



Teknokroma[®]

Lab & Industry Equipment

Professionally Friendly

User Manual



2 *f*[®] Static Head Space Sampler

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DECLARATION OF THE MANUFACTURER

Manufacturer Teknokroma Analítica

Teknokroma is the manufacturer of the device described in this manual, and therefore, responsible for its security, reliability and performance, only if:

- The installation is realized according to the instructions given in this manual.
- Reparations have been made by authorised staff from Teknokroma.
- Local regulations have been fulfilled in the installation of the equipment.
- This device has only been used by trained and qualified personal for the uses described in this manual

Teknokroma informs that a different use of the specified in this manual is a risk for people and for the equipment, because in that case, the protection of the equipment is not completely secured.

CERTIFICATE OF CONFORMITY EC

Manufacturer: Teknokroma Analítica S.A.
Address: Camí de Can Calders, 14
08173-Sant Cugat Del Vallés (Spain)

The equipment, **serial number 563275**, model **SHS 0112** fulfils the following European Directives:

73/23/EEC of Electric Security
89/336/EEC of Electromagnetic Compatibility

Sant Cugat DelVallés, January 2012

Jordi Traveset
Quality Director

To guarantee this compliance, the following norms have been applied:

UNE EN 61010-1: 96
UNE EN 61010-2-10
UNE EN 61326/A1 class B

1 INTRODUCTION

The Head Space Sampling SHS 0112 is an instrument designed to apply the technique of analysis known as Static Headspace, and to heat solid or liquid samples in a closed vial.

Any other use different to this one, may cause a risk for the equipment and for the people who use it.

The use of this equipment implies some previous knowledge about Gas Chromatography and this special sampling technique.

In this manual, some references useful to know the state of the art of this analytical technique are included (see bibliography).

This equipment has a furnace (metal block with holes of different sizes) that has been designed to heat closed vials which inside have a sample or a pattern.

The use of non-closed vials or the pouring of liquids in the holes of the metal block may cause damages in the equipment and might be dangerous for people, in particular in case of flammable liquids.

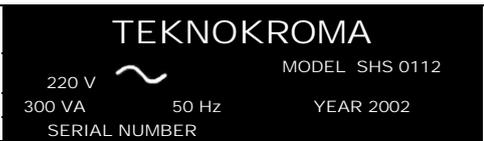
Heating closed vials at a high temperature in relation with the vial contents (solvent boiling temperature) may cause the breakage of the vial and the consequent risk for its user.

This equipment may be useful in the Laboratory of Analysis to prepare derivatization reactions which require a temperature higher than the room temperature (silanization, and so on)

In such a case, the vial must be closed and a temperature compatible with the sample and the derivatization reagent must be applied.

2 SYMBOLS AND EQUIPMENT LABELS

Table 1

Equipment label		
EC Conformity		
Warning symbol for high temperature printed on the oven cover	<p>WARNING High Temperature</p>	
Caution symbol for Hot areas, visible when the oven cover is opened	<p>DO NOT TOUCH HOT ZONES</p>	

3 USE OF THIS MANUAL

This manual describes the installation, use, maintenance and calibration of the Head Space Sampler 2t (model SHS 0112).

This equipment has been manufactured with the aid of the experience in the two former series.

The most significant difference is that this system incorporates in the same module for samples heating and for syringe heating, so it is easier to use it.

It is recommended a thorough and complete reading of this manual, to avoid a wrong use and to make it last longer. (working life)

In this manual, there is a detailed procedure for the Qualification of the Operation.

The application of this procedure regularly, will guarantee that the equipment is in optimum conditions and fulfils the basic requirements when the Laboratory works in a regulated environment (GLP, etc.).

4 INSTALLATION

When you receive the SHS 0112 equipment, check that you have received the following components:

Table 2

COMPONENT DESCRIPTION	Reference number
Heating and control module (220V)	MPH-132310
Syringe holder with thermal protection	MPH-132320
Retainer for syringe stopper	MPH-132370
Protector for syringe stopper end, plug piece 1	MPH-132350
Protection for syringe stopper end, socket piece 2	MPH-132360
Instructions manual	MPH-132330
Separators to adapt vials of 10 ml, in the holes 1,2,3,4,5 and 6 of the furnace	MPH-132380



Figure 1

The installation of this equipment requires only a space corresponding to 40 cm² of a table beside the Gas Chromatograph that is going to be used and an alternating current earth connection of 220 Volts.

It has a very appropriate compact design and reduced dimensions to be used with any type of Gas Chromatography unit because it is very easy to move.

In case it is used to make derivatization reactions, it is not necessary to put it beside the Chromatograph.

5 MATERIAL NECESSARY TO APPLY THE STATIC HEADSPACE TECHNIQUE

The SHS0112 Sampler will permit the transport of the Headspace of a closed system (vial) where the sample rests to the gas chromatograph, in a reproducible way. Besides the Sampler and the Gas Chromatograph, the following material is necessary to work with this technique:

Syringes of the required volume.

The system admits Hamilton syringes of 1.0 ml and 2.5 ml and any other syringe with some dimensions compatible with the geometry of the syringe block.

Table 3

Syringe type	Volume	Part Number
APS Syringe	1 ml	TR-132113
APS Syringe	2.5 ml	TR-132112

The vials of the volume considered appropriate for the analysis.

The SHS0112 sampler has eight different positions to heat vials of different dimensions.

This equipment has been designed to use it as a complement for the Automatic equipments, and it is appropriate to heat the vials used in the automatic equipments (of 11 different equipments).

In the table of the following page there is a list of all the vials and the respective positions and if an adapter is necessary, and there is also the corresponding vial reference.

For derivatization reactions, vials of small volume are frequently used and with SHS 0112 and the respective supplement, it is possible to heat vials of 2 ml and 4 ml. Table 4 indicates the vials that can be used.

Table 4

Automatic equipment	Vial size	Vial volume	Chromacol reference	Position in SHS0112	Required adapter code
Agilent	22 x 75 mm	20 ml	CC-20-CV	1,2,3,4,5,6	NO
	18 X 50 mm	9 ml	CC-9-CV	B	TR-132S01
	18 X 50 mm	9 ml	CC-9-CV	1,2,3,4,5,6	TR-132S02
AI Cambridge	22 x 75 mm	22 ml	CC-22-CV	1,2,3,4,5,6	NO
	18 x 65 mm	12 ml	CC-12-CV	B	NO
	18 x 65 mm	12 ml	CC-12-CV	1,2,3,4,5,6	TR-132S03
	18 x 50 mm	9 ml	CC-9-CV	1,2,3,4,5,6	TR-132S02
CTC/Leap	18 x 50 mm	9 ml	CC-9-CV	1,2,3,4,5,6	TR-132S02
Dani	22 x 75 mm	20 ml	CC-20-CV	1,2,3,4,5,6	NO
	18 x 50 mm	9 ml	CC-9-CV	1,2,3,4,5,6	TR-132S02
Fisons HS 500	18 x 50 mm	9 ml	CC-9-CV	1,2,3,4,5,6	TR-132S02
Fisons HS 850	22 x 75 mm	20 ml	CC-20-CV	1,2,3,4,5,6	NO
	22 x 45 mm	10 ml	CC-10-CV	1,2,3,4,5,6	TR-132N10
Perkin Elmer	22 x 75 mm	20 ml	CC-20-CV	1,2,3,4,5,6	NO
	22 x 38 mm	6 ml	CC-6-CV	1,2,3,4,5,6	TR-132S04
Shimadzu HSS-2B	30 x 56 mm	27 ml	CC-27-CV	A	NO
Tekmar	22 x 75 mm	22 ml	CC-22-CV	1,2,3,4,5,6	NO
	22 x 45 mm	10 ml	CC-10-CV	1,2,3,4,5,6	TR-132N10
	18 x 50 mm	9 ml	CC-9-CV	1,2,3,4,5,6	TR-132S02
Varian	22 x 75 mm	22 ml	CC-22-CV	1,2,3,4,5,6	NO
	22 x 45 mm	10 ml	CC-10-CV	1,2,3,4,5,6	TR-132N10
	18 x 50 mm	9 ml	CC-9-CV	1,2,3,4,5,6	TR-132S02
Standard	15 x 46 mm	4 ml	CC-4-CV	1, 2, 3, 4, 5, 6	TR-132SV01
				A	TR-132SV02
				B	TR-132SV03
	12 x 32 mm	2 ml	CC-2-CV	1, 2, 3, 4, 5, 6	TR-132SV04
				A	TR-132SV05
				B	TR-132SV06

Septums to close vials, according to sample nature and incubation temperature. A Chlorobutyl septum (general use, p/n with cap CC-20-AC-CBT3) or a septum with Teflon liner (in case of strong samples) can be used, p/n with caps CC-20-AC-ST3).

One crimper, the tool to cap or close the vials. It must firmly press the septum to obtain absolute tightness at the incubation temperature. One of the first problems when you start to work with this technique, is that the sample leaks because the vial is not correctly closed.

p/n Hand Crimper 20mm CC-C4020-100

Syringe Installation

Select the syringe to install and prepare the pieces shown in the Figure 2



Figure 2

Place the pieces as it's shown in the Figure 3 and insert the plunger with the right high to pick the desired gas volume.



Figure 3

Insert the mounted syringe inside the syringe holder heating block and set the syringe plunger stop to load the desired gas volume. Figure 4



Figure 4

6 DESCRIPTION OF THE EQUIPMENT



Figure 3

The SHS 0112 sampler is a metal block furnace that has 8 seats for vials of different size and a central hole for the metal block to heat the syringe.

The syringe heating takes place through the transmission of heat from the furnace to the block, and that means that some minutes are required to have equal temperatures in the syringe and in the furnace.

When the Head Space technique is applied, samples within the vials must be equilibrated, and this time to reach the equilibrium is also enough for the syringe to reach the working temperature.

The green led of Ready shows us that the furnace temperature is uniform and that we can put the vials with the samples and the patterns in the furnace.



Figure 4

The SHS 0112 is an equipment composed mainly by two modules, the furnace that includes the control unit of temperatures and times, and the heating block of the syringe (see figure 4).

This latter has two possible positions, the one on the left that corresponds to the block when is introduced into the furnace and the one of the right that it is when the operator seizes it to pick the sample of the vial and to inject it in the gas chromatograph.

To handle the plunger end when the syringe is warm, two pieces of insulator are supplied (MPH-132350 and MPH-132360) that help its use.

The equipment admits any of the two syringes we have described in chapter 5 without the need of any adaptor.

Vials of 20 ml p/n Chromacol 20-CV in positions 1,2,3,4,5 and 6 are also admitted.

Vial of 27 ml in position A and vial of 12 ml in position B. To use vials of 10 ml in positions from 1 to 6, an adapter is required TR-132N10

The furnace cover has 8 needle guides with indication of the position of each vial.

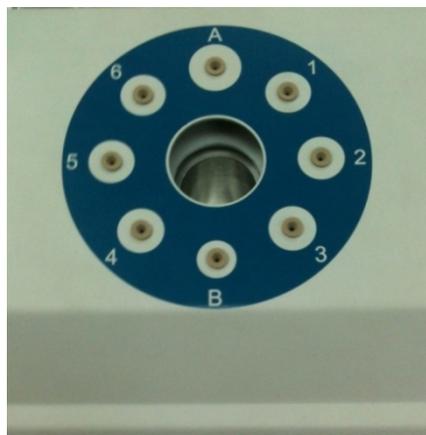


Figure 5

7 BASIC INSTRUCTIONS OF USE AND PROGRAMMING

On switching on the instrument to the power, all the segments of the display are lighted up during 1 second, followed by the software version of the instrument. Once the instrument is **On**, the display shows the actual temperature of the heating block and the °C and Time leds are switched off.

Taping on  or on  we will change the screen readings:

Actual Temperature (3 digits)



Equilibrium time in minutes (*E* blinking and 2 digits)



Program Sampling Time in seconds (*S* blinking and 2 digits)



When with any of the viewed screens, we tap on **START/STOP** button, we will begin to heat the block or we will put on the chronometers.



If we press **START** when the screen shows the temperature, the instrument will blink the °C led and it will begin to heat up to arrive to the set temperature and when it reaches to this value, it will sound a warning beep. With **STOP** we will stop the beep.



If we press **START** when the screen shows the Equilibrium Time the instrument will blink the Time led and the chronometer will begin to run, discounting the programmed minutes and when it finishes, it will sound a warning beep. With **STOP** we will stop the beep.



If we press **START** when the screen shows the Sampling Time, the instrument will blink the Time led and the chronometer will begin to run, discounting the programmed seconds and when it finishes, it will sound a warning beep. With **STOP** we will stop the beep.



PROGRAM SETTINGS

SETTING PARAMETERS

When the instrument is stopped (all the leds are **Off**) is allowed to modify the setting parameters of the method.

Table 5

Parameter	Minimum	Maximum	Resol.	Unit
Working Temperature	10°C	200°C	1°	°C
Equilibrium Time	00 min	99 min	1	minutes
Sampling Time	00 sec	99 sec	1	Seconds
Temperature Offset	-9°C	+9°C	1	°C

Pressing on  or on  we will change the screen readings.

Temperature

Viewing the actual Temperature in the display (3 digits), stable °C led and pressing  button, we will see the programmed temperature.

To modify it, remain pressed the button  and rise or

lower the temperature value taping on  



Equilibrium Time

Equilibrium Time in minutes (**E** blinking and 2 digits), we will see directly the programmed time.

To modify it, remain pressed the button  and rise or

lower the time value taping on   buttons.



Sampling Time

Sampling Time in seconds (**S** blinking and 2 digits), we will see directly the programmed time.

To modify it, remain pressed the button  and rise or

lower the time value taping on   buttons.



Temperature Offset

It's possible to correct the read temperature of the display with respect to the recorded reading of an external temperature probe.

To do so, remain pressed these two buttons at once   and afterwards rise or lower the Offset value on the range of -9 to +9°C. After 4 seconds, the display changes to the standard values.

WORKING PROCEDURE

Once the working parameters are set, we select the temperature reading in the display and we press **START** to heat the system with the syringe aluminium block allocated into the bath block.

Meanwhile, we prepare samples and patterns in the vials, always filling the vial without exceeding more than half of the height of the vial.

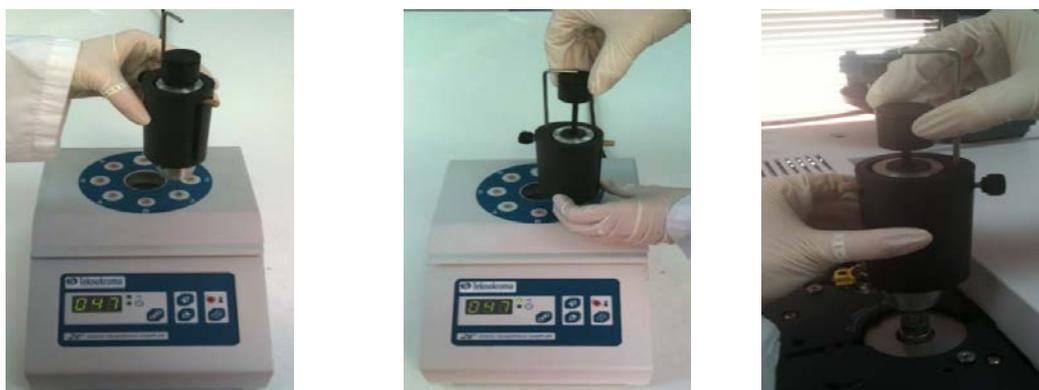
When the set temperature is reached, the instrument beeps, we stop the warning with **STOP** button and we introduce the vial or vials inside the right allocations of the heating block depending on the vial volume. Then, we close the instrument cover and we introduce the syringe inside its allocation moving the aluminium block to the bottom.



In this situation, we have to show the equilibrium time in the display and press **START** to switch on the chronometer. When the programmed time was elapsed, we press **STOP** and all it's ready to take the gas sample from the vial.

To do so, we lift the syringe aluminium block and we secure it in the syringe holder. We take the syringe and we introduce the needle inside the vial through the needle guide of the cover. We aspirate the desired volume of sample lifting the plunger up to syringe stopper.

We remain the syringe needle inside the vial and seeing the sampling time in the screen, we start the timer with **START** button.



After the programmed seconds are elapsed to allow the pressure equilibrium between the vial and the syringe, we press **STOP**, take the syringe with the sample, go towards the GC injector and, inject.

8 MAINTENANCE

The maintenance realised by the user must be based on the cleaning of the different equipment elements. This excludes the cleaning of the syringe and the vials. In case a cleaning operation takes place, the equipment must be completely disconnected of the mains.

To clean the surfaces of the furnace and the control module, use a cloth with a mixture of water and ethanol, in equal parts. The cleaning of the isolating protection must not be done with solvents that could attack it. A mixture of water and alcohol can be used.

FUSE REPLACEMENT

The fuse is in the back part of the equipment. For its renewal, the equipment must be disconnected of the mains. Use only a fuse of the following characteristics:
F2AL 250V

The user must not make any other maintenance operation and must not open the equipment. In case it does not work correctly, call the Technical Service of Teknokroma.

ERROR MESSAGES

The errors that could show the instrument are:

Table 6

Error	Description	Solution
Er1	Reading of the Pt100 probe Temp>175°C or probe disconnected	Inspect connexion of Pt100 probe
Er2	Read/Write error on E2PROM memory	Reset device

For any other problem please, contact with the provider of the instrument.

9 PERFORMANCE QUALIFICATION

To check the proper performance of the Head Space SHS model 0112, the following Reproducibility Test is recommended. In this test, we check not only the performance of the equipment but we also evaluate:

- That the vials are well closed.
- That the sampling procedure followed by the analyst is the correct
- That the Gas Chromatograph works properly
- That the data-acquisition system works properly

Sample preparation

Add 2.5 µl of benzene and 2.5 µl of toluene in 100 ml of water (25ppm), stir until it is completely dissolved.

Chromatographic parameters

Column: TRB-1, p/n TR-113015

Dimensions: 15m x 0.53mm x 3µm

Injection: 0.7 ml, head space, (split 1:2), 150°C

Carrier gas: He, 4 psi (27.6 kPa)

Oven temperature: 60°C (10 min.)

Detector: FID, 250°C

Sample: 5ml in 10ml vials (25 ppm benzene/toluene in water)

Headspace Conditions

10ml vials, p/n TR-132N10

Cap with blue silicone/PTFE seal p/n CC-20-ST3

Furnace temperature: 75°C

Equilibrium time: 30 minutes

Sampling time: 30 seconds

Syringe used: 1001 LTN APE-SYRINGE 1 ml TR-132113

Sampled volume: 0.7 ml

Inject 0.7 ml of headspace of every six vials. Integrate the peaks of benzene and toluene of the 6 chromatograms obtained. The Relative Standard Deviation of the area quotients must be lower than 5%.

Table 7

Benzene area	Toluene area	Area ratio
3418.461	5441.008	0.628
3466.125	5549.905	0.625
3359.176	5381.354	0.624
3316.646	5374.388	0.624
3782.404	6035.683	0.627
3794.026	6063.646	0.626
Mean value (x)		0.626
Standard deviation (s)		0.00163
Relative standard deviation (Sr)		0.26%

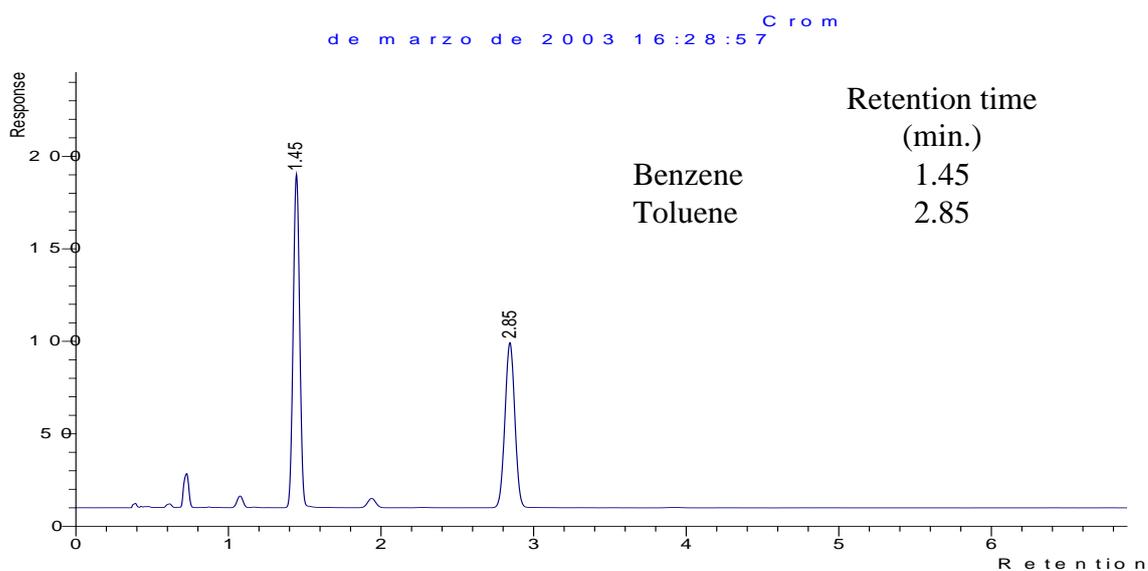


Figure 6

10 SOME PRACTICAL RECOMMENDATIONS

To obtain good results with the SHS 0112, make sure that the syringe is tight at the working temperature.

When a gas is sampled, the dry syringe moves and an accelerated erosion of the Teflon tipare produced, and that could cause leaks in the syringe, and that means that the injections are not repetitive.

This problem will be reduced with a lubrication of the stopper tip of the plunger with polydimethylsiloxane with a viscosity of 100 cs (reference Aldrich 37, 836-4) or with other liquid not interfering in the analysis (water, dimethylsulfoxide).

When the analyst is going to determine the analyte traces in the sample he finds a great difficulty with the residual contamination of the syringe because traces of volatile products cause blanks with multiple peaks that will probably interfere in the analysis.

To clean the syringe the best procedure is to aspirate and blow out air or dry nitrogen several times.

Another solution is to maintain the syringe without stopper at a temperature higher than the working temperature for a period of time.

Sometimes, chromatograms with extraordinary high peaks may appear and this generally will be due to the aspiration of some liquid with the needle.

The Static Headspace technique is based on the sampling of the gas in equilibrium with the liquid or solid sample in a closed vial. It may also happen that the peaks are lower than the usual ones, and the reason will probably be a leak in the syringe or in the vial.

11 TECHNICAL SPECIFICATIONS

Table 8

Operating ambient conditions	Temperature: 10-40°C Relative humidity: 20-80% RH Height: from 0 to 2000 metres
Power	Alternating current: 220 ±10%
Heating temperatures Range	Room temp. +10°C until 140°C (*) (* If a Gas tight Hamilton Syringe designed for HeadSpace technique with p/n TR-132113 or TR-132112 is used to work with the instrument, then the instrument can operate up to 140°C. Room temp. +10°C until Tsyringe max (**) (**) The maximum operating temperature of the instrument shall correspond to the maximum temperature can be held by the syringe.
Temperature accuracy	± 0,5°C
Hamilton syringes, volumes admitted	1,0 ml and 2,5 ml
Vials volumes admitted	2 ml, 4 ml, 6 ml, 9 ml, 10 ml, 12 ml, 20 ml, 22 ml and 27 ml
Sampling time control with acoustic alarm	1 to 99 seconds
Equilibrium time control with acoustic alarm	1 to 99 minutes
Incorporates thermal protection with heating disconnection	Safety temperature is 175°C
Stabilization time from 25°C to 70°C with a syringe of 1 ml and 6 empty vials of 20 ml	20 minutes

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Teknokroma Analítica S.A.
Camí de Can Calders, 14 - 08173 Sant Cugat del Vallés (Barcelona) Spain
Tel: +34) 936 748 800 Fax: +34) 936 752 405 - www.teknokroma.com