

Dedicated to the GC-Olfactive technique Maximum flexibility for future needs Protecting your investments

Table of content

Revision B 2005	1
Read this first!	5
Unpacking	5
Standard Outfit	5
Operating Voltage	5
Warranty	6
Warranty	6
Nariany	6
Safety Instructions	⁰
Precautions when using the unit	7
	<u>′</u>
GC - Olfactometry for the determination of key odorants in food flavours and	framances 8
I. INTRODUCTION	
II. HISTORY AND CURRENT DEVELOPMENTS IN THE CHARACTERIZATION OF KEY FOODS	ODORANTS IN
Gas Chromatography-Olfactometry	
III. IMPORTANCE OF ISOLATION TECHNIQUES	9
IV. GAS CHROMATOGRAPHY-OLFACTOMETRY	
Gas Chromatography-Olfactometry	
A. Limitations of GCO	
General Description	12
GC - Olfactometry is a recently used Method in the Flavour and Frangrances b	ndustry 12
GC - Olfactometry - Principle of operation	12
Technical description	13
Technical specifications	14
External stand alone olfactive measurement system.	14
Description of the Instrument	
Description Base Unit	15
Description Interface outlet support	15
Description Pneumatic controller	16
Pneumatic Lontroller Battery exchange	18
Description Remote Rand Controller	19
Software description Remote Yeard Controller	01
Menustructure Hemote Hand Controller	21
How to move in the Menu	66
How to change Values	22
F1 to F8 function buttons	23
The Tempcont Setup Menu	23
Switch on the Sniffer 9000	24
Selftest successfully performed	24
Set Temperature	25
Set Temperaturesave value	25
Show actual temperature	26
Set analogous output	26
Set analogous outputchoose a range	27
using the analogous output	27 00
Lero setting of the analogous output	28 28
036 u.e anauyous oupu	20 20
Set Dynamics output for Finger Span Ontion •	
, RCU Settings	30
Sniffer 9000 software option	31

Installation of the Sniffer 9000	32
Mechanical installation	32
Installation on a HP 5890 / 6890	34
Installation on a HP 5890 (left side)	34
Installation on a HP 5890 (right side)	35
Installation of a 3 port Sniffer 9000	37
Mechanical installation of a 3 port SNIFFER 9000 system	38
3 port SNIFFER 9000 system3 port mechanical support view	38
Scheme of the fused silica connections 3 port system	39
Pneumatic installation	40
Analytical installation	41
Important!!!	43
T-Piece installation!!!	43
Schematic of the fused silica connections	43
Glass nozzle cleaning	44
Glass nozzle cone dismounting	44
Output length, Gas speed table	45
Ordering informations	47



Brechbühler AG

Steinwiesenstrasse 3	
CH-8952 Schlieren	
Switzerland	
Tel:	+41 44 732 31 31
Fax:	+41 44 730 61 41
e-mail:	info@brechbuehler.ch
Internet:	www.brechbuehler.ch

Sniffer 9000 Instruction Manual

August 2005 Edition

Copyright 1999 - 2005 Brechbühler AG, Switzerland, All rights reserved. Printed in Switzerland

Published by Brechbühler AG, Steinwiesenstrasse 3, CH-8952 Schlieren

Tel:	+41 44 732 31 31
Fax:	+41 44 730 61 41
e-mail:	info@brechbuehler.ch
Internet:	www.brechbuehler.ch

Read this first!

Unpacking

When receiving the product, inspect the packing for any damage.

If the external packing is damaged, make a note and pay particular attention during unpacking.

When the product has been unpacked, inspect it carefully for if any damage occurred.

If any damage is found at the Instrument, notify it immediately your Brechbühler AG representative, who will give you instruction accordingly.

Standard Outfit

Please check also carefully if the material delivered as standard outfit complies with the checklist enclosed in the package.

Operating Voltage

The SNIFFER 9000 operates on 100 - 240 Volt AC.

Warranty

Warranty

Brechbühler AG warrants all of its products to be free from defects in workmanship or material under normal use and service within the period of one year.

Exemption: 6 months warranty for the heated transfer line

The warranty does not cover consumables, damages caused by improper use of the instrument and damages due to transportation.

The warranty will be invalidated (specific agreements excepted) if the instrument is installed in the absence of a authorised and qualified Brechbühler AG service engineer.

Disclaimer

Brechbühler AG makes no warranty of any kind with regard to this material, including, but not limited to, warranties of merchantability and fitness for a particular purpose.

Brechbühler AG shall not be liable for errors contained in this document or consequential damages in connection with the furnishing, performance, or use of this information.

The information contained in this document is subject to change without notice. The contents of this document may be revised without prior notice.

Brechbühler AG also reserves the right to make changes and/or improvements to its products without incurring any obligation to incorporate such changes or improvements in units previously sold or shipped.

Brechbühler AG undertakes no responsibility to any defects of a Gas Chromatograph, in a caused manner trough inappropriate installation or disregard the Instruction Manual.

Safety Instructions

Precautions when using the unit

Warning!

The Sniffer 9000 control unit is a sensitive analytical instrument. All electrical connections must be well grounded.

Poor grounding represents a danger to the operator and may be seriously affect the efficiency of the instrument.

Warning!

The Sniffer 9000 Interface controlled by the Sniffer 9000 control unit is heated during operation and can get very hot.

Do not touch the Interface during operation, you could get burned!

Using Sniffer on unknown samples

Warning!

GC-Olfactometry (GCO) should not be conducted on unknown samples.

Safety and health of the person who is doing GCO should be the primary concern before evaluating samples using the GCO technique. Users conducting GCO should be aware about the nature of the samples they will be exposed to during analysis. Usually Laboratories do have their own internal regulations and safety standards for such a matter.

GC-Olfactometry usually should be applied to Food or Fragrances extracts only. Please do not apply this Method to industrial waste effluents.

Brechbühler AG undertakes no responsibility to any incidental misuse by disregarding the safety regulations in your organisation.

Brechbühler AG will not be responsable for any accident created by analysing unknown sample mixtures.

Theoretical Aspects

GC - Olfactometry for the determination of key odorants in food, flavours and fragrances

I. INTRODUCTION

The early nineteenth century saw the beginnings of analytical work on food flavours. Benzaldehyde was the first flavouring material isolated from bitter almonds by Vogel in 1818 and by Martres in 1819 (1), but it was not identified until 1832. Fifty years later, Tiemann and Haarmann (2), and later Reimer (3) isolated, identified, and synthesized vanillin. This was the first flavour compound to be synthesized (1), and it heralded the era of the flavour industry.

Initial studies in flavour research were hampered by the inadequate separation provided by packed column gas chromatography (GC) and thus flavor chemists were the pioneers of capillary column chromatography. The evolution of capillary column GC and the interfacing of GC with mass spectrometry (MS) resulted in the separation and identification of numerous volatile compounds existing in different foods. Today, more than 6900 volatile compounds have been identified in foods and beverages (4). However, of all these volatile compounds, only a few determine the characteristic odour of a specific food. Sometimes only one compound is responsible for the character impact compound." Great strides have been made in flavour research, which is being directed towards combining olfactometry and sensory techniques with instrumental methods to gain insight into better characterization of food flavours.

In this chapter, we will take the reader through different phases - past and present - of odor and flavor cha-racterization by several pioneers in the field of flavor chemistry. Here we will talk about the development of gas chromatography-olfactometry (GCO), followed by the development of extract dilution methods and Osme. The existing new area of aroma recombination, which is the last piece of the puzzle, will also be mentioned. We will also critically review each method, presenting its strengths and limitations, and provide insight into work needed to further strengthen this aspect of flavor chemistry.

II. HISTORY AND CURRENT DEVELOPMENTS IN THE CHARACTERIZATION OF KEY ODORANTS IN FOODS

In 1997, the concept of odour activity value (also known as OAV, unit flavour base, odour unit, odour value, or aroma value) will celebrate its fortieth year in the field of gas chromatography-olfactometry. The concept of OAV was first proposed by Patton and Josephson (5) shortly after the introduction of gas chromatography by James and Martin in 1952. In this early work, GCO simply referred to smelling the GC effluent after its separation. This was an exciting period for flavour chemists, who were now able to assign odour descriptions to GC peaks and to separate odour active chemicals from volatile chemicals with no or minimal odour response. There were great expectations of GCO being able to unlock flavour secrets; however, when GCO was used, most food systems failed to produce distinct character impact compounds (6,7). The challenge then was to determine the relative importance of a flavour compound to the overall flavour of the food, giving rise to the idea of aroma effectiveness. A value to express aroma effectiveness was originated by Patton and Josephson (5), who proposed relating the concentration of a compound to its sensory threshold. The concept was first termed "aroma value"

by Rothe and Thomas in 1963 (8) Instruction Manual (Revision B 2005)

Gas Chromatography-Olfactometry

Other authors used the terms "odour unit" (6), "unit flavour base" (9), and "odour value" (7). "Aroma value" is the ratio of the concentration of an aroma compound to its threshold, thereby indicating how much the actual concentration of a compound exceeds its sensory threshold.

Since the concept of "aroma value" was proposed, GCO and OAV have been extensively applied in the area of flavour chemistry. These techniques have clearly demonstrated that the most abundant volatiles may have little if any odour significance in a food. These techniques have been invaluable in determining undesirable or foreign off-odours in foods or in determining differences between a control and variant of the control (e.g., aged product, different source, process effect). Unfortunately, the original vision of using GCO and OAV for flavour duplication work has met with very limited success. With the exception of works by Buttery et al. on tomato flavor (10, 11), Blank and Grosch on dill herb flavour (12), Schieberle et al. on butter flavour (13), and Grosch on cheese flavour (14), no sensory confirmations of OAVs of compounds or their contribution to the original flavour have been published.

The last forty years have served as a long incubation time for the evolution of GCO methods. During this time, three methods have emerged: Aroma Extract Dilution Analysis (AEDA) (15), CharmAnalysis (16), and Osme (17). Their emergence as academic and commercial GCO methods and a divergence in theory, technique, and interpretation has only occurred within the last 12 years.

III. IMPORTANCE OF ISOLATION TECHNIQUES

The importance of selecting the appropriate isolation technique for GCO work should not be overlooked. The success of flavor characterization of foods by GCO depends largely on the isolation technique employed to isolate the flavour compounds from the food matrix. The number of odours detected by GCO depends on the method used to isolate the flavour volatiles from the food. Due to large numbers of volatiles compounds found in foods, their differences in physical and chemical properties, and their low threshold levels, isolation of volatiles from foods becomes very challenging. There are innumerable methods of flavour isolation from a food matrix; however, each method will introduce biases into an aroma profile. Therefore, different methods may provide different aroma profiles. Other variables that influence GCO results include the amount of food sampled, dilution of the volatile fraction by the solvent, and aliquot of extract injected into the gas chromatograph. The interested reader can refer to an in-depth article by Reineccius (18) on the biases in flavour profiles introduced by isolation techniques.

The importance of aroma isolation method in aroma recovery is illustrated by Guth and Grosch (19). Known amounts of odours were dissolved in ethyl ether, added to sunflower oil, and then isolated by sublimation in vacuo. The yields of odours obtained by this isolation method ranged from 1% (for 4,5epoxy-(E)-2-decenal) to 78% (for I-octen-3-one). In another example, Leahy and Reineccius (20) compared 10 different methods for the recovery of a flavour model system from dilute aqueous solutions. The percent recoveries of the compounds were unique for each method evaluated (recoveries ranged from 0 to 100%) depending upon aroma compound. and isolation method). By direct solvent extraction of an aqueous solution containing methyl anthranilate, only 10% was recovered using pentane as the solvent compared to approximately 80% recovery using dichloromethane (20). In investigating "foxy" character in wines made from Concord grapes, the contribution of methyl anthranilatc to this "foxy" note (21) may be missed if pentane is chosen as an extraction solvent.

Since any subsequent work by GCO is dependent on how well an extract represents the aroma in question, substantial time must be invested in this phase of the study. Each isolation method has its unique weak-nesses; hence, more than one isolation method may have to be used to accurately view the true aroma of the food (e.g., headspace and vacuum distillation). Numerous aroma isolation methods should first be investigated and the aroma extracts evaluated by formal sensory evaluation techniques. The isolation methods selected should be based on sensory confirmation of the best representation.

IV. GAS CHROMATOGRAPHY-OLFACTOMETRY

GCO, sometimes referred to as "GC sniffing," is an important analytical tool in flavour research because it characterizes the odours of single compounds or complex mixtures of volatiles emerging from the sniffing port. Here, the human nose is the detector used for evaluating the effluent of the GC column. The nose has a theoretical odour detection limit of about 10-19 moles (22), making GCO a very valuable and sensitive tool for the detection of odour active volatiles like 2-methoxy-3-hexyl pyrazine (odor threshold of 1 part in 1012 parts of water) (22). Acree (23) indicated that a person would be able to detect 50-500 fg (10-15) of (-damascenone by GCO. Volatile compounds with different odours, even those with very small differences in retention index, will be differentiated from one another by GCO (23). GCO has often been used to detect trace amounts of character impact compounds or off-odours in foods. For example, when the effluent of a pineapple extract was sniffed by GCO, a typical pineapple odor was recognized in a certain region of the GC profile (24).

Gas Chromatography-Olfactometry

However, the sensitivity of the FID (flame ionization detector of a GQ was too low to detect a peak. Upon subsequent enrichment of the extract, the peak was identified as ally hexanoate (24). We saw a similar problem related to an off-odour in beet-sugar. A musty odour, similar to that of the original beet-sugar sample, was identified by GCO in a particular region of the GC profile. Once again, there were no peaks detected by the FID in that region. Upon further enrichment of the extract, geosmin was identified as the compound producing the musty odour. Since many flavour volatiles have extremely low odour thresholds, detection of these compounds by GC-FID becomes very difficult and one may have to rely on GCO to elucidate the odor of the GC peak of interest.

A. Limitations of GCO

To accurately perform the analysis and evaluate data, one should be aware of the limitations of GCO. Sensory perception of a food (good vs. bad, banana or coffee, etc.) is arrived at by the simultaneous integration of all of the taste and olfactory signals arising from the taste and aroma compounds in a food. In GCO, however, we are attempting to determine the contribution of a given aroma compound separately as it elutes as an individual peak on the gas chromatograph. This compound is being evaluated out of context, so to speak. This may make evaluation of its contribution to a food difficult or impossible to assess accurately. An alternative method might use GC effluent trapping of peak fractions and subsequent sensory evaluation of these collected fractions to simulate the original flavour of the food. Here, one could

trap what is considered to be the most characteristic aroma compound Instruction Manual (Revision B 2005) and compare its odour to that of the entire food aroma complex. Assuming it fails to reproduce the food aroma, one could then add additional compounds until the reconstructed aroma fraction is judged to be indistinguishable from the entire food aroma. This would permit the evaluation of odours in combination as is done in normal tasting. Furthermore, the compounds needed in these fractions would be readily available in the aroma isolate and in the correct proportions as well. This approach would potentially compensate for attempting to evaluate an aroma compound out of context as is typically done by GCO methods.

The elution order, odour character, and perceived intensity of one compound will directly influence the perceived intensity of the compound eluting after it. This is referred to as the "contrast effect" and can be corrected for by balancing the presentation order (25). Manipulation of elution time and order of the compounds can be accomplished by column selection and chromatographic parameters (temperature, pressure, carrier gas). Manipulation of elution order, however, is limited by column selection (polar vs. nonpolar phases). A balanced presentation of elution order is not possible by GCO; however, evaluating the perceived intensities of eluting compounds on a minimum of two column phases (e.g., DB- I and DB-Wax) would provide validity to the data. Another limitation of GCO is that fatigue, sensory saturation, and adaptation on the part of the human subject will negatively influence intensity measures and must, therefore, be minimized (26,27). There is no scientific evidence to show whether the perceived intensity of a compound detected at the fortieth minute of continuous sniffing would be similar to the intensity of the same compound if it were evaluated after 5 minutes of sniffing. These factors could be addressed by dividing the sniffing time into shorter intervals. The order in which a subject evaluated these shortened runs could be randomized and balanced in design among subjects; this would further reduce contrast effects.

The condition or physical environment under which GCO is conducted has a significant influence on the validity of the data. Noise, distractions, extraneous odours, and uncomfortable test conditions may introduce error. Ideally, GCO should be conducted in an isolated, quiet room with a positive pressure airhandling system to prevent and eliminate odours. The subject should be able to perform the test in a comfortable position, preferably seated versus standing over a warm gas chromatograph. The hot, dry carrier gas carrying the GC effluent to the nose may be irritating and uncomfortable. To correct this problem, a gentle stream (20-30 ml/min) of humidified air may be used. This prevents drying of olfactory mucosa and condensation of components on the walls of the olfactometer mask, if used.

GCO should not be conducted on substances of unknown composition. The health and safety of subjects evaluating samples by GCO should be the primary concern. Subjects conducting GCO should be aware of the nature of the material they will be exposed to during the analysis. Laboratories should have their own regulations and safety standards in this matter (e.g., GCO should be applied only to food extracts, not to industrial waste effluents). Currently, there are no OSHA regulations or university guidelines in place regarding this application of human subjects.

Selection, screening, and training of human subjects as "sniffers" during a GCO run should be carried out in an objective manner (25). Unfortunately, in GCO studies the researchers involved with the project may have a biased opinion when conducting GCO.

Reprinted from "Techniques for Analysing Food Aroma" by Ray MarsiliISBN: 0-8247-9788-4Instruction Manual (Revision B 2005)Page - 11 -

General Description

GC - Olfactometry is a recently used Method in the Flavour and Frangrances Industry.

In the past decades, many detection techniques have been hyphenated to gas chromatography. Less attention has been paid to GC-olfactometry (GC-O) in which the human nose plays the role of the detector. However, the human nose is often more sensitive than any physical detector, and GC-O exhibits powerful capabilities that can be applied to flavours and perfumes, as well as to any odoriferous products (e.g. pollutants).

Olfactometric (or "sniffing") techniques allow the determination of impact odours in food. They can be classified into two categories: dilution methods, which are based on successive dilutions of an aroma extract until no odour is perceived at the sniffing port of the chromatograph; and intensity methods, in which the aroma extract is only injected once but the smeller records the odour intensity as a function of time by moving the cursor of a variable resistor.

The Sniffer 9000 System is designed to be a dedicated "Sniffing-Device" as a stand alone unit to be connected to any GC available on the



market.

The new Electronic- and Pneumatic design , based on a new industry standard (LON) provides maximum flexibility for future needs thus protecting your investments in the Laboratory.

GC - Olfactometry - Principle of operation.

The GC - Olfactometry is a simple to use Method, by installing at the end of a chromatographic column a split which allows the sample to be splitted (e.g. 1:50) FID Detector /nose. The peak/odor impression correlation will then be performed by specialised fragrancy chemists.



Technical description

As heart of the Sniffer 9000 GCO technology is the dedicated GC/Olfactometer heated Interface. Brechbühler AG has over 25 years experience in implementing hyphenated techniques by using special designed interfaces. The Sniffer 9000 interface is engineered to transfer the high resolution available on capillary columns to the olfactometry "Sniffing Port" air delivered by any Capillary column without loss of GC resolution and influence of oxidation or turbulence.

Built as part of the current Sniffer 9000 System, the interface is manufactured from a single Stainless steel tube which is heated by direct current, thus giving the best possible temperature profile. The interface consists of a standard fused silica line resulting in a inert olfactometer interface. Engineered to combine hot column effluents with laminar streams of inert make-up gas generated by the Sniffer 9000 System and additional humidification air supply to deliver distinct odours to humans subjects with minimum discomfort and maximum separation.

Integrated into any GC System available on the market, Brechbühler AG's Sniffing 9000 System is one of the most flexible GC/Olfactometer Systems available on the market.

A special analogous output allows the Sniffer 9000 to be connected to an additional Channel on an existing data system, or any integrator or recorder available in the lab, to record intensity via the integrated cursor wheel on the Hand-held Remote control unit.



in this example was done by injecting a Citrus mixture. The peak of interest are ceranial and citral (see fig. 1)

As the humane nose is much more sensitive to certain compounds the result of the odour intensity is shown in fig. 3, where we can see that the odour intensity peak is much more intensive then the FID peak.



Technical specifications

External stand alone olfactive measurement system.



Connection through dedicated heated transfer line of 80 cm (length) x 25 mm (diameter) Optional 140 cm available Great flexibility. Easy installation to any GC Workstation on the table with temperature and pressure settings for the secondary gas

Possibility to be sited while working with elbows on the table

Easy assembling to the left or to the right of the oven

30 mm oven wall drilling diameter

Drill the whole then guide the transfer line up to the oven Outlet with glass nozzle cone. Height : About 30 cm above the table

Temperature of the transfer line : $50^\circ\mathrm{C}$ to $350^\circ\mathrm{C}$ with control of accuracy





Make-up (Optional)

feeding (Nitrogen) with pressure control switch from 0 to 300kPa Flow rate : 0 to 50 ml/min. Connection of the make-up in the oven on Y PressFit and mixing at release gas outlet **Air feeding humidifier (Standard)** bottle (sparging) switch from 0 to 300kPa. Connection in the oven at the transfer line inlet Flow rate of 0 to 50 ml/min. Preheated humid mixture going around melted silicium of the transfer line outlet Fitting the outlet split by choosing different lengths and diameters melted silicium outlet detector blade. Maximum outlet diameter : 0.32 mm

Air Humidifier controller (Standard outfit)

Make Up controller (Optional)



Hand-Held Remote control unit, to program desired interface temperature Power 115/220 V. 50/60 Hz max. 10 Amp

Description of the Instrument

Description Base Unit



Heated flexible interface

Description Interface outlet support

The conditions or physical environment under which GCO is conducted has a significant influence on the validity of the data. Ideally GCO should be conducted in a isolated, quiet room. The user should be able to perform the test in a confortable position, preferably seated versus standing over a warm gas chromatograph.

The Interface outlet support has been designed in a way that the user may adjust the sniffing position in to the most confortable way. No limitation even if the Sniffer 9000 has to be installed on the right or on the left side of the Gaschromatograph.



Description Pneumatic controller

The hot, dry carrier gas carrying the GC effluent to the nose may be irritating, uncomfortable and drying mucous membrane at the users nose. To prevent this, a gentle stream of humidfied air may be used among the fused silica line to the outlet.

The Electronic pressure controller of the Sniffer9000 consists of a air humidification pressure controller to allow the user to adjust the gas flow of the humid Air.

A Electronic Pressure regulator for make-up gas is available as an option

PCU module Humid Air and Make-up





bottle of humidification

Option : PCU module Make-up

The Humid air and Make-Up Pressure controller are Battery driven. The standard life time of the Battery is about 2 Years.

To adjust the flow just turn the Pressure controller knob to the desired flowrate



Maintenance

The Pressure controllers does not require any routine maintenance.

Pneumatic Controller Battery exchange

Open the Pneumatic controller

withdraw the old battery









Install the replacement battery

Description Remote Hand Controller

To add a maximum of additional comfort the remote hand Controller has been developed in a way to give the user access to all the necessary parameters like temperature, analogous outlet signal on a very comfortable way and even while seating.

LCD Display 2x16 Characters (Backlighted)	
Function Keys (F1 to F8)	6 6 6 6
Down Key ———	F5 F6 F7 F8
UP Key	
Shift Key —	U UUWN
Enter Key ————	SHIFT
Escape Key	- Call
Jogg wheel	0



Description Heat controller rear side



Earth / Ground for interface, cable comming from the interface

Instruction Manual (Revision B 2005)

Software description Remote Hand Controller

Menustructure Remote Hand Controller



Kow to use the Sniffer 9000 menu

The Software in the Sniffer 9000 hand controller has a built in menu structure which uses a Jogg wheel to move up and down in the different menu options.

How to move in the Menu.....

To move down in the menu turn the Jogg wheel clockwise and the menu scrolls upwards.

To move up in the menu turn the Jogg wheel counterclockwise and the menu scrolls downwards.

If you placed the cursor at the menu Option you want press ENTER to open this menu



How to change Values.....

To change Values turning the Jogg wheel clockwise will increase them, turning the Jogg wheel counterclockwise will decrease them.

Values increasing or decreasing resolution is set by default to 1 degree steps.

Pressing the UP key once changes the resolution to 2 degree steps,

pressing the UP key a second time changes the resolution to 5 degree steps,

pressing the UP key the third time changes the resolution to 10 degree steps,

pressing the UP key the fourth time changes the resolution to 20 degree steps,

pressing the UP key the fifth time changes the resolution to 50 degree steps

and pressing the UP key the sixth time changes the resolution to 100 degree steps.

To change back the resolution press the DOWN key in the same numbers you pressed the UP key.



F1 to F8 function buttons.....

To access the F1 to F4 press the appropriate function button.

To access F5 to F8 use the red shift button together with the desired function button



The Tempcont Setup Menu.....

By default the Tempcont Setup Menu is disabled. You don't see this Menu point when the Instrument is delivered. The Tempcont Setup Menu is used for Service purposes only.

This Menu point is only accessible by entering a password and has to be used by qualified Service Engineers only.

Note:

The Tempcont Setup menu allows the Service Engineer to set different values for Testpurposes and allows some Diagnostics in the field.



Switch on the Sniffer 9000

The Sniffer 9000 system has a built in Power On check routine which does during power on cycle a internal test to assure that all the components are working correct.

Switch On

After switching on the Heater Control unit the display at the hand controller shows for about 30 seconds "Sniffer 9000", "Status Error". During this time the system performs a selftest routine to check if the heater and the thermocouple is present.

Selftest successfully performed.....

After the self test has been performed and all the checks passed successfully the display at the hand controller shows "Sniffer 9000", "Status OK". Now the system is ready to start working

Set Temperature......

To move the cursor down to the menu "Set Temp" turn the jog wheel to the right side the menu scrolls up and you may point the cursor to the menu "Set Temp". Value 0 on "Set Temp" means heater is off.

Set Temperature......save value.....

If you moved the cursor to "Set Temp" press "Enter" to enter in to the Menu "Set Temp". To increase the set value turn the jog wheel clockwise, to decrease the set value turn it counter clockwise. If you reached the desired value press Enter again to save the value.

Show actual temperature......

The system returns to the Main menu, where you now can se the "Act Temp" increasing and the "Set Temp" Value is at 250.

Set analogous output....

The Sniffer 9000 has an built in analogous output to record odour intensity on a external recorder or a data system. To adjust the range of the output signal move the cursor down to "Res Lev" and press enter.

Set analogous output....choose a range....

You may choose a range between

0...1 Volt 0...5 Volt 0...10 Volt

Press Enter to save the desired output range.

Using the analogous output......

Move the cursor to "Rec Out" and press "Enter" the display shows Analogous followed by a value indicating a certain percentage of the Output (the moving bar on the bottom displays the same value Graphically). If you turn the jog wheel clockwise this value is increasing, if you turn it counter clockwise the value is decreasing.

Zero setting of the analogous output......

The zero setting of the jog wheel is important for the user to know where the zero point of the wheel is, or to set this point to a position which is the most comfortable to use it. Move the jog wheel to the position you want to set it to zero and then press "F1" the zero setting is performed to this position immediately.

Use the analogous output......

After zero setting is performed the system is ready to be used to give your impression on odour intensity to a data system or to a recorder. On the right of the Graphic display you see a number which is indicating how many turns of the jog wheel is giving 100%. The default value is 1 (means 1 turn = 100%). This value may be changed to 2, 5 or 10 turns. To increase the value use the "DOWN" key, to decrease this value use the "UP" key.

Set Analog Output

Choose the range in volt :

RES LEV Enter choose (0,5-1,0-5,0 and 10 V)

REC OUT Enter choose 1, 2, 5, and 10 turns by press Down

F1 = zero setting Or : F2 = Marker 100% signal

Set Dynamics output for Finger Span Option :

REC OUT press Up = **Dyn** Enter

F1 = zero setting move the curser and then press :

F3 =100%

RCV Settings.....

To enter in to the RCU Settings menu move the cursor down to "RCU Settings" and press "ENTER". In this menu you may adjust the Contrast and Brightness of the display, the speed of the jog wheel and you may set all the values to default parameters. To exit this menu move the cursor down to "Return" and press "ENTER". The menu points "Save Values" and "Load Values" are not active. These menus are for future extensions.

Sniffer 9000 software option

There are three modes a Standard	vailable:			
Ramp man Ramp Auto	manual start external start			
For example :				
Mode Standard choose Ramp man	Enter			
Temp 1	0° C			
choose temperature Time 1 choose time	0.0min			
Rate choose rate	0°/Cmin			
Temp 2 choose temperature	0° C			
Time 2 àchoose time	0.0min			
To start Temp, program				

To start Temp. program **Press Shift F5 = Start Ramp**

To stop Temp. program **Press Shift F6 = Stop Ramp**

Installation of the Sniffer 9000

Mechanical installation......

The Sniffer 9000 may be installed to any GC available on the market. The most GC's in the field do have on the left or on the right side already a whole to enter with a interface. You may choose the appropriate whole which fits to the needs of the flexible heated interface of the Sniffer 9000 or you have to drill a new whole to install the flexible heated interface to your GC.

You may fix the interface by using the aluminium support and the included screws in the standard outfit as you see on the picture. Please note that the interface is using a direct heating system. Therefore it is mandatory that you use only the aluminium support which is delivered with the Sniffer 9000 system otherwise you may have a short circuit to the GC which may result in damaging the heat controller of the Sniffer 9000 system

To change the installation side from the left to the right side (or vice verca) turn the Interface outlet support in a 90 degree angle to the front. Use the include Inbus key to loosen the fixing screw then turn the complete support to the desired position and fix the screw again

After you changed the Interface direction to the desired position and you fixed again the support you may install the Interface to the GC.

Installation on a HP 5890 / 6890......

On HP 5890/6890 the SNIFFER9000 may be installed on the left or on the right side. It depends what type of hyphenated detectors are installed to the Instrument. If there is no IRD or MSD installed on the left side of the GC usually the SNIFFER9000 will be installed at the left side using the existing hole for the MS Interface.

Installation on a HP 5890 (left side)......

If your GC HP 5890 is not equipped with a MSD you may enter with the SNIFFER9000 interface from the left side of the GC oven. Mount the included interface support at the outer oven wall and the you may easy insert the Interface in to the support and fix it with the screw at the interface support.

Installation on a XP 5890 (right side)......

If your GC HP 5890 is equipped with a MSD you must enter with the SNIFFER 9000 interface from the right side of the GC oven.

After fixing the aluminium support you have also to drill a hole in to the outer plastic cover of the GC.

You can install a 1/8" stainless steel tube as guide for the fused Silica lines coming from the Pressure controller fore make-up gas and humid Air to enter easy in to the oven

Installation of a 3 port Sniffer 9000

Mechanical installation of a 3 port SNIFFER 9000 system......

The Sniffer 9000 concept allows the installation of up to 3 ports to one GC.

GC-Olfactometric measurements are time consuming and therefore the need of more then one Sniffer port on one GC is a real advantage for research laboratories.

The Sniffer 9000 does allow to install up to 3 ports on one GC using a special designed adaptation kit to the GC. To avoid the drilling of big holes in to your GC a special mechanical support has been designed to connect the different inter-

face lines.

3 port SNIFFER 9000 system......3 port mechanical support view

Scheme of the fused silica connections 3 port system......

Here below you see the schematics how to connect the fused silica lines to the SNIFFER 9000 3 port system interface

Pneumatic installation......

The Standard Electronic Pressure control unit for humid air . On the right side of the pressure control unit there is the output connector for the gas line going to the metallic t-piece.

Air line comming from Pneumatic Controller for HUMID AIR

Analytical installation......

After the Sniffer 9000 is physically connected to the GC we need to install the 4-way splitter inside the oven, which allows us to connect the make-up gas. The 4-way splitter usually is a pressfit connector which allows us to install fused silica- lines coming from the pressure controller of the Sniffer 9000.

At the left or right side of the pressure controller we have a special designed support where the fused silica lines going to the GC oven may be connected. Inside the GC oven we have a pressfit 4-way splitter where we connect the make-up gas line. Additionally we do have a special designed metallic piece which allows the connection of the hunid air line.

Important!!!.....

Please take care that the Interface metallic part (including the metallic t-piece) does not make any contact to the GC oven wall. Due to the fact that the dedicated heated interface is using direct current to the interface metallic tube, you may create a short circuit, which may influence the temperature regulation.

T-Piece

installation!!!..... After you have in-

stalled your interface you have to connect the metallic t-piece to the interface outlet by

straight end 0.32 fused silica comming from the 4-way pressfit

90 degree angle 0.53 fused silica comming from HUMID AIR

Schematic of the fused silica connections......

Here below you see the schematics how to connect the fused silica lines to the SNIFFER 9000 interface

Sniffer 9000

Glass nozzle cleaning......

The Glass nozzle mounted on the SNIFFER9000 interface exit may be dismounted by pulling them of the glass nozzle cone. It may be necessary to clean the glass nozzle from time to time due to contamination of the glass surface with your sample.

Glass nozzle cone dismounting......

If necessary you can also dismount the glass nozzle cone by turning it counter clock wise.

Page - 44 -

Output length, Gas speed table

Sniffer 9000, Output length, Gas speed

30m Zebron Column. ID 0.53 ZB-1 film thickness 1,5 µm On-column Injector, Carrier gas: H₂ 40 kPa.

Theoretical flow rate:

Length m.	ID mm.	Film	Gas	Pressure kPa	Temperature	Flow ml/min	Speed	Holdup time	Comments
		μm					Cm/sec		
30	0.53	1,5	H ₂	40	40°	19.46	126.14	V _o 23.78 sec	

Measure: The actual flow rate is slower than expected. The drop in pressure is greater than expected. The measured flow gives us a 0.508mm ID.

Length m.	ID mm.	Film	Gas	Pressure kPa	Temperature	Flow ml/min	Speed	Holdup time	Comments
		μm					Cm/sec		
30	0.508*	1,5	H ₂	40	40°	16.4	115.88	V _o 25.89 sec	Ø 0.53

Theoretical calculus for the detector output: 80 cm transfer line.

Length m.	ID mm.	Film µm	Gas	Pressure kPa	Temperature	Flow ml/min	Speed Cm/sec	Holdup time	Comments
1.0	0.25	0	H ₂	10	40°	6.33	272.25	V _o 0.47 sec	Ideal 95cm

Theoretical calculus for the Sniffer output: 80 cm transfer line

Length m.	ID mm.	Film	Gas	Pressure kPa	Temperature	Flow ml/min	Speed	Holdup time	Comments
		μm					Cm/sec		
1.2	0.32	0	H ₂	10	40°	14.16	289.90	V ₀ 0.41 sec	

To get comparable elution speed the length of the detector output (0.25mm ID) should be adjusted to 95cm, the elution speed for the sniffer (120cm, 0.32mm) and detector will be about 280 to 290 cm/sec with V_o at 0.41 sec.

The difference in diameter will yield to a FID: Sniffer split ratio of 1 : 2.2.

Measures for both output: (split ratio 1 : 2) with 30m. 0.53mm ID column installed. (No Make-up).

Length m.	ID mm.	Film µm	Gas	Pressure kPa	Temperature	Flow ml/min	Speed Cm/sec	Holdup time	Comments
0.95	0.25	0	H ₂	40	40°	5.5			FID
1.20	0.32	0	H ₂	40	40°	10.9			Sniffer

Experimentation shows that the speed of elution is identical. Chromatograms FLAIR35.DAT and ODORAT35.DAT shows the overlay of the FID trace and Odor intensity.

Theoretical calculus for the detector output: 14

140 cm transfer line.

Length m.	ID mm.	Film	Gas	Pressure kPa	Temperature	Flow ml/min	Speed	Holdup time	Comments
		μm					Cm/sec		
1.3	0,25	0	H ₂	10	40°	4.87	163.27	V _o 0.80 sec	Ideal 1,25m.

Theoretical calculus for the Sniffer output: 140 cm transfer line

Length m.	ID mm.	Film	Gas	Pressure kPa	Temperature	Flow ml/min	Speed	Holdup time	Comments
		μm					Cm/sec		
1.6	0.32	0	H ₂	10	40°	10.62	217,35	V _o 0.74 sec	

Theoretical calculus for a split ratio of 1 : 2 (Calculated ratio 1 : 2.18)

Length m.	ID mm.	Film	Gas	Pressure kPa	Temperature	Flow ml/min	Speed	Holdup time	Comments
		μm					Cm/sec		
1.25	0,25	0	H_2	10	40°	4.87	163.27	V _o 0.76 sec	FID
1.60	0.32	0	H_2	10	40°	10.62	217,35	V₀ 0.74 sec	Sniffer

3. Theoretical calculus for three Sniffers output and one detector. 3 x 140 cm transfer lines.

Length m.	ID mm.	Film	Gas	Pressure kPa	Temperature	Flow ml/min	Speed	Holdup time	Comments
-		μm			-		Cm/sec	-	
1.6	0.25	0	H_2	10	40°	3.96	132.66	V ₀ 1.21 sec	FID
1.6	0.25	0	H_2	10	40°	3.96	132.66	V ₀ 1.21 sec	Sniffer 1
1.6	0.25	0	H_2	10	40°	3.96	132.66	V ₀ 1.21 sec	Sniffer 2
1.6	0.25	0	H_2	10	40°	3.96	132.66	V ₀ 1.21 sec	Sniffer 3

Equal flow (speed) for each output can be obtained with 4 outputs of 1.60m. The total flow is 15.84ml/min, which is sufficient for a 30m wide bore (0.53) column.

For such installation, you must measure the flow for each branch and adjust the length if necessary. Because of the small diameter of the output lines, no make-up is needed. The pressure from the make-up gas would actually reduce the flow out of the column.

4. Theoretical Calculus for 4 Outputs and one Make-up. 3 transfer lines (140cm) and one detector.

Length m.	ID mm.	Film	Gas	Pressure kPa	temperature	Flow ml/min	Speed	Holdup time	Comments
		μm					Cm/sec		
1.6	0.25	0	H ₂	10	40°	3.96	132.66	V ₀ 1.21 sec	FID
2.1	0.32	0	H ₂	10	40°	8.29	167.67	V ₀ 1.25 sec	Sniffer 1
2.1	0.32	0	H ₂	10	40°	8.29	167.67	V₀ 1.25 sec	Sniffer 2
2.1	0.32	0	H ₂	10	40°	8.29	167.67	V₀ 1.25 sec	Sniffer 3

This setup will allow for a greater total flow (28.83ml/min). This allows for the addition of make-up gas without affecting the column flow. The split ratio is not affected.

Ordering informations

Part No.	Description
	Base Unit
9 1000050	Sniffer 9000 GC/Olfactive measurement system includes: Dedicated flexible heated interface (Standard 80 cm length) Heater control unit Hand held control unit Special analogous output to record odour intensity standard outfit, manual

	Accessories / Spare parts
9 1000055	Flexible heated interface 80 cm (standard)
9 1000056	Flexible heated interface 140 cm (optional)
9 1000060	Outlet glass nozzle
9 1000061	O-Ring for outlet glass nozzle cone
9 1000062	Outlet glass nozzle cone (alu minium part)
SUNI 4X(B)	4-way pressfit universal
9 1000064	T-Piece metallic 1x1/16" 2x 4mm
9 1000065	O-Ring for Sparger bottle
9 1000066	Sparger bottle complete
9 1000080	Recordecable set (analog output)
9 1000081	Inbus key 5mm
35032423	Nut 303 M4 (set of 5)
290 13487	Ferrules graphite ID 0.45 mm
29013486	Ferrules graphite ID 0.80 mm
160-2325-10	Fused Silica ID 0.32 (10 meter)
160-2535-10	Fused Silica ID 0.53 (10 meter)

Manufactured by:

Steinwiesenstrasse 3 CH-8952 Schlieren, Switzerland Tel. +411732 31 31 FAX +411730 61 41

Document Type: BREAG MAN-SNIFFER9000-ENG-R-2.00-6/99