Thermo Finnigan

TRACE GC

HS 2000 Head Space Autosampler

User's Manual

Published by Technical Publications, Thermo Finnigan Italia S.p.A Strada Rivoltana 20090 Rodano-Milan Italy

Printing History: Revision D printed March 2002.

*Xcalibur*TM and *TRACE* GCTM are trademarks and/or product names of Finnigan Corporation. Microsoft[®] is a registered trademark of *Microsoft Corporation*.

Technical information contained in this publication is for reference purposes only and is subject to change without notice. Every effort has been made to supply complete and accurate information; however, Thermo Finnigan Corporation assumes no responsibility and will not be liable for any errors, omissions, damage, or loss that might result from any use of this manual or the information contained therein (even if this information is properly followed and problems still arise).

This publication is not part of the Agreement of Sale between Thermo Finnigan Corporation and the purchaser of a Thermo Finnigan system. In the event of any conflict between the provisions of this document and those contained in Thermo Finnigan Corporation's Terms and Conditions, the provisions of the Terms and Conditions shall govern. Reference to System Configuration and Specifications supersede all previous information and are subject to change without notice.

The GC/MS and LC/MS products of the Thermo Finnigan Division are produced under VISION 2000 accredited quality management systems.

U.S.A.: Thermo Finnigan Corporation ● 355 River Oaks Parkway ● San Jose, CA 95134-1991 ● [1] (408) 965-6000

Thermo Finnigan Technical Support ● 3661 Interstate Park Road North ● Riviera Beach, FL 33404 ● (800) 685-9535, (561) 844-5241 ● Fax (561) 881-8431

Australia: Thermo Finnigan • P.O. Box 239 Rydalmere • Unit 20, Metro Centre • 38 – 46 South Street • Rydalmere, N.S.W. 2116 • [61] (02) 9898-9000

Austria: Thermo Finnigan GmbH ● Wehlistrasse 27b ● A-1200 Wein ● [43] (01) 333 50 34-0 ● info.quest@thermo.at

Belgium: Thermo Finnigan BVBA ● Groenenbrogerlaan 84 ● B-2610 Wilrijk (Antwerpen) ● [32] (03) 825 0670

Canada: Thermo Finnigan Canada ● 5716 Coopers Avenue, Unit 1 ● Mississauga, Ontario ● L4Z2E8 ● [1] (905) 712-2258

France: Thermo Finnigan France SA ● Parc Hightec Sud ● 12 Avenue des Tropiques ● Z.A. de Courtaboeuf BP141 ● F-91944 Les Ulis Cédex ● [33] (01) 69 18 88 10

Germany: Thermo Finnigan Analytische Systeme GmbH

Boschring 12

D-63329 Egelsbach

(49] (06103) 408 0

Italy: Thermo Finnigan Italia S.p.A. ● Strada Rivoltana ● I-20090 Rodano (Milano) ● [39] (02) 95059 1

Japan: Thermo Finnigan K.K. ● Nishi-Shinjuku Toyokuni Bldg. 3F ● 2-5-8 Hatsudai, Shibuya-ku ● Tokyo 151-0061 ● [81] (03) 3372-3001

Japan: Thermo Finnigan K.K. ● Esaka Grand Building ● 2-3-1 Esaka-cho, Suita City ● Osaka 564-0063 ● [81] (06) 6387-6681

Netherlands: Thermo Finnigan BV ● Druivenstraat 33 ● NL – 4816 KB Breda ● [31] (076) 587 8722

P.R. China: Thermo Finnigan China ● Suite 912-916, Ping An Mansion. ● No. 28, Jin Rong Street ● Xicheng District ● Beijing 100032 ● [86] (010) 6621 0839

Spain: Thermo Finnigan SA ● Acer 30 – 32 ● Edificio Sertram – Planta 2, Modulo 3 ● ES-08038 Barcelona ● [34] (093) 223 0918

Spain: Thermo Finnigan SA ● Avenida de Valdelaparra 27 ● Edificio Alcor – Planta 2a ● ES-28108 Alcobendas (Madrid) ● [34] (091) 657 4930

Sweden: Thermo Finnigan AB • Pyramidbacken 3 • S-141 75 Kungens Kurva (Huddinge) • [46] (08) 680 0101

United Kingdom: Thermo Finnigan Ltd. ● Paradise ● Hemel Hempstead ● Herts HP2 4TG ● [44] (01) 442 233 555

Notes: The country code is enclosed in square brackets []. The city code or area code is enclosed in parenthesis (). For countries other than the U.S.A., when you are dialing from within the specified country, dial the 0 of the city code. For countries other than Italy, when you are dialing from outside the country, do not dial the 0 of the city code.

Copyright© 2002 Thermo Finnigan, a member of the Thermo Electron family of companies. All rights reserved.

Printed in Italy



Contents

Chapter 1 HS2000 Set Up View	5
1.1 Configuration	6
Configuration Screen 6	
1.2 HS 2000 Method Editor	8
Sample Group Boxes	
Incubation Group Boxes	9
Syringe Group Boxes	10
Stand-by Conditions Group Boxes	11
HS 2000 Command Menu	12
1.3 Setting Up HS 2000 Parameters	



Chapter 1 HS2000 Set Up View

This chapter contains informations to configure and to set the HS 2000 Head Space Autosampler Parameters.

This	chapter	contains	these	topics:	

Configuration	6
HS 2000 Method Editor	8
Setting Up HS 2000 Parameters	13



1.1 Configuration

Configuration Screen

Items listed under this heading contain definitions to each control you see listed in this Instrument Configuration window.

H52000 Configuration	×
Communication:	ОК
Serial port: CUM2	Cancel
GC start mode	Help
 Normal O Anticipated 	
C Ctrap norm C Ctrap A-St	
Syringe Syringe volume (ml) 5.0 Firmware version: Firmware version 3.3 or above	

Figure 1-1. HS 2000 Configuration Screen

Communication Group Box

Serial Port This parameter specifies the communication port to be used for the Sampler, it can be through the GC or alternatively can be any COM Port from 1 to 8.

GC Start mode group box

	This parameter specifies the synchronization mode between the Sampler and the GC.
Normal:	This start mode permits to start the GC program when the needle enters the Injector
Anticipated:	This start mode permits to start the GC program when the needle enters the Injector
Ctrap Norm, Ctrap A-St:	The Ctrap norm and Ctrap A-St options are purposely provided for the cold trap synchronism. The meaning of Norm and A-St remains the same as above.



Syringe group box

Syringe volume	This parameter specifies the volume of the syringe in use. This parameter is then used in the HS2000 method editor for maximum volumes check.
Firmware Version	Check the Firmware version 3.3 or above box if your HS 2000 is equipped with the 3.3 or above firmware version.
	Click OK after you are satisfied with your selection. Click Cancel if you would like to exit without saving your changes. Click Help for this page to display.



1.2 HS 2000 Method Editor

The Method Editor screen contains all of the parameters used to set up injection events. Click the links below to find out more about the parameters.

📰 Senza titolo - Instru	ument Setup	_ 🗆 ×
File HS2000 Help		
HS200 Autosampler TRACE GC	HS2000 Sample Sample draw (ml): Enrichment (H): Enrichment (H): Enrichment (H): Coven incubation mode Coven incubation mode Coven incubation mode Coven incubation settings Temperature (*C): 40 Incubation itme (min): Oven incubation settings Temperature (*C): 40 Progressive incub. time (min): Shaker time on (min): Shaker time off (min): Multiple extraction counts: Total GC cycle time (min): 0 Syringe temperature (*C): 40 Filling delay (sec): 0 Filling volume (ml): 0 Shaker time off (min): 0 Filling delay (sec): 0 Stand-by temperatures Oven temperature (*C): 40 Syringe temperature (*C): 40	
Ready		11.

Figure 1-2. HS 2000 Page

Sample Group Boxes

Sample Group Box

This group of controls is used to set parameters for the sample draw and for use with the Multiple Headspace Extraction (MHE) option.

Sample draw (ml) The amount of sample volume to be injected.

Enrichment (#) The number of MHE enrichments to make. Read the HS 2000 operating manual for more detailed information.

Enrichment delay (min) The delay to flush the vial with carrier gas in the MHE technique. See the HS 2000 operating manual for more details.

Speed Group Box

This group of controls contains the parameters to control the speed the sample is drawn into the syringe and the speed it is injected into the inlet.



Filling speed (ml/min)The speed the sample is withdrawn from the headspace into the syringe.Injection speed (ml/min)Speed of the injection in milliliters per minute of the sample into the inlet.
The speed should be at least as fast as the injector flow. For split injections, a
fast speed reduces tailing.Analysis Time group box

The analysis time group box contains.....

Total GC Cycle (min) Set this time for the full cycle time of the GC analysis. At the end of the run observe the amount of time it takes the GC to cool down and then go ready. Add this time to get the GC cycle time. After the first cycle, this time is precisely calculated by the software for subsequent cycles.

Incubation Group Boxes

Oven incubation settings group box

This group of controls sets the parameters for incubating the sample.

Temperature (°C) The temperature to incubate the sample. For any Headspace separation, the appropriate incubation temperature is the most important parameter. Here the temperature is selected from 40 to 150 C in increments of 1 C.

Note: If in HS 2000 Configuration the Firmware version 3.3 or above box has been checked, a check box is displayed as shown below. Click on this check to enable/ disable the control of the temperature.



Incubation time (min)	This box specifies the total incubation time for all sample to be analyzed with the method being used. The time here specified is in minutes (01499 min).
Progressive delta	This box specifies the time added progressively to the incubation time for all sample to be analyzed with the method being used. The time here specified is in minutes (0599 min).
Shaker time on (min)	The number of minutes to shake the sample.
Shaker time off (min)	The amount of time for the shaker to be turned off.



Multiple extraction counts	This box is accessible if Multiple Head-Space extraction mode has been set. Enter here the number of injections and vial headspace extractions that will be carried out from each vial.
	Oven incubation mode
	This box permits to select the three incubation modes supported by the HS 2000:
Constant incubation	It allows the sample to be sequentially conditioned at a programmed temperature with a constant conditioning time.
Progressive incubation	It allows the sample to be sequentially conditioned at a programmed temperature with a conditioning time that increase for each sample according to a programmed additional time (Dt)
	Refer to <i>Progressive delta</i> .
Multiple head-space extraction	It allows automatic multiple extraction steps of head space from the same sample vial repeatedly performing an operation sequence.
	Syringe Group Boxes
	Syringe Group Box
	This group box controls syringe parameters.
Temperature	Since a heated syringe is used to transfer headspace gases from a vial into the injection port of the GC, the temperature of that syringe can be controlled. The range is 40 to 150 C. One degree is the smallest increment.
	Note: If in HS 2000 Configuration the Firmware version 3.3 or above box has been checked, a check box is displayed as shown below. Click on this check to enable/disable the control of the temperature.
	Syringe
Enable pre-filling	Activating this control allows the syringe to enter the vial with the plunger in the top position; therefore, pressurinzing the vial before withdrawing the sample. The syringe enters the vial, which is filled with the sample volume of purge gas. Use this function only if using sample enrichment. When multiple samplings from the same vials are used, the vial remains at constant pressure. Check this box to download the method to the autosampler. The method



downloads to method IS, IP, or IM depending on the HS configuration.

- Enable flush This box enable/disable the pre-filling mode. This function allows the vial to be pressurized before sampling the headspace vapors. This function must be set only in cases of sample enrichment, that is, when the number of samplings from the same vial is higher than 1. The volume used to pressurize the vial is the syringe volume.
- Filling volume (ml) Specify the sample volume to initially pull-up into the syringe. Subsequently, the autosampler pushes the plunger down to the actual sample volume injected.
 - Filling counts (#) Specify the number of sample rinses of the syringe. Enter a number from 1 to 15.
- Filling delay (sec) Specify the delay in seconds the plunger is held at the Sample Volume while pre-filing the syringe.

Injection delay Group Box

The delays are the amount of time the syringe needle resides in the injector before or after the sample is injected.

Pre-injection delay Enter the time in seconds to hold the syringe needle in the inlet before (sec) injecting the sample. A delay of more than a few seconds will preheat the needle.

Post injection delay Enter the time in seconds to hold the syringe in the inlet after injecting the (sec) sample. A short delay may be used if disruption of the injection appears to occur when the syringe is pulled out too early.

Stand-by Conditions Group Boxes

Standby temperatures group box

The parameters in the group box permit to enter the stand-by conditions of HS2000:

Oven temperature (°C) Select a temperature for the incubation oven to stay while the autosampler is not running.

> Note: Note: If in HS 2000 Configuration the Firmware version 3.3 or above box has been checked, a check box is displayed as shown below. Click on this check to enable/disable the control of the temperature.



Stand-by temperatures	
🔽 Oven temperature (°C):	50
Syringe temperature (°C):	50

Syringe temperature (°C)

Select a temperature for the syringe to stay while the autosampler is not running.

Note: If in HS 2000 Configuration the Firmware version 3.3 or above box has been checked, a check box is displayed as shown below. Click on this check to enable/disable the control of the temperature.

- Stand-by temperatures	
🔽 Oven temperature (*C):	50
Syringe temperature (°C):	50

HS 2000 Command Menu

Take your choice in using your mouse to access the autosampler menu commands.

Send Method to This function transfers the analytical method from the memory of the GC to Sampler the autosampler and enables the method. **Get Method from** This function allows to transfer method data from autosampler to GC. Sampler Import command Loads an autosampler method (.as) from an earlier version of GCQ software (2.1 or earlier).



1.3 Setting Up HS 2000 Parameters

For all the incubation modes

- 1. From the Syringe flush group box, specify which flush parameter to use: One Minute or Permanent.
- 2. In the Stand-by temperature group box, specify values for the Oven temperature and the Syringe temperature.
- 3. In the Injection Delay group box, specify values for the Pre-injection delay and the Post-injection delay parameters.
- 4. In the Sample Incubation Oven group box, specify values in these parameters: Temperature, Incubation time and Progressive incubation time, Shaker time on, and the Shaker time off.
- 5. In the Start mode group box, select either Normal or Anticipated.
- 6. In the Speed group box, specify values in these parameters: Filling speed and Injection speed.
- 7. In the Sample group box, specify values in these parameters: Sample, Enrichment, Enrichment delay.
- 8. In the Syringe group box, specify values in these parameters: Temperature, Filling Volume, Filling counts, and Filling delay. Check the Enable pre-filling parameter if using the Multiple Headspace Extraction (M) mode.