Attention 7000HT – 220Volt Customers

For the 230V unit, an external transformer is required to step-down the voltage to 115 VAC. The maximum current draw is 4 amps to the input of the step-down transformer. Eight amps is the maximum output of the external step-down transformer supplying the 7000HT unit.

Teledyne Tekmar 7000*HT*

High Temperature Headspace Autosampler

Updates
Teledyne Tekmar may update the information contained in this booklet without notice to purchaser.

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Document Part Number: 14-4400-H74 • Rev. B • 08.26.03

Introduction to the Tekmar 7000*HT* (High Temperature) Headspace Autosampler

The 7000*HT* consists of several new advantages, features, and applications for high temperature headspace analysis:

Advantages

- Ability to extend heating range for compounds that exceed boiling points of 200°C
- Highly automated
- Reproducible precision
- Improved sensitivity

Features

- 300°C platen temperature*
- Silcosteel sample pathway
- Standard 7000/7050 features

* *Requires the use of specialized high temperature vials*

Applications

- Polymers
- Chemical/Petrochemical
- Food and Flavors
- Packaging

Please take some time to review this addendum to/and the *7000 Headspace Autosampler User Manual* (p/n 14-4333-000). All section numbers and content in this 7000*HT* addendum directly append the very same section numbers and content contained within the *7000 Headpsace Autosampler User Manual* (p/n 14-4333-000) unless otherwise noted.

2.2 Warnings

(pp. 2-1 — 2-3)

DANGER!

Never connect a flammable gas (such as hydrogen) to the pressurize bulkhead fitting on the rear of the 7000*HT*. 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*HT*. 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*HT*, 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.

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*HT* 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. Vial pressurization or static vial pressurization should not exceed 45 psi.

WARNING!

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

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.

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*HT* power off before connecting or disconnecting the 7000*HT*/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.

CAUTION!

High temperature vials (Tekmar HT) are required for running samples >200°C.

CAUTION!

Sample loop temperature must be at 300°C for 15 minutes before operating unit! Cycle 6-port valve 10 times and let cool to operating temperature. Failure to do so will damage theT slider and void warranty!

2.3 Specifications
(pp., 2-4, 2-5)

7000 HT Headspace Autosampler Specifications:

Capacity:	12 vials simultaneously heated, sampled via stationary needle design
Column Interface:	Standard side-port connect for packed and capillary inlets. Optional: Septum Needle Adaptor, Low Volume Insert, Cryofocusing Module
Interface Capability:	Capable of interfacing to virtually all commercially available gas chromatographs, equipped with packed, widebore or capillary columns
Vial Size:	22mL (High Temperature Vials *must* be used for applications above 200°C; Configurable 9, 12, and 22 mL standard vials for applications below 200°C)
Sample Loops:	Interchangeable 0.5, 1.0, and 2.0 mL Silcosteel™ loops
Heated Sample Platen:	Oil-free, resistance heated, jacketed, variable temperature (ambient +15°C to 300°C), settable in 1°C increments
Valving:	Motor-actuated 6-port valve, variable temperature (ambient +15°C to 300°C), 2 solenoid operated 2-port valves for sample pressurization and sample vent
Sample Path:	Teflon-free continuously swept 1/16” Silcosteel transfer line, variable temperature (ambient +15°C to 300°C), Silcosteel sample loops
Sample Mixing:	OPTIMIX Equilibration System, time settable 0.1 to 999.9 minutes, power setting in 9 increments

Modes of Operation:

Manual:	Single sample.
Standard:	Multiple samples, minimum time for equilibrium, successive samples are heated for long periods.
CHT:	Constant Heat Time, identical thermal exposure and preheating time for each sample (requires 7050).
MHE:	Multiple Headspace Extraction, up to 9 extraction steps per sample with intermediate vent, with single or multiple puncture, with Standard or Concentrate Mode.
MOM:	Successive Samples receive incremental changes in method parameter setpoints for time, temperature, or mixing power (requires 7050 Carrousel) with selectable number of times each increment is run.

Methods:	4 Methods with scheduling feature for grouping up to 16 different sets of samples in the 7050 Carrousel
Electronics Control:	Parameter entry via a front panel, self-contained microprocessor and software
Display:	64 x 240 pixel graphics LCD screen
Interface:	Card cage mounted: optional two-way instrument interface, BCD, RS232 serial communication; baud rate selectable 150 to 19200
Size:	20”W x 22”H x 13”D
Weight:	79 lbs.
Utilities:	110V 50/60 Hz, 10A, optional external transformer for additional voltage requirements, carrier/pressurization gas

7050 Carrousel Specifications:

Operational Capacity:	50 Sample vials with Constant Heat Time, Method Scheduling and Standard Tekmar 7000 operating modes
Not Heated:	All vials are held at ambient temperature, shielded with a protective cover
Size:	20”W x 9”H x 17”D
Weight:	30 lbs.

Tekmar Part Numbers:

7000HT Headspace, 115V	14-4400-00H
7000HT Headpace (Base Unit), 115V, with external transformer, for 230V	14-4400-10H
7000HT Headpace (Base Unit), 100V	14-4400-20H
7050 Headspace Carrousel	14-4401-000
7000/7050 HT Headspace Autosampler (Base + Carrousel), 115V	14-4402-00H
7000/7050 HT Headspace Autosampler (Base + Carrousel) 115V, with external transformer, for 230V	14-4402-10H
7000/7050 HT Headspace Autosampler (Base + Carrousel), 200V	14-4402-20H

Specifications subject to change without notice

4.2 Preliminary Procedures

(pp., 4-1, 4-2)

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	Tekmar Part Number
Cord, Power, 115V, 15A	14-1197-039
Cap (crimp), Aluminum 20 mm Neck (pk of 125)	14-4436-000
Septa, Silicon (pk of 125)	14-5818-043
Backing, foil, 002” (for septa)	14-7418-080
Vials, Headspace, with 22 mL top, for high temperature use (pk of 125)	14-4440-324
Nut, Plug, Brass, 1/16"	14-2792-016
Fuse, 2 AGC	14-0140-034
Fuse, 8 AMP Rectifier	14-3043-034
Fuse, 10A, 250V, 5 x 20 mm, slow blow	14-5180-034

Installation Kit Contents	Tekmar Part Number
Assy, Trap, HC SGE	14-1362-000
Tubing, 200 Nickel, .064-.060 OD, .042-.038 ID	14-5229-002
Tubing, 1/8”, copper	14-0546-002
Reducer, 1/16" to 1/8", Tube Stub, Brass	12-0042-016
Tee, Brass, 1/8"	12-0070-016
Union, Tube, Brass, 1/8"	12-0073-016
Union, SS, 1/16" - 1/16"	14-0051-016
Tool, Wrench, 1/4 - 5/16 Open End	14-4845-027
Tool, Wrench, 7/16 - 1/2 Open End	14-4846-027
Tool, Screwdriver, Slot Stubby	14-4847-027
Tool, Screwdriver, Phillips #2, 4” long	14-5013-027
Tool, Wrench, 3/8" Single, Open End	14-5014-027
Tool, Cap Crimper, 20 mm Neck	14-4863-027
Valve, Element, Flow 40-400 cc/min	14-4928-050

Other Materials Needed for Installation

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

Power Requirements
(pp., 4-1, 4-2)

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, an external transformer is required to stepdown the voltage to 115VAC. The maximum current draw is 4 amps to the input of the stepdown transformer. Eight amps is the maximum output of the external stepdown transformer supplying the 7000*HT* unit. For the 100V unit, the maximum current draw is 10 amps and maximum power consumption is 1,000 watts (when accessories are included).

TekLink™ Option
(p. 4-43)

TekLink is a software program offered by Tekmar which allows you to automatically schedule and monitor up to four 7000 or 7000*HT* 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.

Note: The 7000*HT* TekLink 2.0 software is compatible with the 7000*HT* and current 7000. TekLink versions prior to 2.0 are not compatible with the 7000*HT*.

Some of the benefits of TekLink include:

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

Note: For more information using TekLink with the 7000*HT* or 7000, please refer to the *TekLink 7000 User's Manual.*

IMPORTANT!
PLEASE READ!

The following operational procedures are specific to the 7000 HT (high temperature) Headspace Autosampler and do not apply, in any way, to the 7000 Headspace Autosampler.

HT.1 Preparing Tekmar 22mL HT (High Temperature) Vials for Sampling

HT-1.1 Introduction to preparing vials for sampling:

Some factors that can 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. Teledyne Tekmar recommends that you use an adjustable cap crimping tool to allow for these variations (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.

Teledyne Tekmar recommends the Crimp Station (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-Dohrmann at (800) 543-4461 or (513) 247-7000 locally and outside the U.S.

HT-1.2 To prepare vials for sampling:

1. Place the cap on a firm surface.
2. Insert the foil backing followed by the septum into the cap (avoid contamination of the septum face — the shiny Teflon face should always be face down toward the vial.)
3. Place the septum assembly 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.

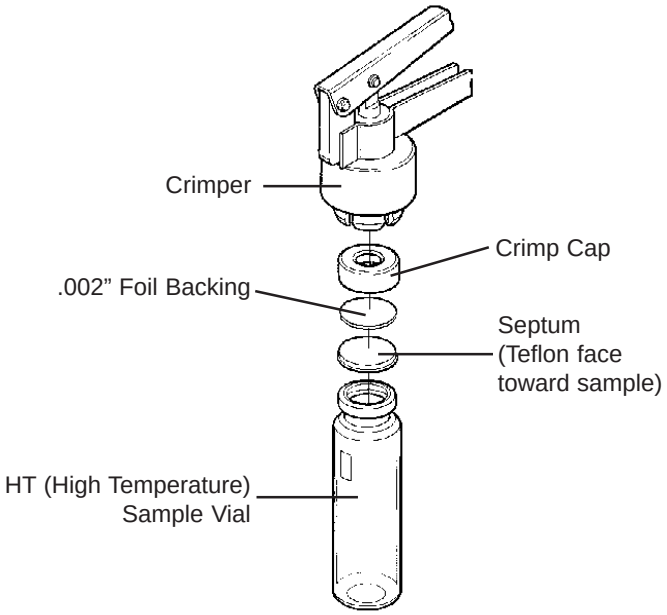

Figure HT-4 Hand Crimper and Vial

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

HT-1.3 Inspecting Crimped Sample Vials

A properly capped and sealed vial (see Figure HT-2) is critical in maintaining sample integrity during headspace autosampling. Following are guidelines for proper high temperature vial preparation.

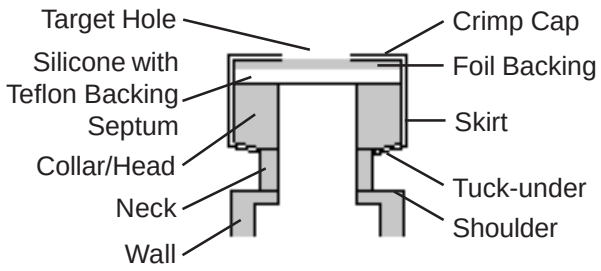

Figure HT-2
Properly Sealed Vial

When a vial is undercrimped (see Figure HT-3), 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.

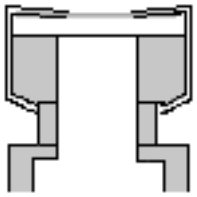

Figure HT-3
Undercrimped Vial

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 (see Figure HT-4), the top surface of the septa assembly 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.

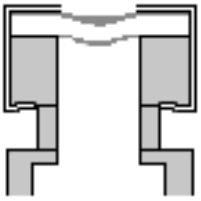

Figure HT-4
Overcrimped Vial

HT.2 Accessing, Cleaning, Reassembling, and Conditioning the 7000*HT* 6-Port High Temperature Valve and Rotor

6-port High Temperature Valve and Rotor Conditioning Information

The Valco® 6-port Valve Rotor used with the 7000*HT* Headspace Autosampler is made of a polyimide/ PTFE/carbon composite and has been used successfully for many years and still cannot be surpassed when applications demand operating temperatures in the 250°C - 350°C range. (Standard specs for most series are 300 psi at 300°C.) However, when running at temperatures below 150°C there is a tendency for the seal material to stick to the valve body, making the valve difficult to turn and causing the rotor to crack in extreme cases. The "T" material is susceptible to attack from steam, ammonia, hydrazines (anhydrous liquids or vapor), primary and secondary amines, and solutions having a pH of 10 or more. Chemical reagents which act as powerful oxidizing agents (nitric acid, nitrogen tetroxide, etc.) must also be avoided. Although the Tekmar7000*HT* is designed for high temperature applications, it can also be used for methods requiring sample loop setpoints of 200°C or less. Please refer to the table below to determine whether or not the 7000*HT* 6-port valve must be conditioned.

The 7000*HT* 6-port Valve Must Be Conditioned If:

- initial power up of the 7000*HT,*
- the 7000*HT* is turned off and the sample loop has cooled to a temperature below 200°C, or
- if during a two week time period (or longer), the 7000*HT* is run continuously with the sample loop at below 200°C.

Conditioning the 7000*HT* 6-port Valve is Unnecessary If:

- applications run alternately between < 200°C and 200 -300°C within a two week time period,
- applications run continuously at 200°C or higher
- the rotor is new and has never been run above 200°C, or
- a new rotor is installed and will not be run above 200°C (this is desirable for lengthy, low temperature applications.

CAUTION!

Perform all other system checks before working on the 6-port valve. Since any contact between the interior of the valve body and the metal of the rotor is likely to cause damage, do not take the valve apart unless system malfunction is definitely isolated to the valve. If valve disassembly is absolutley necessary, carefully observe the instructions listed below for disassembly, cleaning, and reassembly.

NOTE: Perform all disassembly, cleaning, and reassembly procedures in a clean, well-lighted area. Flush all hazardous or toxic materials from the valve before starting.

NOTE: The entire valve body does not have to be removed to perform the rotor cleaning and replacement procedures.

Recommended Tools

Small magnet (a pencil-type magnet works well)
Pair of hemostats or small needle-nosed pliers

HT-2.1 Conditioning Procedure for High Temperature Valves

1. Power up the 7000*HT* and allow the unit to run through Self Test.
2. In Standby mode, press F1 on the unit keypad.
3. Select Method #1.
4. Press F3 (Edit).
5. Press Page Down 3 times until you reach the Sample Loop Method Edit screen.
6. Press Enter.
7. Enter a temperature value of 300ºC in the Sample Loop temperature field.
8. Press Enter to accept the change.
9. Press Exit.
10. Wait until the sample loop reaches the 300ºC setpoint and equilibrate for 15 minutes (while in Standby).
11. While in Standby, press Hold and then 3.
12. Select #4 (Outputs).
13. Press Page Down 3 times until you reach the 6-Port Output screen.
14. At this point, you must cycle the valve 10 times to seat the sealing surfaces. To actuate the valve:
    a) press the 7/Y (On) button once,
    b) press the 9/N (Off) button once,
    c) repeat steps 14a and 14b four more times.
15. Press F4 (Exit) twice to return to Standby.
16. Repeat steps 1 through 9 to reset Sample Loop to desired temperature and proceed with next application.

HT-2.2 Accessing and Removing the Rotor

1. To avoid a burn, power down the 7000*HT* and let unit cool.
2. Turn off the compressed gas supply to the 7000*HT*.
3. Unscrew the knurled preload assembly from the valve body (Figure HT-6). Do not tamper with the preset socket adjustment screw
4. Engage the end of the rotor (Figure HT-7) with a magnet and gently wiggle the rotor to break the "shear seal" between the rotor and interior valve body until it can be lifted out easily.

Note: If additional force is needed to remove the rotor, use a small flatblade screwdriver to gently loosen the rotor from its seat.

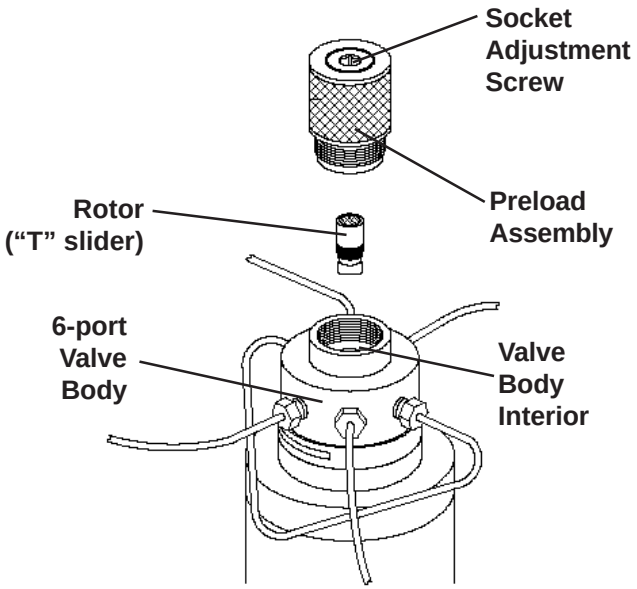

Figure HT-6 Removing the 6-port Valve Rotor

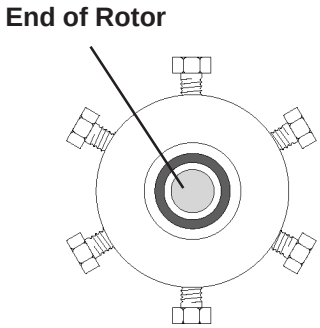

Figure HT-7 Valve with Preload Removed

CAUTION!

Any contact between the interior of the valve body and the metal of the rotor or any tool used is likely to cause damage. Do not force or pry the rotor out. Do not attempt to scrape the interior valve body walls with any sharp instruments — doing so may scar the wall and make the valve susceptible to leaking.

5. Once the rotor is removed from the valve body, note the orientation of the rotor tab, which is marked with the letter "T" (Figure HT-8).

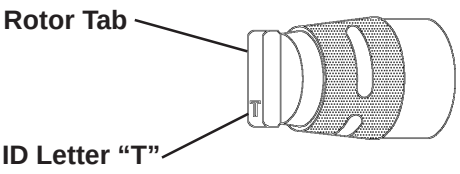

Figure HT-8 Location and Orientation of the "T" ID Letter

HT-2.3 Cleaning the Valve Body

1. Wet a cotton swab or an optical-quality lint free wiper (like Kimwipe®) with a solvent which is compatible with the chromatographic system. Isopropyl alcohol is recommended.

NOTE: If the valve has been used with aqueous buffer solutions and some leakage has occurred, wipe the sealing surfaces of the valve with a water-moistened Kimwipe before using a nonpolar solvent to clean any seal material still adhering to the valve's interior.

2. Gently swab the polished interior of the valve to remove any loose residue. Wipe away any loosely bound seal material which may have worn free and adhered to either surface. Avoid using hydrocarbon solvents if the valve is to be used in a system with electron capture detection, since some of the solvents may persist at the trace level. Consult the Manufacturer's Data Safety sheet for whatever solvent is utilized.
3. Blow with clean compressed gas to remove any lint left by the swab.
4. Visually inspect the interior of the valve body. The conical surface should appear highly polished. If any scratches are visible between the ports or anywhere which might suggest a potential leakage path or wear source, the valve should be replaced.

HT-2.4 Cleaning the Rotor

1. Carefully grasp the rotor on either end and briefly immerse it in solvent. If it is difficult to grip the roto securely, hemostats or needle-nosed pliers may be helpful (Figure HT-9). Grip the tab end, being careful not to mar the metal or touch the polymer.
2. Gently wipe the polymer with a clean tissue.
3. Blow with clean compressed gas to remove any lint left by the tissue.
4. Visually inspect the rotor. If it shows any scratches and/or a narrowing of the flow passages, replacement is necessary.

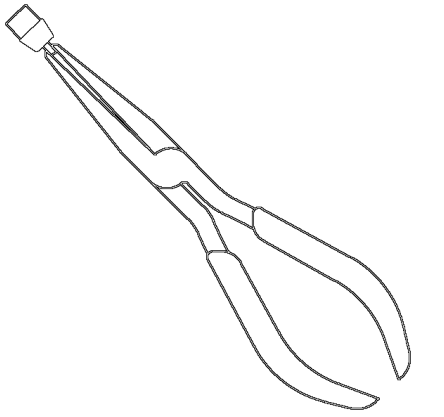

Figure HT-9 Holding the Rotor

HT-2.5 Assembling a New or Previously Used Rotor

1. Place the clean rotor on the pencil magnet and orient it so that tha tab will properly engage the slot of the drive mechanism (Figure HT-10).
2. Insert the rotor into the valve body, again being careful that the tab does not touch the polished interior of the valve body. Make sure that the rotor tab is fully inserted into the slot in the driver. Using a pencil or other small pointed instrument, hold the rotor in place in the valve body while the magnet is pulled free.
3. Make sure that the rotor is oriented correctly within the driver. The “T” on the rotor tab must be directly between ports 3 and 4.
4. Replace the knurled preload assembly, tightening it into the valve body by hand just beyond the point where it touches the rotor.
5. Turn on the gas supply and test the transfer line for flow.

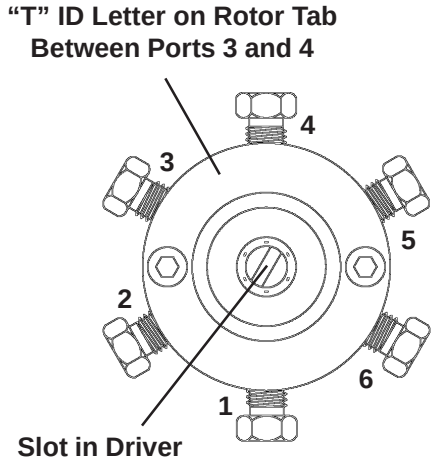

Figure HT-10 Proper Orientation of Rotor ID Letter

NOTE: The HT valve needs to be conditioned whenever the rotor is replaced. Section HT-2.1 explains how to perform the valve conditioning procedure.

HT.3 Replacement Parts and Accessories

CONSUMABLE ITEMS FOR THE 7000*HT* ONLY

SEPTA

14-7418-080	Backing, foil, .002”

VIALS

14-4440-324	22mL, headspace, w/ 20mm top, for high temperature use (125 per pack)

ELECTRONICS

14-1197-039	Power Cord, 15A, 115V
14-3151-038	Transformer, stepdown, 230V/115V, external
14-4872-275	ROM, with program

TEKLINK 7000

14-5731-076	TekLink 7000 Instrument Control Software. Includes: TekLink 7000 software, (1) 3.5” diskette, 12 ft. (9 pin to 9 pin) RS232 cable, 9 pin to 25 pin cable adapter, and instruction manual.

HEATERS

14-3581-120	Cartridge heater, 1/4” x 1 1/4” (for sample loop and valve oven)
14-4311-220	Assembly cartridge heater, 7000 high temperature (for platen)
14-4572-600	Transfer line, heater, 60” with Silco tubing .04 ID

MISCELLANEOUS

14-4983-253	Needle, Silcosteel coated .040 ID

VALVES AND PNEUMATICS

14-2658-050	Valve rotor, 6-port, T-type
14-7439-000	Pressure gauge assembly, 0-60psi, 1/16” tube

LOOPS

14-4924-267	Sample, 1 mL, Silcosteel coated
14-4919-267	Sample, 2 mL, Silcosteel coated