul, 500ul, 2 ml, 3 ml, 5 ml*
	14-4883-067	Sample Loop 100ul
	14-4870-067	Sample Loop 250ul
	14-4869-067	Sample Loop 500ul
	14-4382-067	Sample Loop 1ml
	14-4944-067	Sample Loop 2ml
	14-4945-067	Sample Loop 3ml
	14-4946-067	Sample Loop 5ml
	14-5606-167	E-form Sample Loop Kit 250ul, 500ul, 1ml, 2ml
	14-5544-067	E-Form Sample Loop 250ul**

continued

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14-5545-067	E-Form Sample Loop 500ul**
14-5541-067	E-Form Sample Loop 1ml**
14-5542-067	E-Form Sample Loop 2ml**

***Note: 2 ml, 3 ml, and 5 ml loops may require Cryofocusing Module (p/n 14-2530-200) for certain maximum sensitivity applications.**

****Note: E-Form is for use with alcohols and other polar compounds.**

Column Inlet Interface Accessories

14-5045-000	Variable Injection Pressure Regulator (VIPR)
14-6058-002	Polar Option Kit (tubing and fittings for transfer line, sample pathway, 1 ml Polar sample loop and E-type slider for 6-port valve); recommended for blood alcohols, sulfur, nitrogen, and phosphorous compounds.
14-5531-000	E-Form Option Kit (tubing and fittings for transfer line, sample pathway, 1 ml E-Form sample loop and E-type slider for 6-port valve); recommended for environmental VOCs and pharmaceutical residual solvents.
14-2530-200	Cryofocusing Module
14-3938-000	External Pressure Regulator Assembly
14-4925-050	Flow Element, 1-10 ml/min
14-4936-050	Flow Element, 0-25 ml/min
14-4926-050	Flow Element, 6-60 ml/min
14-4928-050	Flow Element, 40-440 ml/min
14-5408-050	Flow Element, 0-144 ml/min
14-4983-053	Vial Needle, replacement (after S/N 91032017)
14-4434-053	Vial Needle, replacement
14-4572-000	60" Sample Transfer Line Sheath
14-4913-153*	Septum Needle Adapter Kit, Varian 1040/1041 Packed Injection Port
14-4913-253*	Septum Needle Adapter Kit, HP 5890A Capillary Injection Port
14-4913-353*	Septum Needle Adapter Kit, HP 5890A Packed Injection Port
14-4913-453*	Septum Needle Adapter Kit, Varian SPI 1075/1077 and Tracor 540 Injection Port
14-4913-553*	Septum Needle Adapter Kit, all Perkin Elmer Injection Ports
14-4913-653*	Septum Needle Adapter Kit, Shimadzu 9A and 14A Injection Ports
14-5009-043	Septum Replacement for Septum Needle Adapter (5)

*** Note: You may need an additional flow element when using the septum needle adapter.**

Septum nuts available to adapt any of the above septum needle kits to a particular GC:

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14-1591-110	Septum Nut, Varian 1040/1041 Injection Port
14-5036-010	Septum Nut, HP 5890A Capillary Injection Port
14-5036-110	Septum Nut, HP 5890A Packed Injection Port
14-1591-410	Septum Nut, Varian SPI 1075/1077 and Tracor 540 Injection Ports
14-1591-510	Septum Nut, Perkin Elmer, all Sigma Series, all 8000 Series and Autosystem Injection Ports
14-1591-610	Septum Nut, Shimadzu 9A and 14A Injection Ports
14-4952-000	Low Volume Injector, HP Packed Injector
14-4952-100	Low Volume Injector, HP Purged Packed Injector
14-5506-000	Low Volume Injector, HP Split/Splitless Injector
14-4633-000	Low Volume Injector, Varian (except Varian 1075 Capillary Injector)
14-4634-000	Low Volume Injector, Perkin Elmer
14-4635-000	Low Volume Injector, Tracor 540, 585
14-4635-100	Low Volume Injector, Tracor 560, 565, 570
14-4635-200	Low Volume Injector, Tremetrics 9000
14-5046-000	Parameter Optimization Kit
13-0079-000	Digital Flow Meter
16-0013-000	Gasmet I Flow Meter
16-0128-000	Digiflow4000
21-0076-000	Model 21 Gas Leak Detector, 110V with rechargeable battery

Consumable Items

Vials

14-4440-124	22 ml Headspace Vials w/20 mm top (1000)
14-4440-024	22 ml Headspace Vials w/20 mm top (125)
14-4439-124	12 ml Headspace Vials w/20 mm top (1000)
14-4439-024	12 ml Headspace Vials w/20 mm top (125)
14-4438-124	9 ml Headspace Vials w/20 mm top (1000)
14-4438-024	9 ml Headspace Vials w/20 mm top (125)
14-5083-000	Insert Kit for 9ml or 12ml vials (contains 12 inserts, carrousel collar, and insert removal tool)
14-4048-079	Insert for 9 or 12 ml Headspace Vials
14-4365-027	Insert Removal Tool for 9 ml or 12 ml Insert
14-4522-079	Carrousel Collar for 9 ml or 12 ml Vials
14-4661-079	Carrousel Collar for 22 ml Vials

Septa

14-5818-143	Silicon Rubber/Teflon Face Septa for 20mm top vials (1000)
14-5818-043	Silicon Rubber/Teflon Face Septa for 20mm top vials (125)
14-4385-143	Butyl Rubber Septa w/out Teflon Face for 20mm top vials (1000)
14-4385-043	Butyl Rubber Septa w/out Teflon Face for 20mm top vials (125)

continued

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	Caps	
	14-4436-100	Crimp Cap for Headspace Septa and Vials (1000)
	14-4436-000	Crimp Cap for Headspace Septa/Vials (125)
Sample Preparation Accessories	14-4863-027	Tool, Hand Crimper 20mm Top Vials
	14-5020-027	Crimp Station, Air-Powered
	14-4864-027	Tool, Decapper for 20mm Top Vials
	14-4865-027	Crimp Station for 20mm Top Vials
	14-4386-027	20 mm Jaw Set for Crimp Station
	14-4866-027	Vial Rack Holder for 10 Vials
Fittings	14-0264-016	Fitting 1/16" Bulkhead S.S.
	14-0356-016	Fitting, 1/8" Bulkhead Filter Assy
	14-0243-016	Nut, Valco 1/16"
	14-4695-016	Tee, Union, 1/8" Brass
	12-0070-016	Tee, Union 1/8" Brass Swagelok
	14-0051-016	Union, 1/16" S.S.
	12-0069-016	Nut, Cap 1/8" Brass Swagelok
	14-0798-016	Nut, 1/16" Upchurch Fitting
	14-0241-016	Ferrule, 1/16" S.S.
	12-0073-016	Union, 1/8" Brass
	14-0799-016	Ferrule, 1/16" Upchurch Fitting
	12-0044-016	Ferrule 1/8" Brass Set, Swagelok
	14-0540-016	Ferrule, Vespel, 0.5mm ID
	14-2074-016	Ferrule, Graphite Vespel, 0.8" ID
	14-1967-016	Cap Plug 1/8" Brass Swagelok
	14-2792-016	Nut Plug 1/16" Brass
Tubing	14-5229-002	Tubing, 1/16" Nickel (Large Bore)
	14-0546-002	Tubing, 1/8" Copper
	14-3592-002	Tubing, Fused Silica 0.32 mm ID
	14-3591-002	Tubing, Fused Silica 0.53 mm ID
	14-5540-002	Tubing, Electroform, 0.04 ID
	14-5543-002	Tubing, Electroform, 0.02 ID
Electronics	14-4966-000	Kit, BCD, 7000
	14-4129-000	Cable, BCD, 7000
	14-4966-090	BCD Interface
	14-4616-090	Mother Board Assy
	14-4619-090	Interconnect Board
	14-4618-090	Thermocouple Conditioner Assy
	14-4623-090	Encoder Board

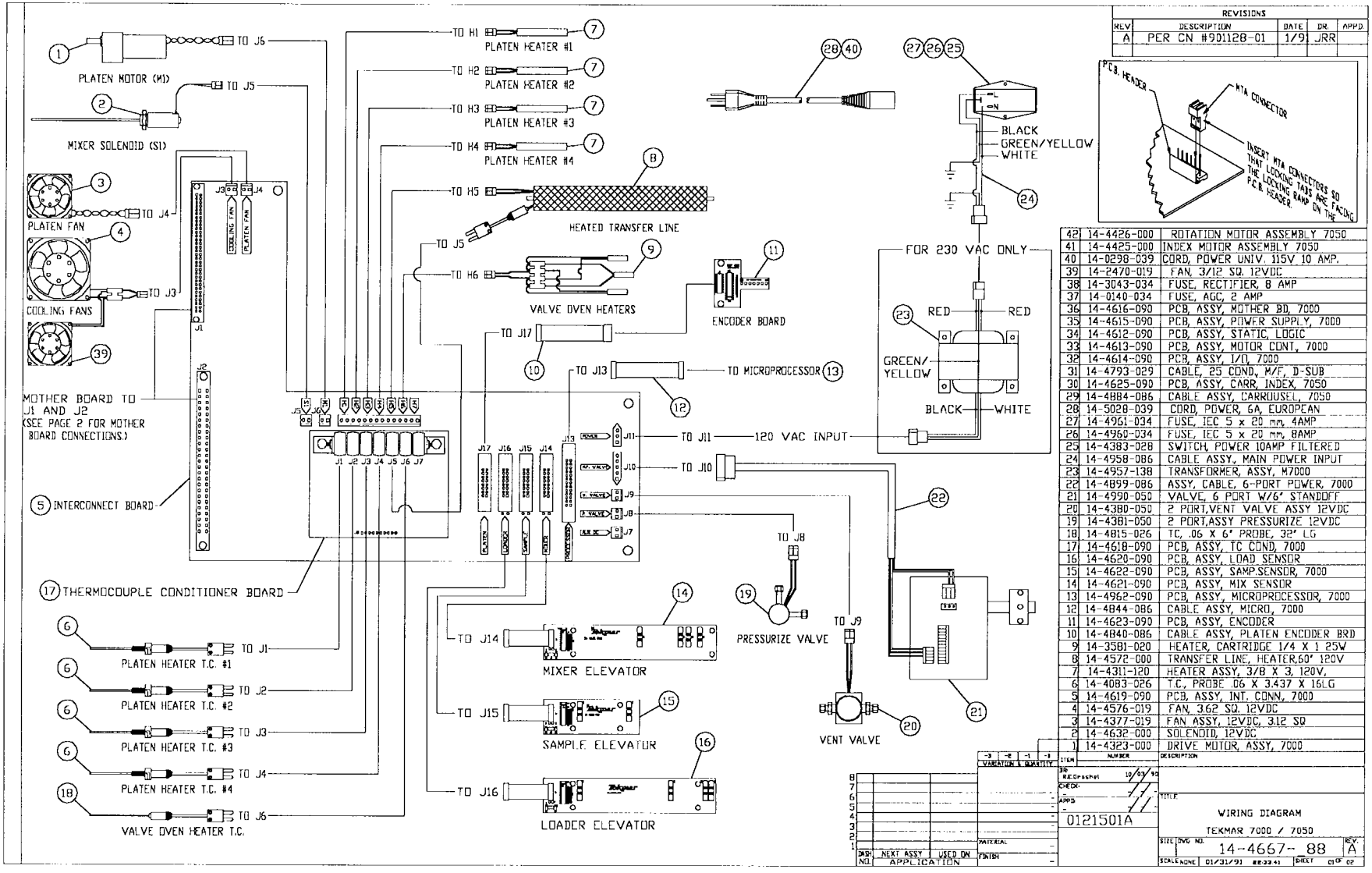
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	14-4844-086	Cable, Microprocessor, 7000
	14-4840-086	Cable, Platen Encoder
	14-4615-090	Power Supply Board
	14-4612-090	Logic Board
	14-4613-090	Motor Control Board
	14-4614-090	Interface Board
	14-4621-090	Mixer Board
	14-4620-090	Loader Board
	14-4622-090	Sample Board
	14-4625-090	Carrousel Index Board (7050)
	14-1719-050	Actuator Board
	14-4383-028	Switch, Power 10 amp Filtered
	14-4960-034	Fuse, 1ec 8 amp 5x20 mm (120V)
	14-4961-034	Fuse, 1ec 4 amp 5x20mm (220V)
	14-4957-138	Transformer Assy (220V)
	14-5177-138	Transformer Assy (100V)
	14-0298-039	Cable, Power Universal
	14-0140-034	Fuse AGC, 2 amp
	14-3043-034	Fuse Rectifier, 8 amp
	14-4962-090	Microprocessor 7000
	14-4872-075	ROM w/Program 7000, Version 1.1
	14-4793-029	Cable, 25 Cond. M/F
	14-4589-000	Electronics Front Panel
	14-4958-086	Cable, Power Module to Interconnect Bd
	14-4884-086	Cable, 7050 Carrousel, Encoder
	14-5107-186	Cable, RS232 (9 pin to 9 pin IBM)
	14-5106-186	Cable, RS232 (9 pin to 25 pin)
	14-4565-083	Front Panel Keypad (Grey) SN< 91346009
	14-4565-883	Front Panel Keypad (White) SN> 91346009
Heaters	14-4311-120	Heater 3/8 x 3" lg Assy for Platen
	14-4573-000	Valve Heater Assy, 7000
	14-4083-026	Thermocouple Probe (Platen Heater)
	14-4572-000	60" Sample Transfer Line Sheath, replacement
Valves and Pneumatics	14-4897-050	Regulator, Pressure, with 1/8" Fitting
	14-5026-050	Regulator, Pressure, with 1/16" Fitting
	14-4974-050	Flow Controller, 0-60 cc/min
	14-4570-000	Pressure Gauge Assy, 0-30 psi, 1/16" Tube
	14-3939-000	Pressure Gauge Assy, 0-30 psi, 1/8" Tube
	14-4381-050	Valve, 2-Port, Pressure 12 VDC
	14-4380-050	Valve, 2-Port, Vent 12 VDC
	14-4306-050	Valve, 6-Port 6" Standoff
	14-4306-150	Valve, 6-Port Actuator w/Boards
	14-3149-050	Valve, 6-Port Body and Slider

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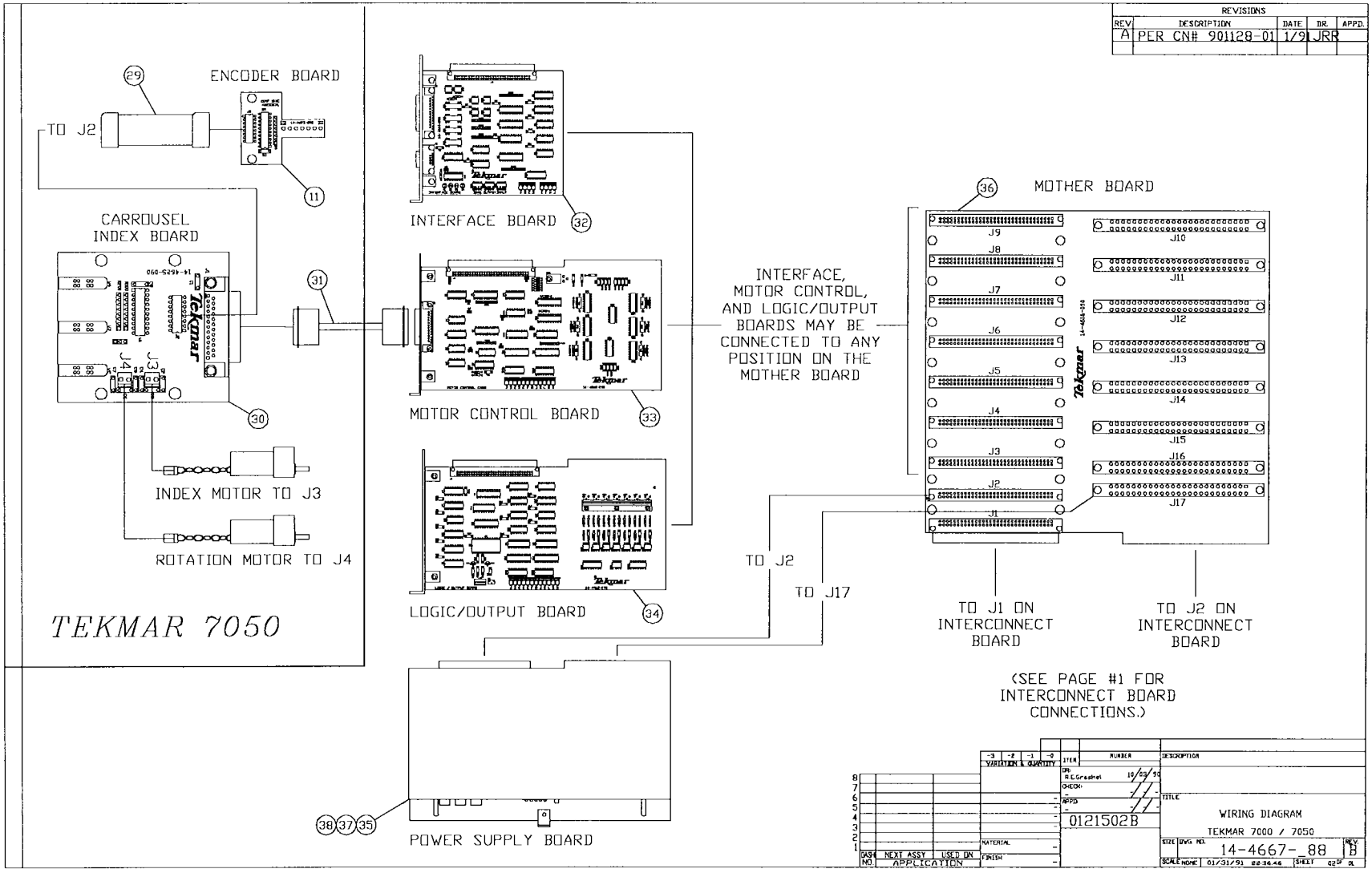
	14-5573-050	Slider, E-type, for 6-port valve
	14-4312-000	Valve Plumbing Assembly
	14-5004-050	6" Standoff Valve
Miscellaneous	14-5093-000	7000 Installation Kit
	14-4976-000	7000 Enhancement Kit
	14-1362-000	Hydrocarbon Trap Assy
	14-4900-167	Internal Vent Restrictor Assy
	14-4377-019	Fan Assy, 3.12 SQ 12 VDC, Platen Heater Exchanger
	14-4378-019	Fan Assy, 3.62 SQ 12 VDC, Rear Panel (before S/N 91032017)
	14-2470-019	Fan, Low Profile, plumbing cooling (from S/N 91032018)
	14-4576-019	Fan, Box, 12 VDC, lower unit cooling
	14-4632-000	Solenoid 12 VDC
	14-5162-000	Mixer Elevator Assy
	14-5175-000	Elevator Loader Assy
	14-5167-000	Elevator Sampler Assy
	14-4674-080	Bracket, Expansion, Slot Cover
	14-4332-000	Carton, 7000
	14-4417-000	Carton, 7050
	14-2987-000	Tool, Phillips Screwdriver
	14-4847-027	Tool, 5/16 Blade Screwdriver
	14-4871-027	Tool, #6 Nut Driver
	14-4845-027	Tool, Wrench 1/4-5/16"
	14-4846-027	Tool, Wrench 7/16-1/2"
	14-4365-027	Tool, Insert Removal
	14-4849-000	Thermal Plug Assy
	14-4262-080	Clip, Transfer Line
	14-2454-010	Thumb Nut, #6-32
	12-0323-001	Screw, 6-32 x 3/8 100° Flat HD
	14-4522-079	Carrousel Collar for 9-12ml Vials
	14-4661-079	Carrousel Collar for 22ml Vials
	14-4048-079	Platen Inserts, 9-12 ml Vials
	14-4891-000	Dust Cover Assy, 7050
	14-4218-000	Carrousel 50 Position Deck (7050)
	14-4896-031	Knob, Clamp (For 50 Position Deck, 7050)
	14-4314-000	Valve Oven Cover Assy
	14-4568-079	Shipping Spacer, Hold Down Platen
	14-4366-001	Screw, 1/4 - 20 x 5, Round Head
	14-4983-053	Sample Needle (after S/N 91032017)
	14-4333-000	Instruction Manual
	14-4400-074	7000 Service Manual

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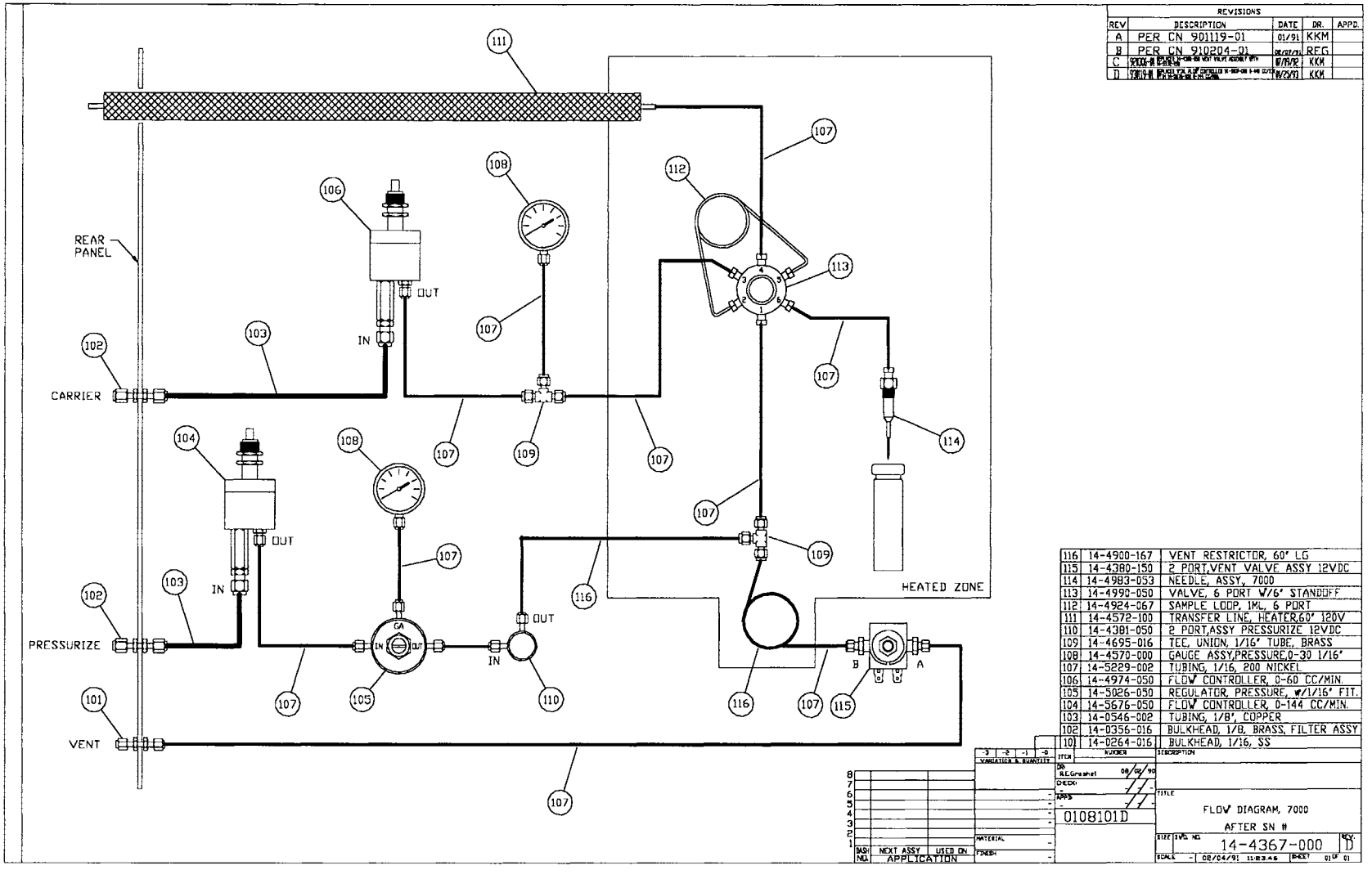
7000 Wiring Diagram page 1

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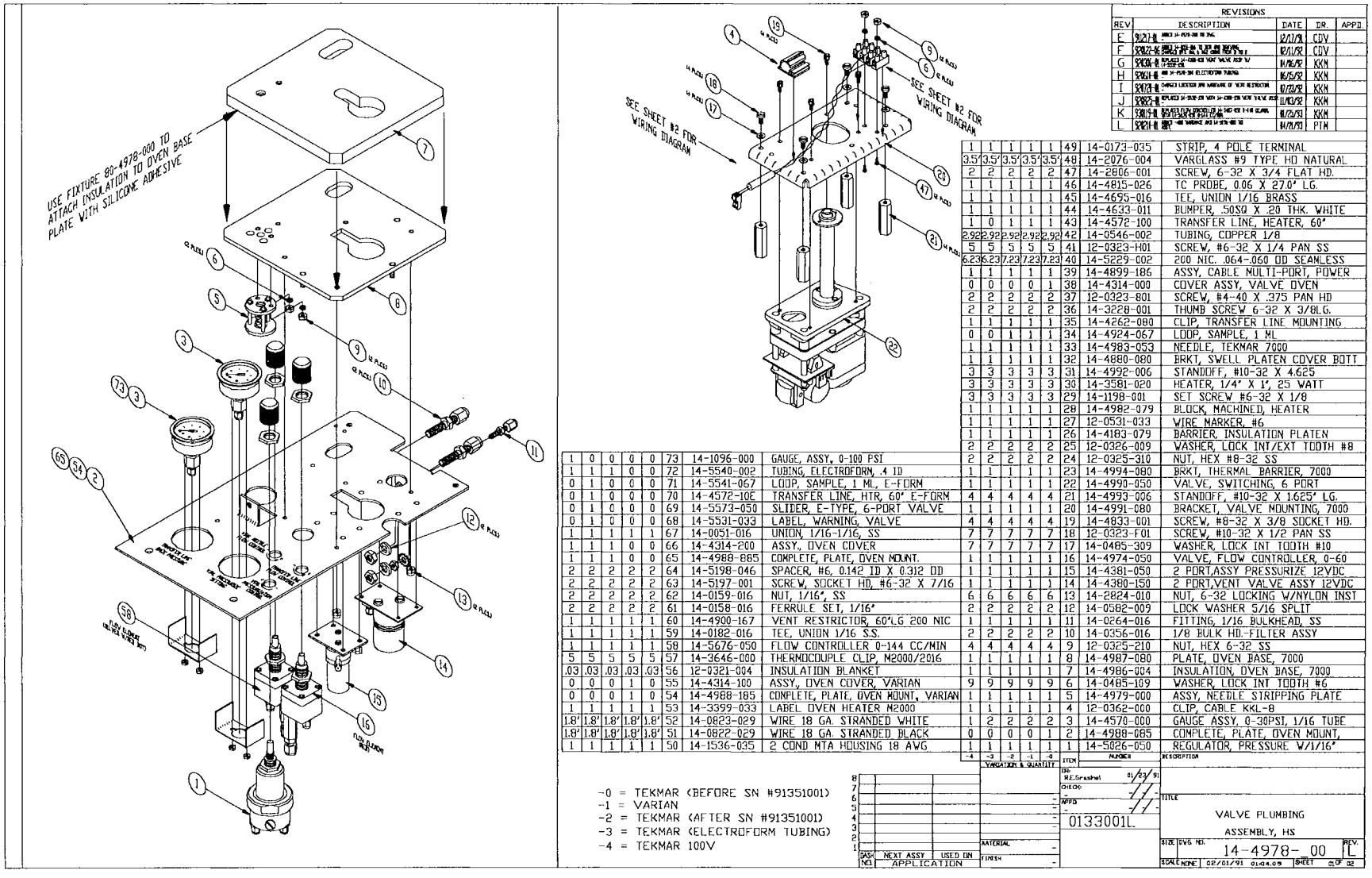
7000 Wiring Diagram page 2

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7000 Flow Diagram

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REVISIONS				
REV.	DESCRIPTION	DATE	DR.	APPD
E	[illegible]	12/01/89	CDV	
F	[illegible]	12/01/89	CDV	
G	[illegible]	11/06/92	KKM	
H	[illegible]	11/25/92	KKM	
I	[illegible]	12/22/92	KKM	
J	[illegible]	12/23/92	KKM	
K	[illegible]	01/22/93	KKM	
L	[illegible]	01/27/93	PTM	

-0	-1	-2	-3	-4	ITEM	PART NO.	DESCRIPTION
1	1	1	1	1	49	14-0173-035	STRIP, 4 POLE TERMINAL
3.5'	3.5'	3.5'	3.5'	3.5'	48	14-2076-004	VARGLASS #9 TYPE HO NATURAL
2	2	2	2	2	47	14-2806-001	SCREW, 6-32 X 3/4 FLAT HD.
1	1	1	1	1	46	14-4815-026	TC PROBE, 0.06 X 27.0' LG.
1	1	1	1	1	45	14-4695-016	TEE, UNION 1/16 BRASS
1	1	1	1	1	44	14-4633-011	BUMPER, .50SQ X .20 THK. WHITE
1	0	1	1	1	43	14-4572-100	TRANSFER LINE, HEATER, 60'
2.92	2.92	2.92	2.92	2.92	42	14-0546-002	TUBING, COPPER 1/8
5	5	5	5	5	41	12-0323-H01	SCREW, #6-32 X 1/4 PAN SS
6.23	6.23	7.23	7.23	7.23	40	14-5229-002	200 NIC. .064-.060 OD SEAMLESS
1	1	1	1	1	39	14-4899-186	ASSY, CABLE MULTI-PORT, POWER
0	0	0	0	1	38	14-4314-000	COVER ASSY, VALVE OVEN
2	2	2	2	2	37	12-0323-801	SCREW, #4-40 X .375 PAN HD
2	2	2	2	2	36	14-3228-001	THUMB SCREW 6-32 X 3/8LG.
1	1	1	1	1	35	14-4262-080	CLIP, TRANSFER LINE MOUNTING
0	0	1	1	1	34	14-4924-067	LOOP, SAMPLE, 1 ML
1	1	1	1	1	33	14-4983-053	NEEDLE, TEKMAR 7000
1	1	1	1	1	32	14-4880-080	BRKT, SWELL PLATEN COVER BOTT
3	3	3	3	3	31	14-4992-006	STANDOFF, #10-32 X 4.625
3	3	3	3	3	30	14-3581-020	HEATER, 1/4' X 1', 25 WATT
3	3	3	3	3	29	14-1198-001	SET SCREW #6-32 X 1/8
1	1	1	1	1	28	14-4982-079	BLOCK, MACHINED, HEATER
1	1	1	1	1	27	12-0531-033	WIRE MARKER, #6
1	1	1	1	1	26	14-4183-079	BARRIER, INSULATION PLATEN
2	2	2	2	2	25	12-0326-009	WASHER, LOCK INT/EXT TOOTH #8
2	2	2	2	2	24	12-0325-310	NUT, HEX #8-32 SS
1	1	1	1	1	23	14-4994-080	BRKT, THERMAL BARRIER, 7000
1	1	1	1	1	22	14-4990-050	VALVE, SWITCHING, 6 PORT
4	4	4	4	4	21	14-4993-006	STANDOFF, #10-32 X 1.625' LG.
1	1	1	1	1	20	14-4991-080	BRACKET, VALVE MOUNTING, 7000
4	4	4	4	4	19	14-4833-001	SCREW, #8-32 X 3/8 SOCKET HD.
7	7	7	7	7	18	12-0323-F01	SCREW, #10-32 X 1/2 PAN SS
7	7	7	7	7	17	14-0485-309	WASHER, LOCK INT TOOTH #10
1	1	1	1	1	16	14-4974-050	VALVE, FLOW CONTROLLER, 0-60
1	1	1	1	1	15	14-4381-050	2 PORT,ASSY PRESSURIZE 12VDC
1	1	1	1	1	14	14-4380-150	2 PORT,VENT VALVE ASSY 12VDC
6	6	6	6	6	13	14-2824-010	NUT, 6-32 LOCKING W/NYLON INST
2	2	2	2	2	12	14-0582-009	LOCK WASHER 5/16 SPLIT
1	1	1	1	1	11	14-0264-016	FITTING, 1/16 BULKHEAD, SS
2	2	2	2	2	10	14-0356-016	1/8 BULK HD.-FILTER ASSY
4	4	4	4	4	9	12-0325-210	NUT, HEX 6-32 SS
1	1	1	1	1	8	14-4987-080	PLATE, OVEN BASE, 7000
1	1	1	1	1	7	14-4986-004	INSULATION, OVEN BASE, 7000
9	9	9	9	6	6	14-0485-109	WASHER, LOCK INT TOOTH #6
1	1	1	1	1	5	14-4979-000	ASSY, NEEDLE STRIPPING PLATE
1	1	1	1	1	4	12-0362-000	CLIP, CABLE KKL-8
1	2	2	2	2	3	14-4570-000	GAUGE ASSY, 0-30PSI, 1/16 TUBE
0	0	0	0	1	2	14-4988-085	COMPLETE, PLATE, OVEN MOUNT,
1	1	1	1	1	1	14-5026-050	REGULATOR, PRESSURE W/1/16'
-4	-3	-2	-1	-0	ITEM	PART NO.	DESCRIPTION

-0	-1	-2	-3	-4	ITEM	PART NO.	DESCRIPTION
1	0	0	0	0	73	14-1096-000	GAUGE, ASSY, 0-100 PSI
1	1	1	0	0	72	14-5540-002	TUBING, ELECTROFORM, .4 ID
0	1	0	0	0	71	14-5541-067	LOOP, SAMPLE, 1 ML, E-FORM
0	1	0	0	0	70	14-4572-10E	TRANSFER LINE, HTR, 60' E-FORM
0	1	0	0	0	69	14-5573-050	SLIDER, E-TYPE, 6-PORT VALVE
0	1	0	0	0	68	14-5531-033	LABEL, WARNING, VALVE
1	1	1	1	1	67	14-0051-016	UNION, 1/16-1/16, SS
1	1	1	0	0	66	14-4314-200	ASSY, OVEN COVER
1	1	1	0	0	65	14-4988-885	COMPLETE, PLATE, OVEN MOUNT.
2	2	2	2	2	64	14-5198-046	SPACER, #6, 0.142 ID X 0.312 OD
2	2	2	2	2	63	14-5197-001	SCREW, SOCKET HD, #6-32 X 7/16
2	2	2	2	2	62	14-0159-016	NUT, 1/16', SS
2	2	2	2	2	61	14-0158-016	FERRULE SET, 1/16'
1	1	1	1	1	60	14-4900-167	VENT RESTRICTOR, 60'LG .200 NIC
1	1	1	1	1	59	14-0182-016	TEE, UNION 1/16 SS
1	1	1	1	1	58	14-5676-050	FLOW CONTROLLER 0-144 CC/MIN
5	5	5	5	5	57	14-3646-000	THERMOCOUPLE CLIP, M2000/2016
.03	.03	.03	.03	.03	56	12-0321-004	INSULATION BLANKET
0	0	0	1	0	55	14-4314-100	ASSY., OVEN COVER, VARIAN
0	0	0	1	0	54	14-4988-185	COMPLETE, PLATE, OVEN MOUNT, VARIAN
1	1	1	1	1	53	14-3399-033	LABEL OVEN HEATER M2000
1.8'	1.8'	1.8'	1.8'	1.8'	52	14-0823-029	WIRE 18 GA. STRANDED WHITE
1.8'	1.8'	1.8'	1.8'	1.8'	51	14-0822-029	WIRE 18 GA. STRANDED BLACK
1	1	1	1	1	50	14-1536-035	2 COND MTA HOUSING 18 AWG
-4	-3	-2	-1	-0	ITEM	PART NO.	DESCRIPTION

-0 = TEKMAR (BEFORE SN #91351001)
-1 = VARIAN
-2 = TEKMAR (AFTER SN #91351001)
-3 = TEKMAR (ELECTROFORM TUBING)
-4 = TEKMAR 100V

DASH NO.	NEXT ASSY USED ON APPLICATION
8	
7	
6	
5	
4	
3	
2	
1	

VARIATION & QUANTITY

MATERIAL

FINISH

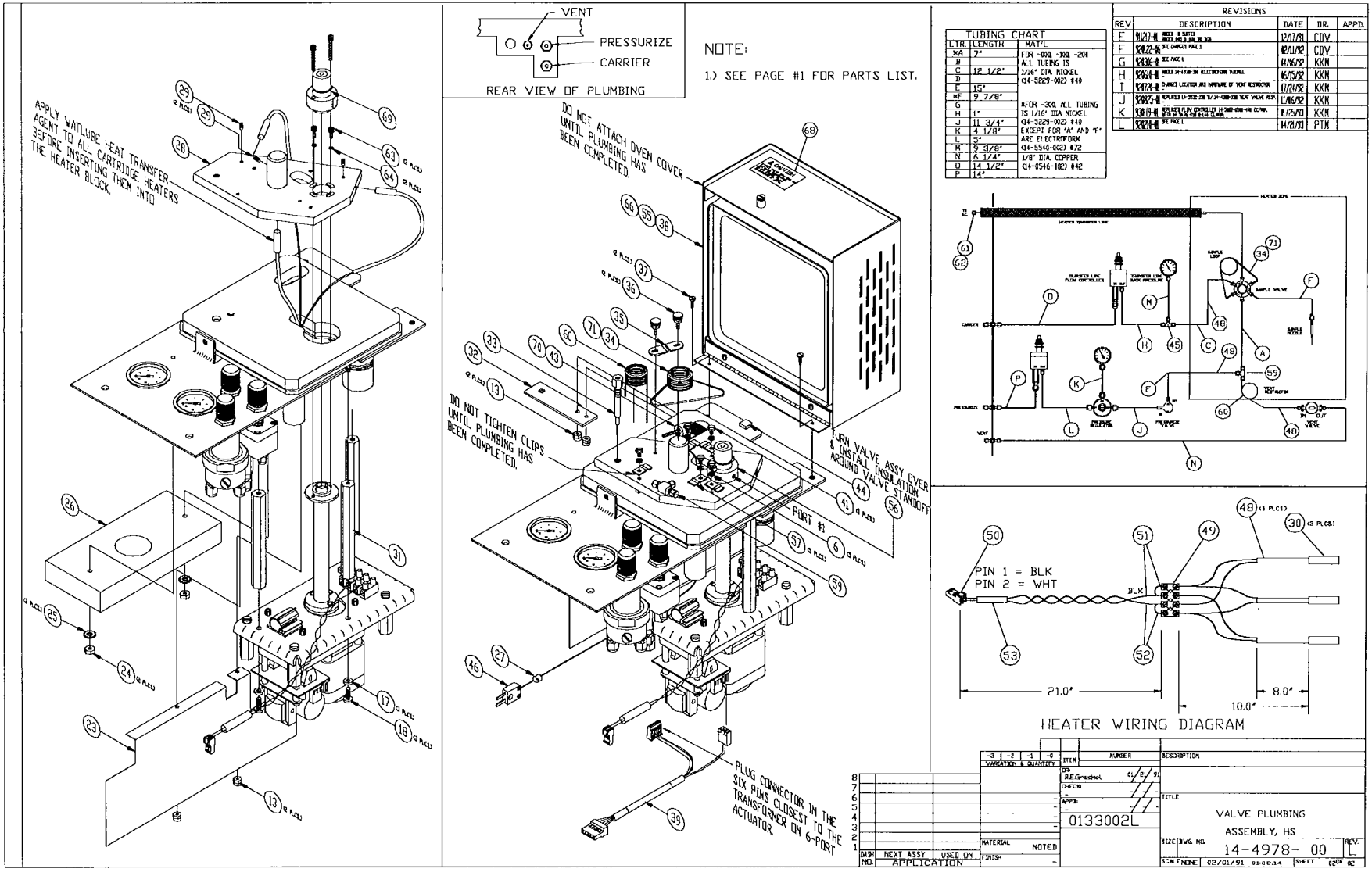
DR. M.E.Smallwood 01/23/91
CHECK
APPD
0133001L

TITLE
VALVE PLUMBING
ASSEMBLY, HS

SIZE D DWG NO. 14-4978-00 REV. L
SCALE NONE DO/01/91 01:04:09 SHEET 01 OF 02

7000 Valve Plumbing Assembly page 1

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7000 Valve Plumbing Assembly page 2

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Index

Aborting Runs 10-5
Accessories
  cryofocusing module 4-29 - 4-33
  reviewing configurations 14-1
Accessory Boards
  installing 4-42
Angle of LCD screen, changing 14-2
Applications, of 7000 1-7
  examples 9-28 - 9-35
Automatic Sampler Control 6-2, 8-2, 8-28, 10-2, 10-5, 13-2
Automated Runs, executing 12-9 - 12-10

Cap Crimper 6-9
Card cage, electronics 3-10 - 3-11
Carrier Gas
  connections 4-8 - 4-14
  control by 7000 flow controller 4-8 - 4-9
  control by external pressure regulator 4-11 - 4-13
  control by GC 4-13 - 4-14
  requirements 4-6
Carrousel, 7050
  description 3-8
  installation 4-44 - 4-45
Carrousel Collar
  changing 6-1 - 6-3
Cautions and Warnings 2-1 - 2-3
Cleaning the 7000/7050 14-6
Clock
  resetting 14-3
Column Carrier Flow 5-5
Components of 7000 3-1 - 3-11
Configuration Screen 12-6, 12-7
Constant Heat Time 1-5, 8-1 - 8-5, 9-15
Constant Heat Time Conflict 8-1 - 8-4
Constant Heat Time and Parallel Sample Introduction 8-5
Constant Start Signal
  I/O board settings 4-38 - 4-39
Copying Method Parameters 8-21
Crimping Vials 6-8 - 6-10
Cryofocusing Module
  connections 4-29 - 4-33
Current Configuration
  screen 8-2, 12-6, 12-7, 14-3

Default Methods 7-1
Diagrams, wiring and flow 17-1 - 17-5
Direct Column Connection
  of Transfer Line to GC 4-19
DIP Switch Settings 4-38 - 4-39
Distribution Constant, defined 9-3

Electronics Card Cage 3-10 - 3-11
Electronic Connections - 7000 to GC 4-36 - 4-37
Equilibrium 9-3, 9-13 - 9-15
Error Screens
  Constant Heat Time 8-1
  GC Cycle Time Error 13-6
  power fail - memory kept 13-3
  power fail - memory lost 13-4 - 13-5
  vials in platen 13-1 - 13-2

Failure Screens
  setpoint not reached 13-8
  thermocouple open 13-7
Fatal Failure Screens 13-9 - 13-17
  Motor Failures
    motor: 7050 carrousel index 13-17

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motor: platen (or 7050) 13-17
motor: sample loader, vial loader or mixer 13-12 - 13-15
Positioning Failures
motor: 7050 carrousel 13-11
motor: platen position 13-9 - 13-10
motor: vial loader 13-12
Sensor Failures
motor: 7050 carrousel index 13-16
motor: mixer 13-15
motor: sample loader 13-13
motor: vial loader 13-14
Flow
column carrier 5-5
vial needle 5-6 - 5-7, 5-9
Flow Diagram 17-3
Flow Element
installing 4-10
selecting 4-9
Flow Path 11-2
Flow Rates
and GC cycle times 5-6 - 5-7
setting 5-1 - 5-11
Fractionation 4-17, 4-18
Full Evaporation Technique 9-5 - 9-7

Gas Supply
choosing 4-6
GC
cycle time conflict error 8-4, 13-6
electronic connections 4-36 - 4-39
pneumatic connections to 7000
with 7000 controlling flow 4-8 - 4-9
with GC controlling flow 4-13 - 4-14
Headspace Analysis
fundamentals of 9-2 - 9-3
Headspace Autosampler, 7000
applications 1-7
components 3-1 - 3-11
description 1-5 - 1-6
installing 4-1 - 4-53
specifications 2-4 - 2-5
warnings 2-1 - 2-3
Heated Transfer Line
connections 4-15 - 4-33
Heater Failures
setpoint not reached 13-8
thermocouple open 13-7
Heaters
warning 2-1

I/O Board
accessing 4-37
I/O Cables (spare parts) 16-2 - 16-3
Inject Mode
explained 11-16 - 11-17
screen 11-16
Inject Time 9-20, 9-21
Injection Port Fractionation Error 4-17 - 4-18
Injections Per Vial
multi-puncture 8-10, 8-13 - 8-15
single puncture 8-11 - 8-12
Installation, 7000 4-1 - 4-53
materials needed 4-1 - 4-2
power requirements 4-3

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Keypad
description 3-1 - 3-3
locking 14-4 - 14-5
Keys
Auto 7-2
Copy 7-4
Enter 7-4
Hold 7-2
Load/Unload 7-3
Page Up/Page Down 7-4
Start 7-3
Step 7-3
Leak Checking 4-48 - 4-51
Loading Vials
(with 7050) 6-10, 6-13
(without 7050) 6-11 - 6-12
Loop Fill Time 9-20
Loops
changing 4-46 - 4-47
sizes 2-4

Maintenance
cleaning the 7000 14-6
cleaning the 7050 14-6
Methods
copying method parameters 8-21
default methods 7-1
developing 9-1 - 9-31
Method Optimization Mode™ 8-22 - 8-28
modified methods 7-1
reviewing method parameters 8-6 - 8-7
running a method , steps before 12-1 - 12-4
scheduling (7050 only) 10-1 - 10-4
setting method parameters 8-8 - 8-19
mixer mode 8-9
multiple headspace extraction 8-10 - 8-19
vial sizes 8-20
Method Optimization Mode™ 1-6, 1-7 8-22 - 8-28, 9-23
parameters to optimize 8-22 - 8-28
Microprocessor 7-1 - 7-4
program panel 7-2 - 7-4
ROM, replacing 4-40 - 4-41
self tests 12-5 - 12-6
Mixing
guidelines 9-15 - 9-16
parameters 9-15
Modes
inject 11-16 - 11-17
leak check 4-48 - 4-51
loop equilibrate time 11-14 - 11-15
loop fill 11-13
method optimization 8-22 - 8-28
mixing 11-9
platen equilibrate 11-6
pressure equilibrate time 11-12
pressurize 11-11
re-equilibrate 8-19, 11-17 - 11-18
sample equilibrate 11-7 - 11-8
stabilize 11-10
standard and concentrate 8-16 - 8-18
standby 11-4
tracking 8-27, 8-28, 9-24
vent check 4-52 - 4-53

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Motor Test Mode 15-3
Multiple Headspace Extraction 8-10 - 8-19, 9-9
multi-puncture 8-10, 8-14
re-equilibrate mode 8-19
single puncture 8-11 - 8-12
standard and concentrate modes 8-13, 8-16 - 8-18

Operation Sequence 11-1 - 11-20
Optimization of Headspace Sensitivity 9-11 - 9-27
Optimix™ 1-7
Outputs
verifying 15-4

Packed Inlet/Packed Column Operation 4-24
Packed Inlet/Megabore Column Operation 4-24 - 4-25
Parallel Sample Introduction 8-5
Parameter Optimization Kit 9-7
Parameters
Constant Heat Time 8-1 - 8-5
copying 8-21
examples 9-24 - 9-31
optimization 8-22 - 8-28
reviewing values 8-6 - 8-7
setting new values 8-8 - 8-20
vial sizes 8-20
Partition Coefficient 9-3, 9-8, 9-11
Phase Ratio 9-3, 9-8, 9-11
Platen
description 3-9
preparing for 9 and 12 ml vials 6-1 - 6-3
preparing for 22 ml vials 6-4 - 6-5
removing shipping inserts 4-4
specifications 2-4
Plumbing Assembly 3-6
Pneumatic Connections 4-5 - 4-31
Pneumatic Controls, overview 3-6 - 3-7
Pneumatics, setting 5-1 - 5-11
Power Fail 13-3 - 13-5
Power Requirements 4-3
Pressurization Gas
connection 4-7
requirements 4-6
Priority Resolution Form 15-18
Priority Sampling 10-5
Problems and Solutions (Troubleshooting)
actuator (6-port) 15-13
carrousel and platen failures 15-16 - 15-17
carryover 15-7
elevator assembly failures 15-14 - 15-15
no peaks and/or sensitivity 15-9
no response from the 7000 15-5
reduced reponse from the 7000 15-6
poor peak shape and/or tailing 15-8
Program Panel 3-1 - 3-3
Program Steps 9-10 - 9-31

Quantitation of Headspace 9-8 - 9-9

Rear Panel, description 3-4 - 3-5
Re-Equilibration 1-7
Replacement Parts 16-2 - 16-8
Reviewing Method Parameter Values 8-6 - 8-7

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ROM chip
replacing 4-40 - 4-42
RS232 Interface Option 4-43

Safety Considerations 2-1 - 2-3
Sample Loops, Changing 4-46 - 4-47
Sample Needle, Changing 4-46 - 4-47
Sample Platen
description 3-9
specifications 2-4
Sample Submission Form 15-18
Sample Temperature 9-5, 9-11, 9-12
Sample Vials
loading (with 7050) 6-13
loading (w/out 7050) 6-11 - 6-12
sizes 2-4, 6-1
unloading (with 7050) 6-15
unloading (w/out 7050) 6-14
Sampling
sequence 11-2 - 11-9
Scheduling Methods 10-1 - 10-4
Screen Viewing Angle 14-2
Screens
automatic sampler control 6-2, 8-2, 8-28, 10-2, 10-5, 13-2
abort 10-5
clock 14-3
Constant Heat Time error 8-1, 11-15, 13-6
copying parameters 8-21
current configuration 8-2, 12-6, 12-7, 14-3
diagnostics 15-2
error
GC Cycle Time conflict 13-6
vials in platen 13-1 - 13-2
failure 13-7 - 13-8
fatal failure 13-9 - 13-19
GC Cycle Time Conflict error 13-6
inject 11-16
leak check 4-48 - 4-51
loop equilibrate time 11-14
loop fill 11-13
method parameters, copying 8-21
method scheduling 10-2
mixing 11-9
motor test mode 15-3
pressure equilibrate time 11-12
pressurize 11-11
re-equil mode 11-18
sample equilibrate 11-7
sample positioning 11-6
self test 12-5
sensor 15-2
sequence of in a method 11-3 - 11-18
setpoint not reached 13-8
stabilize 11-10
standby 11-4
thermocouple open screen 13-7
troubleshooting 15-2
vent check 4-52
Self Tests
routine 12-5
running 12-5
screens 12-5
skipping 12-6
Sensitivity, Optimizing 9-11 - 9-27
Sensor Screen 15-2
Septum Needle Adapter
connection method to GC 4-20 - 4-26
Septum Outgassing 4-17

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Septum Purge Flow 4-17
Sequence of Operation, 7000
11-2 - 11-19
Service Calls 16-1
Setting New Method Parameter Values
8-1 - 8-28
injections per vial 8-10 - 8-19
Setpoint Not Reached - Failure 13-8
Setting Pneumatics 5-1 - 5-11
Shipping Inserts
removing 4-4
Spare Parts List 16-2 - 16-8
Specifications 2-4 - 2-5
Split Flow 4-16 - 4-18
Split Ratio 4-17 - 4-18
Splitless Capillary Operation 4-16,
4-21 - 4-22
Standard Inlet Line Connection
of Transfer Line to GC 4-16 - 4-18
Standby
mode 11-4
screen 11-4
Standby Vial Needle Flow Rate 5-11
Start-up
steps before beginning 12-2 - 12-4
Static Vial Pressure 5-8 - 5-10, 9-3,
9-17 - 9-18
Step
command in installing platen inserts 6-2
Steps of Operation
Inject 11-16 - 11-17
Loop Equilibrate 11-14 - 11-15
Loop Fill 11-13
Mixing 11-9
Platen Equilibrate 11-6
Pressure Equilibrate 11-12
Pressurize 11-11
Re-Equilibrate 11-17
Sample Equilibrate 11-7 - 11-8
Stabilize 11-10
Standby 11-4 - 11-5

TekLink™ Software/Hardware 1-6, 4-43
Temperature
effect on sample 9-5
optimizing 9-24
Thermocouple Open Screen 13-7
Tracking, Feature in MOM 8-27 - 8-28,
9-23
Transfer Line Connection Methods
Cryofocusing Module 4-29 - 4-33
direct column 4-19
low volume injector 4-26 - 4-28
Septum Needle Adapter 4-20 - 4-25
standard inlet line 4-16 - 4-18
Troubleshooting
flow charts 15-5 - 15-17
forms 15-18
instructions 15-1 - 15-18

Unloading Vials
with 7050 6-15
without 7050 6-14
Unscheduled Automated Runs 12-9 -
12-10

Valve Configurations (in following modes):
Equilibration 11-7
Inject 11-17
Loop Equilibrate 11-14
Loop Fill 11-13
Mixing 11-9
Pressure Equilibrate 11-12

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Pressurize 11-11
Stabilize 11-10
Valve Output Chart 11-20
Valve Oven 3-7
Variable Injection Pressure Regulator
(VIPR) 4-34 - 4-35, 5-11, 9-4, 9-21
Vent
check 4-52 - 4-53
Vent Outlet Connection 4-5
Vial Flow Rate 9-19
Vial Needle Flow 5-6 - 5-7
Vial Pressurization Gas
connections 4-7
values 9-17 - 9-21
Vial Pressurize Time 9-19
Vials
capping and sealing 6-8 - 6-10
loading into 7000 with 7050 6-10
loading into 7000 without 7050
6-11 - 6-12
preparing the 7000 for 6-1 - 6-7
programming sizes 8-20
sizes 2-4, 6-1
unloading (with 7050) 6-15
unloading (without 7050) 6-14
Voltage Requirements 4-3

Warnings 2-1 - 2-3
Wiring Diagram 17-1

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Glossary

The following technical terms and phrases appear in the 7000/7050 instruction manual and are further defined in this glossary.

A

Aliquot - A small sample representative of a larger quantity.

Area counts - A measurement (height and width) of the quantity of sample component.

C

Capillary column - An open (unpacked) tubular or fused silica column.

Carrier gas - The gas (usually helium or nitrogen) used to transport the sample through the system.

Concentrate mode - A mode available when using Multiple Headspace Extraction. Concentrate mode is used to collect multiple samples from a single vial using a Cryo-focusing Module or subambient oven. It then allows for this collection of samples to be injected on the analytical column for analysis. In this mode, the GC runs only on the last injection.

Column - Separates the sample components.

Constant heat time - A feature of the 7000 Headspace Autosampler that permits identical thermal exposure times for each sample.

Constant heat time mandatory - A parameter to set on the 7000 that causes the 7000 to check for a ready signal from the GC before injection. It looks for the ready signal during Loop Equilibrate Mode.

Cryofocusing - A technique used to increase sensitivity of the method by cryogenically trapping the substances contained in the equilibrium gas before introducing them to the gas chromatograph.

D

Distribution constant - A constant that describes the equilibrium considerations for a particular analyte/matrix combination at a given temperature.

E

Equilibrium - The state at which the diffusion rate of sample entering the vapor phase and the vapor re-entering the sample are equal. When the concentration of analytes in the gas phase is constant, equilibrium is achieved.

F

Full evaporation technique - Complete transfer of analytes from a liquid or solid matrix into the vapor phase.

G

Gas chromatography - A separation method achieved by distributing substances between two different phases -- a mobile phase and a stationary phase.

GC cycle time - A value equal to GC run time plus cooldown time.

H

Headspace - The vial's gas phase.

Headspace sampling - A technique that removes volatile compounds from a gaseous matrix (the vial atmosphere).

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I

Injection - Transfer of the analytes from the headspace autosampler to the analyzer (gas chromatograph or mass spectrometer).

Injection port fractionation - A phenomenon that occurs when a sample is already in the vapor phase as it is introduced to the split capillary inlet (from the carrier inlet line) and significant calibration errors result from fractionation.

L

Lability - Stability.

Linearity - The ability of the detector to accurately show consistency in peak height when the same concentrations of a certain compound are injected.

M

Matrix - The combination, or mixture, of components within the sample.

Megabore column - A column of about .53 mm in diameter. Also called wide bore. Commonly used as a packed column replacement, direct injections, and for applications requiring high carrier gas flow rates.

Method - The procedure, or sequence of steps and parameters, used in an analytical sampling run.

Method Scheduling - A feature on the 7000 that allows a single vial or as many as 16 sets of vials to be run in up to four different programmed methods.

Monomer - A simple molecule capable of combining with a number of like or unlike molecules to form a polymer.

Multiple headspace extraction - A technique that uses multiple analyses of one sample to determine the partition coefficient or to detect low concentrations of compounds.

Multipuncture mode - A selectable mode on the 7000 Headspace Autosampler that results in more than one puncture of the sample vial septa. For use with Multiple Headspace Extraction.

N

Narrow bore column - A column of about .25 mm in diameter. Commonly used for split injections, and for high resolution gas chromatography and mass spectrometry.

O

Outgassing (septum) - Development of organic compounds from the septum material that can be analyzed.

P

Parameter - A variable (such as time, temperature, mix time, etc.) of a headspace method.

Partition coefficient (K) - The ratio of concentrations (or solubilities) of an analyte between the sample matrix and the solvent ($K = C_M/C_G$).

Phase ratio - The ratio of volume of vapor space in the vial to the volume of sample (V_G/V_L).

Platen - A solid block, electrically-heated device that serves as a stand-alone 12-position autosampler on the 7000. It can accommodate 9, 12, and 22 ml vials.

Pneumatic controls - Controls located on the top of the 7000 that allow for control of vial needle flow, transfer line flow, vial pressurization.

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Polymer - A substance made of large molecules formed by the union of simple molecules (monomers).

Pressurization gas - Gas (usually helium) used to pressurize the sample vial and create the conditions within the vial necessary to cause the analytes to reach the equilibrium state.

Q

Quantitation - Analysis of gas, liquid, or solid samples to determine the precise concentration of certain compounds.

S

Sensitivity - A factor in headspace analysis that increases the ability to detect trace volatile compounds. Sensitivity is increased through highly efficient columns, selective detection, cryogenics, low partition coefficients, and other techniques.

Signal-to-noise ratio - The ratio of the amplitude of a desired signal at any point to the amplitude of noise signals at that same point.

Single puncture mode - A selectable mode on the 7000 Headspace Autosampler that results in only one puncture of the sample vial septa but one or more samplings as programmed through Multiple Headspace Extraction.

Split ratio - The ratio of the amount of sample that travels down the column to the detector and the amount of sample that is expelled from the split vent.

Standard mode - This mode injects the sample onto the GC column and vents the vial after each sampling.

Static headspace analysis- An analytical technique whereby the extraction process consists of removing analytes from a gas matrix.

Static vial pressure - The pressure in the vial generated by the sample, before extra pressurization is added.

T

TekLink 7000 - A Microsoft® Windows™ (3.1 or greater) based instrument control software that interconnects one or more 7000s to a PC. The gas chromatograph, data collection system, and Tekmar instrument can be controlled from a single station.

V

Vial Atmosphere Purge - A parameter to set on the 7000 that enables the unit to vent (or displace) the headspace out of the vial using inert pressurization gas. This is helpful with samples that show reactivity when exposed to air during equilibration.

VIPR - The Variable Injection Pressure Regulator is a backpressure regulator module used to control the injection pressure of the headspace sample during the 7000's Loop Fill Mode. The VIPR offers more precise control of injection pressure in constant volume injections.

Volatile organics - Substances capable of readily changing to a vapor phase under certain temperature conditions.

Volatility - Solubility of the gas.

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