BTEX GC User Manual

Version 2.3

mlu-recordum

Environmental Monitoring Solutions GmbH

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Safety message

Please note that all maintenance on this device should be performed by trained personal. Hazards include: heated elements parts, electricity, toxic and carcinogenic compounds (benzene).

Introduction

The BTEX-GC is a Microsystems-based device for real-time monitoring of ppb and sub-ppb concentrations of aromatic volatiles (Benzene, Toluene, Ethylbenzene, Xylenes, or BTEX) in air. Its reduced size makes it ideal for being included in the airpointer, and the absence of a carrier gas cylinder significantly decreases the need for periodic maintenance.

1- Specifications

General specifications

Measurement principle	Gas Chromatography – Photoionization Detection (GC-PID 10.6 eV), column MEMS packed
Carrier gas	Zero air ~10 ml/min
Compounds monitored	Benzene, Toluene, Ethylbenzene, Xylenes
Weight	4 Kg
Dimensions	Version1: Width: 660 mm, Depth: 300 mm, Height: 100 mm Version2: Width: 300 mm, Depth: 250 mm, Height: 200 mm
Sample flow rates	~300 ml/min (or ~100 ml/min depending on the application)
Operating temperature	-20 to +40°C Optional heater for -40°C available
Cycle time	10/15 minutes
Measurement units	µg/m^3, ppb
Autocalibration	Permeation tubes
Power	Two versions are available: 115V/60Hz or 230V/50Hz
Power consumption	10W average, 18W peak
Maintenance interval	Twice a year
Performances	
Lower Detectable Limit	0,2 ppb
Range	100 ppb (extended range on request)
Memory effect (carryover)	<1% benzene

Warranty

Same terms as described in ‘Chapter 4.3. Warranty’ of the airpointer manual apply.

2- Principle

ThermoDesorption Gas Chromatography (TD-GC)

Thermo-Desorption Gas Chromatography is the USEPA reference method for measuring VOCs including BTEX. The VOCs are preconcentrated on a solid adsorbent at ambient temperature prior the analysis. The sudden heating of the adsorbent releases the trapped compounds into the column. The chromatographic column, swept by carrier gas, separates the compounds according mainly to their volatility: the most volatile compound is the first to reach the detector.

Photo-Ionisation Detector (PID)

A 10.6 eV lamp ionizes the molecules that enter the detector. The ions are collected on an electrode, thus creating an electrical signal.

MicroElectroMechanical Systems (MEMS)

MEMS are miniaturized devices, with components typically smaller than 1 mm in size. This technology allows to reduce dramatically the size, the weight and the electrical consumption of an analyzer. *Figure 1* shows the BTEX-GC column and pre-concentrator next to a 1€ coin.

MEMS Pre-concentrator MEMS GC column

FIGURE 1: MEMS

3- Overview of the BTEX-GC system

The BTEX-GC consists of the following main blocks:

A. Sampling and pre-concentration unit

B. Gas-chromatographic (GC) separation unit

C. PID detector unit

D. Manifolds for flow distribution and filter

E. Housing, including cooling fans and electrical connectors

Some of the main blocks include sub-blocks, which are listed below:

A. Sampling and pre-concentration unit

   A1 Sampling pump

   A2 Injection valve

   A3 MEMS pre-concentration chip

   A4 Pre-concentrator control electronics and MEMS interface

   A5 Pre-concentrator fluidic block with block temperature control

B. GC separation unit

   B1 Carrier-gas pump

   B2 Injection valve

   B3 MEMS packed GC separation chip

   B4 GC control electronics and MEMS interface

   B5 GC fluidic block with block temperature control

C. PID detector unit

   C1 PID detector containing the PID lamp

   C2 PID control electronics

   C3 PID fluidic block

Please refer to *Figure 2* and *Figure 3* for identifying the above blocks and sub-blocks.

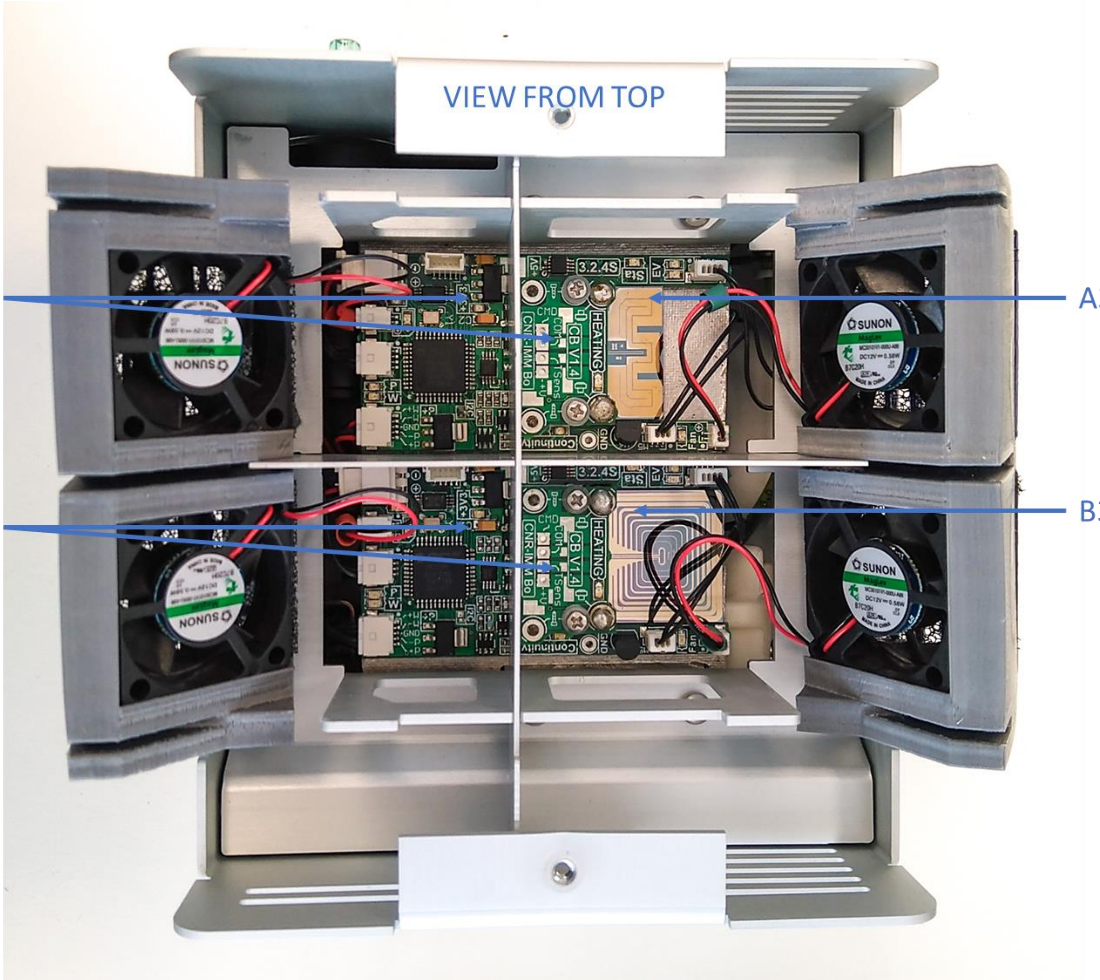


FIGURE 2: OVERVIEW OF THE BTEX-GC SYSTEM CORE HARDWARE (VIEW FROM TOP)

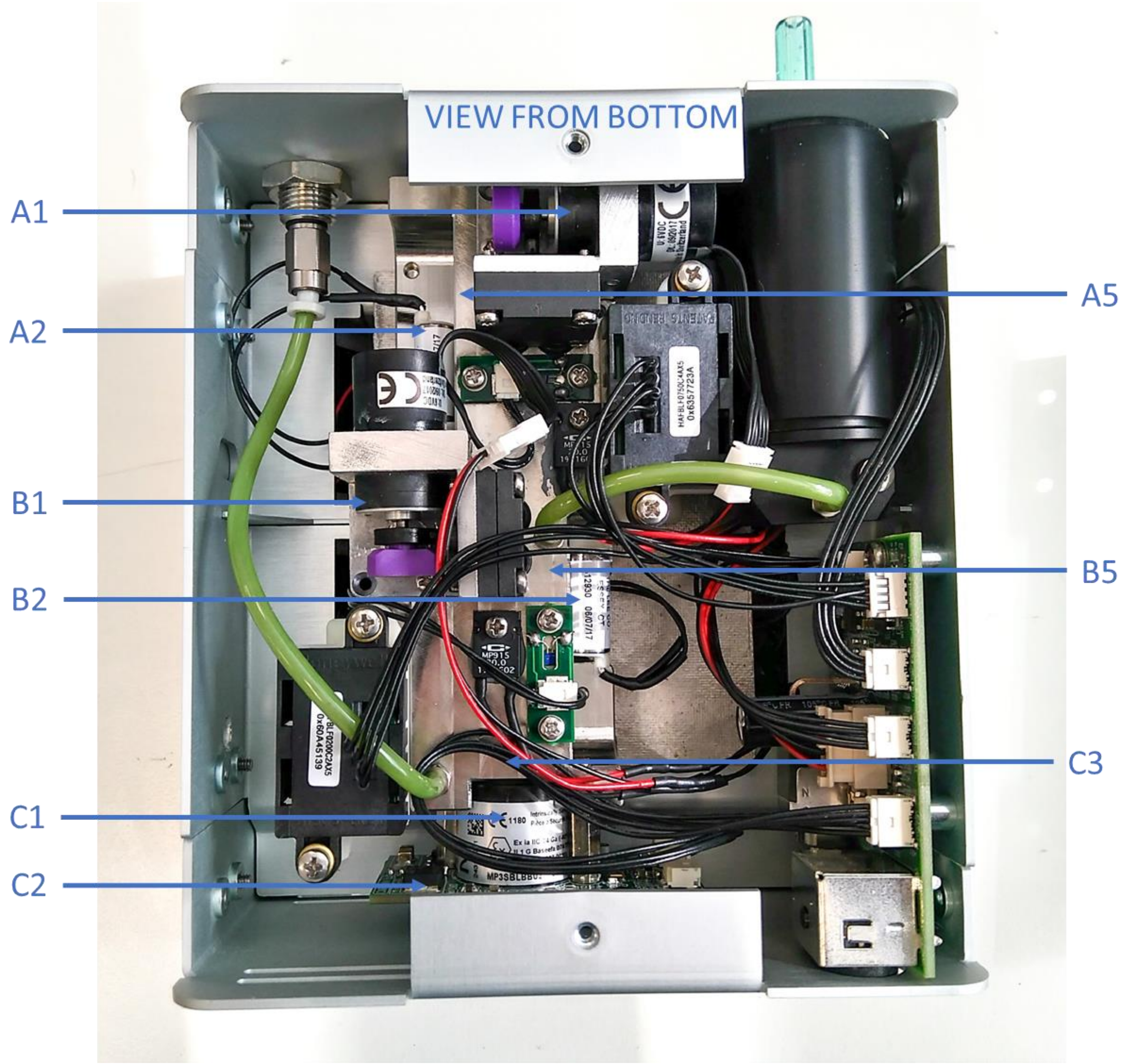


FIGURE 3: OVERVIEW OF THE BTEX-GC SYSTEM CORE HARDWARE (VIEW FROM BOTTOM)

The operating principle of the BTEX-GC is described in the following:

An analytical cycle consists of a complete set of sequential system states which implement a complete measurement. The result of each single measurement cycle is a chromatogram, which is processed to extract the height of the peaks of the compounds, namely benzene, toluene, ethylbenzene and xylene.

The height of each peak is linearly correlated, by means of a calibration factor, to the concentration of each single compound sampled during a sampling cycle.

In the current set-up, the BTEX-GC has a fixed cycle duration of 15 minutes. This means that the height of each of the BTEX peaks is computed 4 times every hour.

The height of each peak is proportional to an average concentration of the compound, as sampled during approximately 10 minutes during a 15 minutes cycle. 4 minutes are needed for the cleaning of the pre-concentrator between successive measurement cycles, and 1 minute is used as a buffer, to allow the device to start the next cycle at the exact time. This is due to the fact that the sampling time varies slightly because the sampling stops when a sampled volume of 2800 ml is reached. This enhances the repeatability of the measurement. The contents of the pre-concentrator are injected at 60” after the beginning of the following cycle, so the first 60” of each cycle, all units are idle.

The phases during a 15 minutes cycle are described in more details in *Table 1* and *Figure 4.*

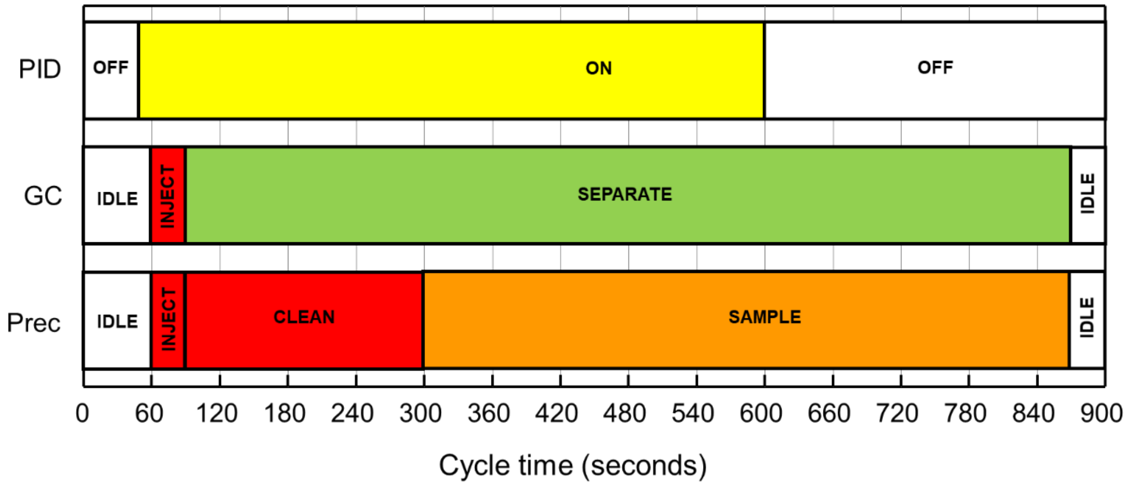


TABLE 1: STATES OF THE SINGLE BTEX-GC UNITS DURING A MEASUREMENT CYCLE

As can be seen from the table above, the pre-concentrator samples the air during the final 600” of the cycle.

This means that the chromatogram peaks acquired by the PID detector refer mostly to the air sampled during the previous cycle!

The preconcentrator flow is written F_1 and the GC column flow F_2. The temperature of the preconcentrator is noted T_{prec}.

The first phase is the idle phase, from t = 0” to t = 60”, in which F_1 = 0 ml/min; F_2 = 30 ml/min; T_{prec} = 40°C.

The second phase is injection phase, from t = 60” to t = 90”, in which F_1 = 30 ml/min; F_2 = 30 ml/min; T_{prec} = 110°C

The third phase is the cleaning phase, from t = 90” to t = 300”, in which F_1 = 300 ml/min; F_2 = 30 ml/min; T_{prec} = 110°C

The fourth phase is the sampling phase, from t = 300” to t = 900”, in which F_1 = 300 ml/min; F_2 = 30 ml/min; T_{prec} = 40°C

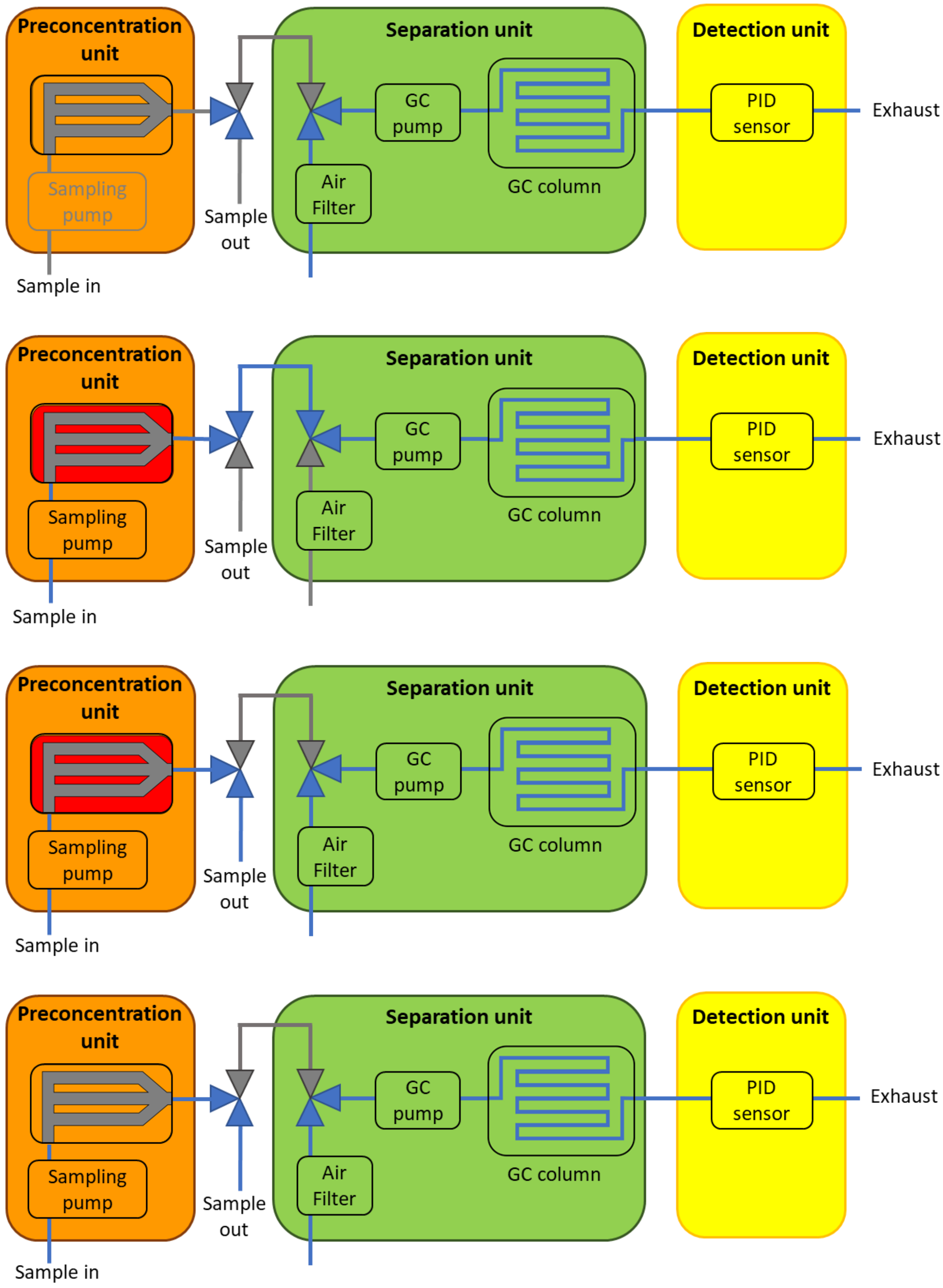


FIGURE 4: THE 4 PHASES OF A CYCLE, FROM TOP TO BOTTOM: IDLE, INJECTION, CLEANING, SAMPLING

A typical example of a chromatogram, as acquired by the PID detector during its 720” acquisition between 60” and 780” of the cycle, is shown below in *Figure 5*. In this example, the benzene peak elutes with a maximum signal at t=125” after injection and has a height of 11.173mV. The toluene peak elutes at 377” and is 49.367mV high, while no ethylbenzene was injected, therefore no clear peak is found.

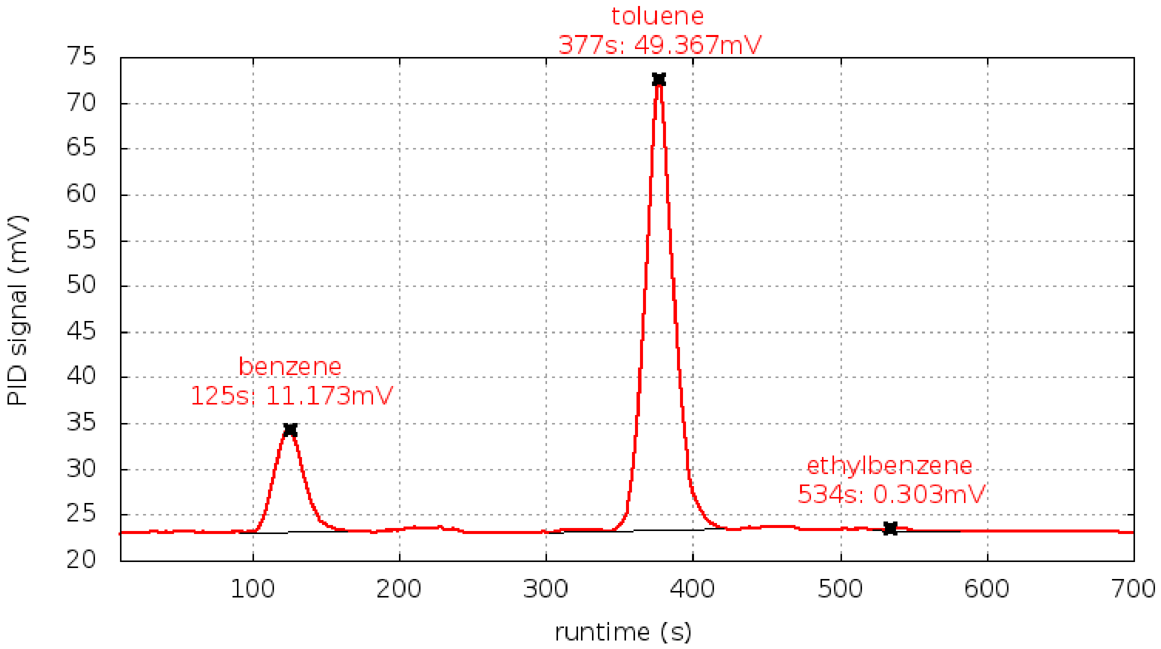


FIGURE 5: EXAMPLE OF CHROMATOGRAPH ACQUIRED BY A BTEX-GC SYSTEM

4- Hardware

Installation

The BTEX module comes in 2 version: the drawer version (*Figure 6*), suitable for all kind of Airpointers, and the compact version (*Figure 7*), available for the HC Airpointers only.

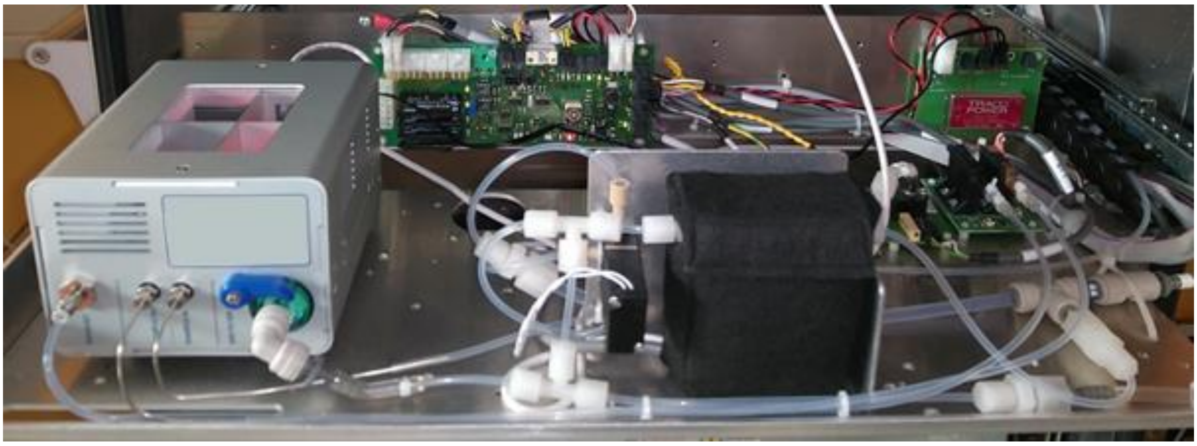


FIGURE 6: AIRPOINTER GC BTEX MODULE IN A DRAWER VERSION

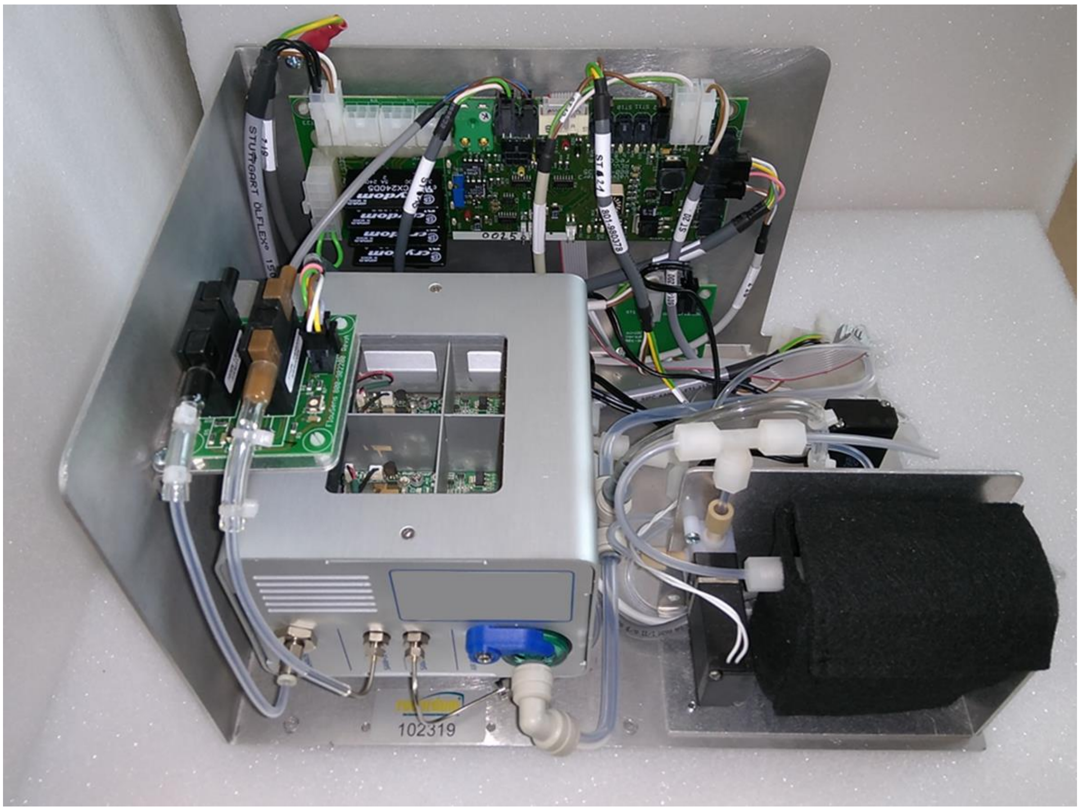


FIGURE 7: AIRPOINTER GC BTEX MODULE IN A COMPACT VERSION

The procedure for the mounting/unmounting of the module in the drawer version is identical to the one described in '*Chapter 10.3.4 – Lift a module out or in*' of the airpointer manual, except that there is an extra USB cable that needs to be connected to the Airpointer's PC.

The procedure for the mounting of the module in the compact version is similar to this of the drawer version, except that there is no rail to pull the module out.

Connections

FIGURE 8: FRONT VIEW OF THE BTEX-GC BENCH WITH PNEUMATIC CONNECTIONS

The fluidic connections identified in *Figure 8* are:

EXHAUST
This is the PID outlet. The flow eluting from the GC column and flowing through the PID chamber flows OUT OF this connector whenever the GC unit pump is on. In this phase, the flow out of this connector should be in the 10-20mL/min range. This flow increases to >20mL/min when the pre-concentrator unit is injecting into the GC separation unit.

SAMPLE OUT
This is the sample outlet. The sample air flows OUT OF this connector while the pre-concentrator is in the sampling phase. In this phase, the flow out of this connector should be around 300 mL/min. During the injection phase, the flow out of this connector must drop to 0 mL/min.

SAMPLE IN
This is the sample inlet. The sample air flows INTO this connector all the time the pump of the pre-concentration unit (A1) is on.

AIRFILTER
This is the carrier gas inlet. The air flowing through this activated carbon filter is used as GC carrier gas. Ambient air flows INTO this connector whenever the sampling pump of the GC unit (B1) is on and the system is not in the injecting phase.

SAMPLE IN, SAMPLE OUT, and EXHAUST are 1/16” Swagelok® connectors. The filter connection OD is compatible with 1/4” or 6mm Teflon® tube fittings.

FIGURE 9: BACK VIEW OF THE BTEX-GC BENCH WITH ELECTRICAL CONNECTIONS

The electrical connections identified in *Figure 9* are:

USB
Mini-USB port for controlling the BTEX-GC

POWER
Power supply: DC 12V 3A max rating

ON/OFF
Power switch, turns off only the pre-concentrator and GC units

Gas Flow of the BTEX-GC module

As shown on *Figure 10*, the BTEX-GC includes an internal span module. The drift can therefore be checked and if needed corrected daily. The procedure for the drift correction is described in the 'calibration' section.

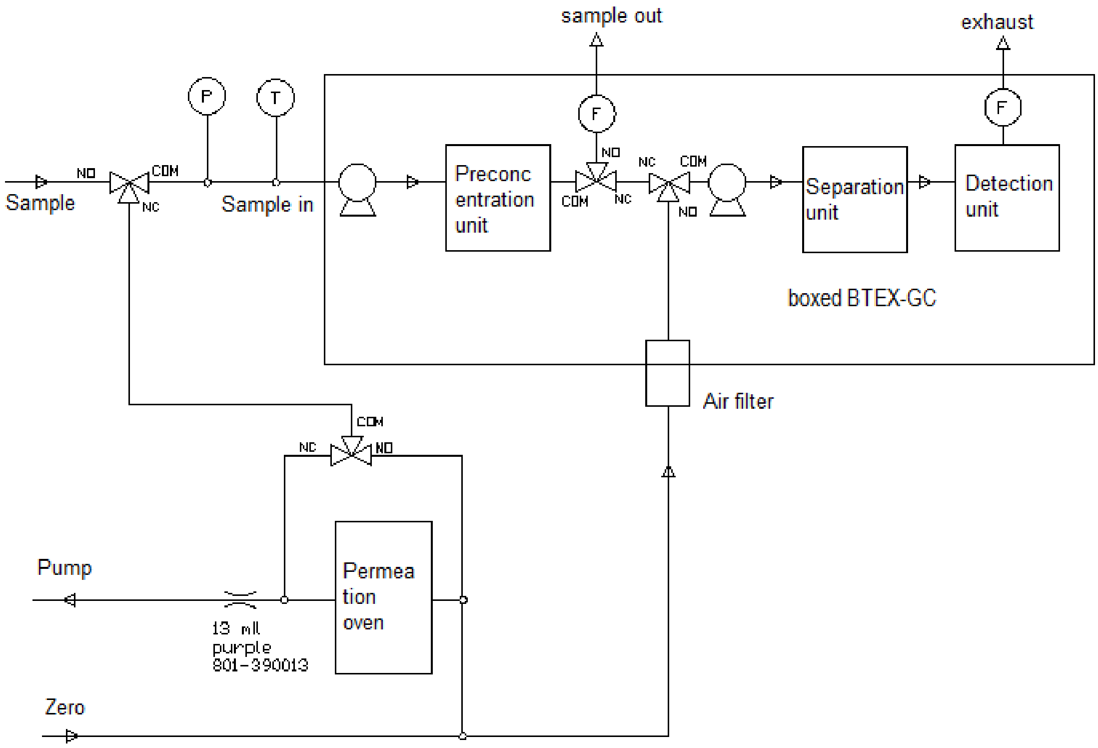


FIGURE 10: FLOW DIAGRAM OF THE BTEX-GC MODULE

5- Data Acquisition

In the following, the 6 parameters of the benzene peak are explained, as shown below in *Figure 11*.

PN = Peak name

PBS = Peak base start time (in seconds)

PBE = Peak base end time (in seconds)

PBW = Peak base search width (in seconds)

PMP = Peak maximum position (in seconds)

PMW = Peak maximum search width (in seconds)

Peak height calculation procedure:

The peak height is calculated as the difference between the PID signal at the peak maximum time and a baseline, which is constructed at the base of the peak by finding two PID signal minima.

The peak minima are searched close to the times **PBS** and **PBE**, at a maximum distance of **PBW** seconds. The baseline is a straight line connecting the two minima found close to **PBS** and **PBE**.

The peak maximum is searched close to the time **PMP**, at a maximum distance of **PMW** seconds.

Once the exact position of the peak is found, it is subtracted by the baseline at the same position, resulting in the peak height.

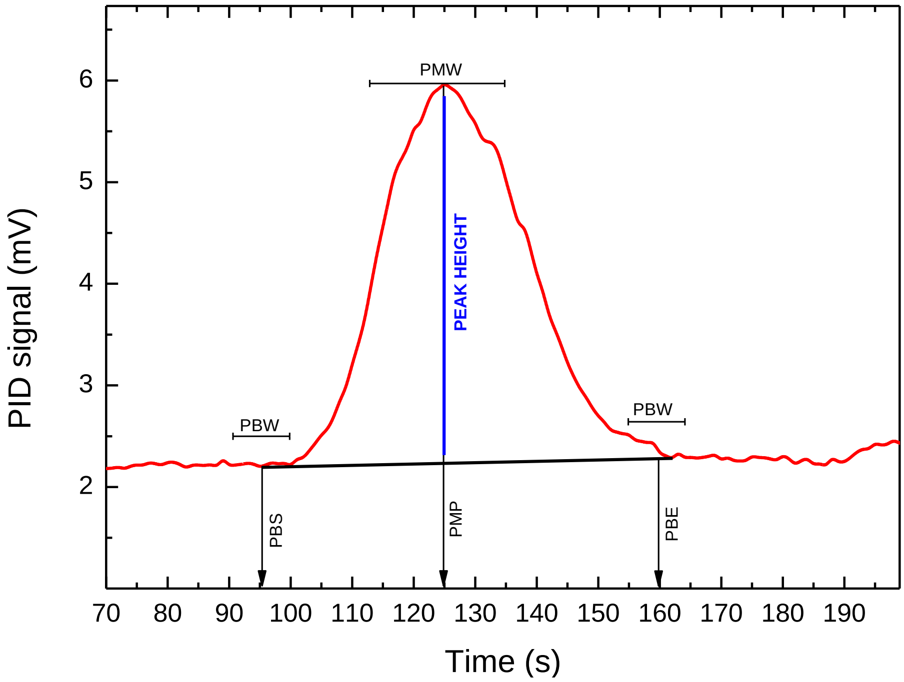


FIGURE 11: VISUAL REPRESENTATION OF THE PEAK INTEGRATION PARAMETERS

6- User Interface

Command Interface

In this section, the BTEX-GC can be reset or turned off as shown in *Figure 12*.

FIGURE 12: COMMAND INTERFACE

Zero and span checks

As shown in *Figure 13*, the Valve control interface is identical to the one described in 'Chapter 7.6 - Calibration' of the airpointer manual. However, contrary to most airpointer modules, the BTEX module works in 15 minutes cycles, and the sample n-1 is being analysed while the sample n is being sampled. Therefore, the user must wait a for minimum time of 30 minutes before obtaining the first result after switching a valve.

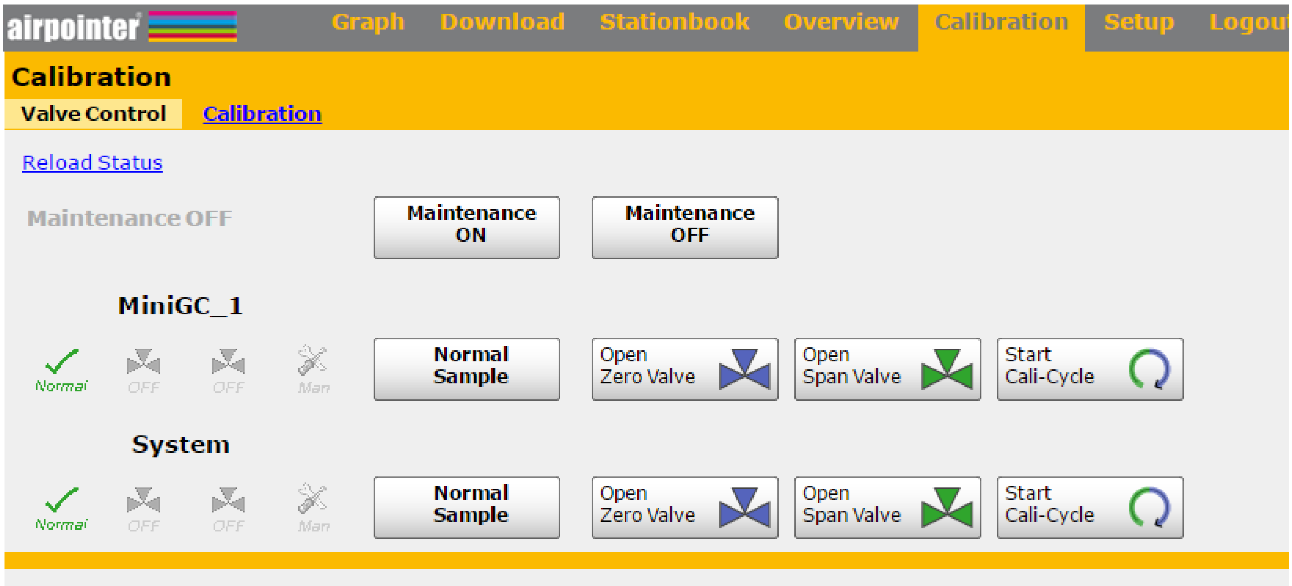


FIGURE 13: VALVE CONTROL OF THE BTEX-GC

Linsens

In the miniGC tab of the Linsens interface, the current values of the module are shown (see *Figure 14*).

Actual Values MiniGC 1 Diagram
no calibration active next automatic calibration cycle starts: 20160615 10:15:00

Parameter	Value	Unit	Status: BS-FS-SS	
Benzene	0.13	ppb	0 0 0	
Toluene	0.10	ppb	0 0 0	
Ethylbenzene	0.04	ppb	0 0 0	
Xylene	0.09	ppb	0 0 0	

Benzene_all (25/6)	0.13	ppb
Toluene_all (25/7)	0.10	ppb
Ethylbenzene_all (25/8)	0.04	ppb
Xylene_all (25/9)	0.09	ppb
Benzene_peak_height (25/12)	0.2	mV
Toluene_peak_height (25/13)	0.1	mV
Ethylbenzene_peak_heig (25/14)	0.0	mV
Xylene_peak_height (25/15)	0.1	mV
Benzene_baseline (25/18)	4.1	mV
Toluene_baseline (25/19)	4.0	mV
Ethylbenzene_baseline (25/20)	4.0	mV
Xylene_baseline (25/21)	4.1	mV

SampleFlow (25/51)	296.8	ml/min	SensorFlow (25/52)	13.5	ml/min
SamplePress (25/53)	968.1	mbar	SampleTemp (25/54)	25.6	°C
PermT (25/49)	49.9	°C	PowerToPerm (25/50)	28.8	%

FIGURE 14: ACTUAL VALUES

The StatList tab shows the current error status (color coded) and value, unit, lower and upper error limit, lower and upper warning limit of each parameter from the system and the installed modules. If limits were set, OK, warning or fail status are shown. OK is written in green, warning in orange and fail in red (see *Figure 15*).

MiniGC_1

G/P	Status	Parameter	Actual	Average	Unit	lower limit fail	lower limit warn	upper limit warn	upper limit fail	Board Adr
G25P1	OK	Benzene	-	-	ppb	-	-	-	-	-
G25P2	OK	Toluene	-	-	ppb	-	-	-	-	-
G25P3	OK	Ethylbenzene	-	-	ppb	-	-	-	-	-
G25P4	OK	Xylene	-	-	ppb	-	-	-	-	-
G25P6	OK	Benzene_all	-	0.36	ppb	-	-	-	-	-
G25P7	OK	Toluene_all	-	0.14	ppb	-	-	-	-	-
G25P8	OK	Ethylbenzene_all	-	0.04	ppb	-	-	-	-	-
G25P9	OK	Xylene_all	-	0.06	ppb	-	-	-	-	-
G25P12	OK	Benzene_peak_height	-	0.5	mV	-	-	-	-	-
G25P13	OK	Toluene_peak_height	-	0.1	mV	-	-	-	-	-
G25P14	OK	Ethylbenzene_peak_heig	-	0.0	mV	-	-	-	-	-
G25P15	OK	Xylene_peak_height	-	0.1	mV	-	-	-	-	-
G25P18	OK	Benzene_baseline	-	4.5	mV	-	-	-	-	-
G25P19	OK	Toluene_baseline	-	4.1	mV	-	-	-	-	-
G25P20	OK	Ethylbenzene_baseline	-	4.0	mV	-	-	-	-	-
G25P21	OK	Xylene_baseline	4.1	4.1	mV	-	-	-	-	-
G25P49	OK	PermT	50.0	49.9	°C	45.0	47.0	53.0	55.0	042
G25P50	OK	PowerToPerm	29.2	28.9	%	-	-	-	-	042
G25P51	OK	SampleFlow	297.5	72.0	ml/min	40.0	50.0	250.0	300.0	042
G25P52	OK	SensorFlow	13.4	5.0	ml/min	-	-	25.0	30.0	042
G25P53	OK	SamplePress	968.8	970.0	mbar	500.0	550.0	1090.0	1100.0	042
G25P54	OK	SampleTemp	25.4	25.4	°C	0.0	5.0	35.0	60.0	042

FIGURE 15: STATLIST

Configuration

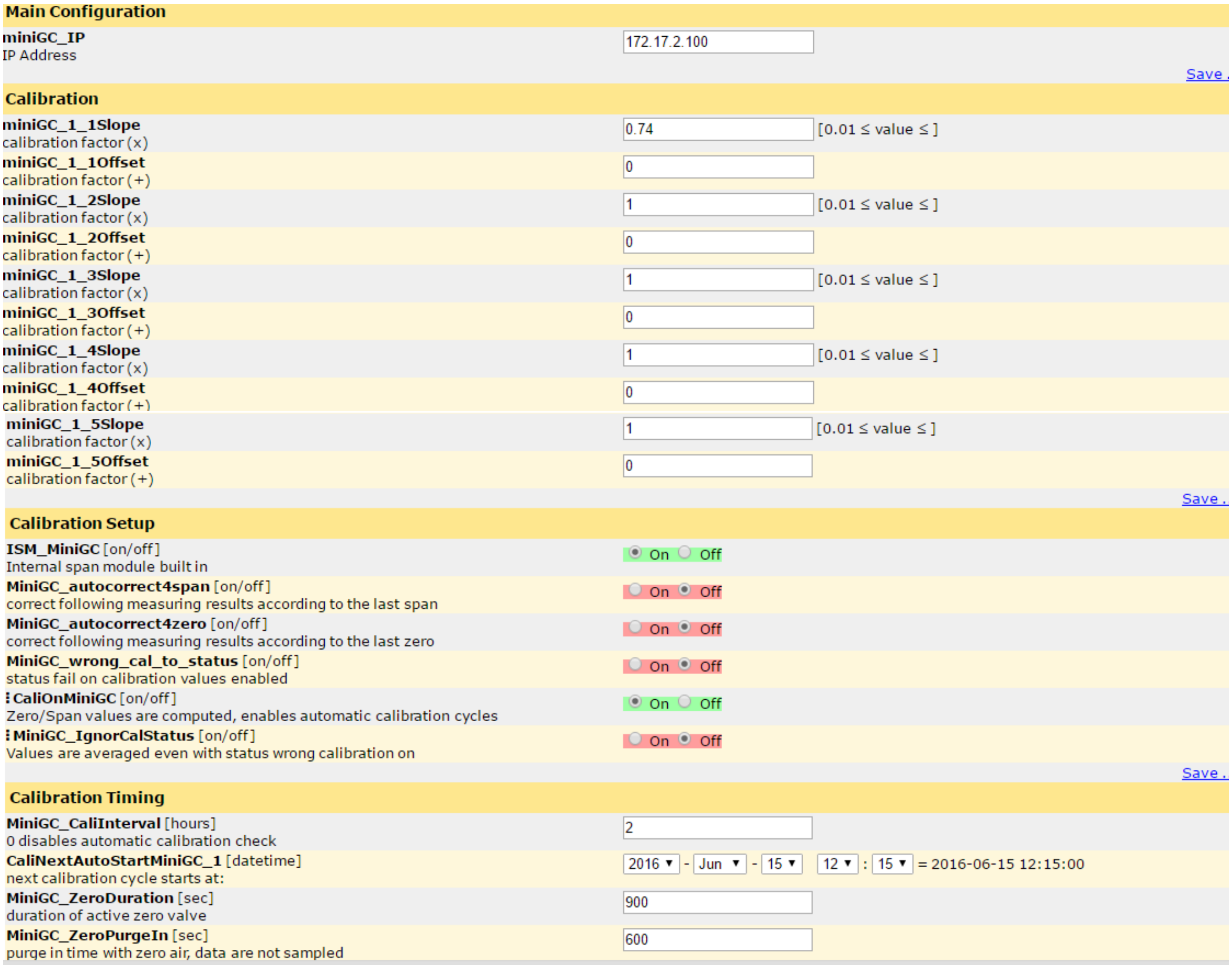
Figure 16 below shows the parameters you can configure on the BTEX-GC. Contrary to most airpointer modules, the calibration factors are not automatically adjusted after a calibration, but need to be inserted manually in the fields 'slope' and 'offset' of the 'calibration' section.

The interval between calibrations and their durations can be configured in the 'calibration timing' section.

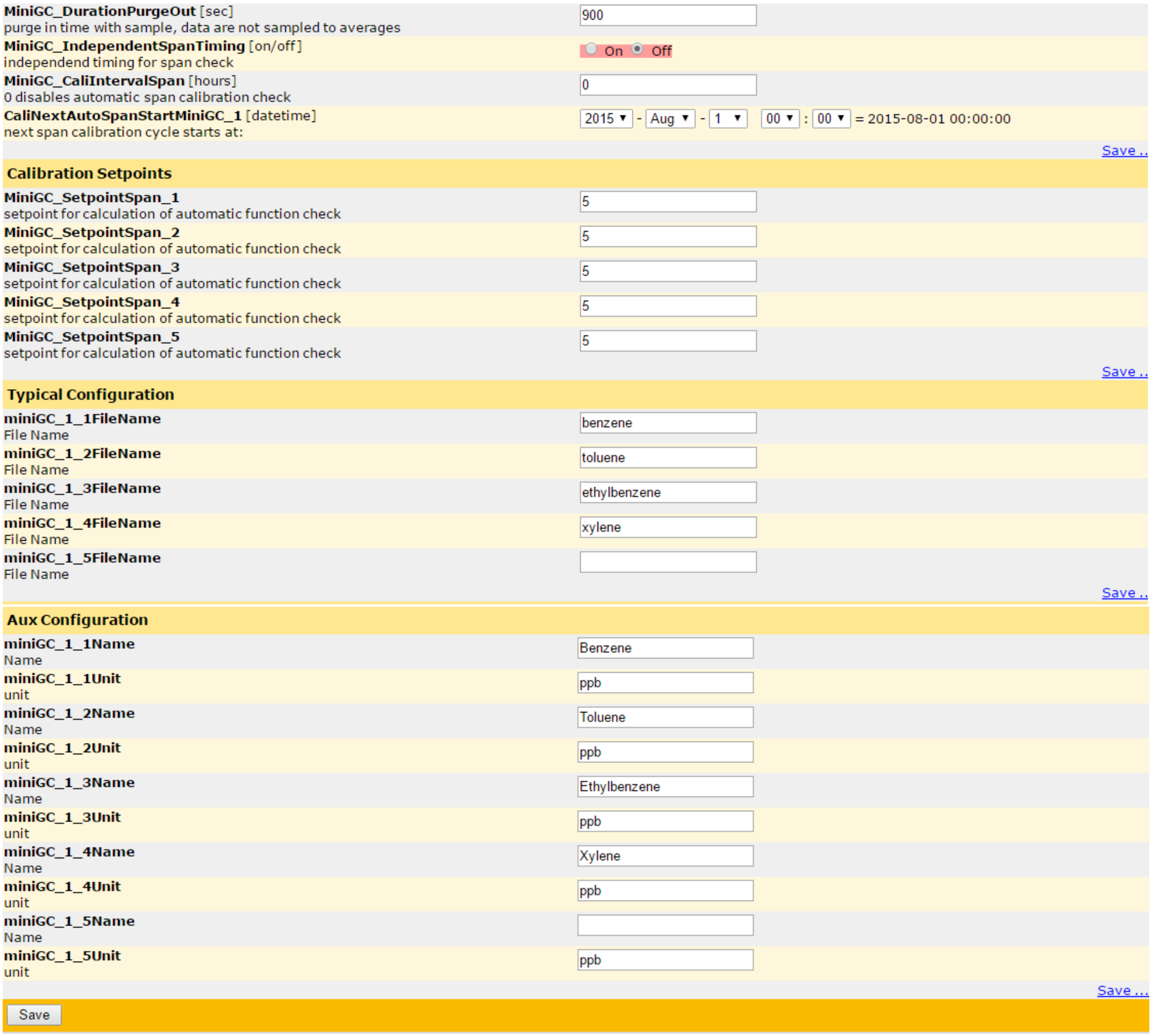
The concentration used for calibration can be configured in the 'calibration setpoint' section.

The name of the species can be configured in the 'typical configuration' section.

The units can be configured in the 'aux configuration' section.



Parameter	Value	Range
Main Configuration		
miniGC_IP IP Address	172.17.2.100	
		Save .
Calibration		
miniGC_1_1Slope calibration factor (x)	0.74	[0.01 ≤ value ≤]
miniGC_1_1Offset calibration factor (+)	0	
miniGC_1_2Slope calibration factor (x)	1	[0.01 ≤ value ≤]
miniGC_1_2Offset calibration factor (+)	0	
miniGC_1_3Slope calibration factor (x)	1	[0.01 ≤ value ≤]
miniGC_1_3Offset calibration factor (+)	0	
miniGC_1_4Slope calibration factor (x)	1	[0.01 ≤ value ≤]
miniGC_1_4Offset calibration factor (+)	0	
miniGC_1_5Slope calibration factor (x)	1	[0.01 ≤ value ≤]
miniGC_1_5Offset calibration factor (+)	0	
		Save .
Calibration Setup		
ISM_MiniGC [on/off] Internal span module built in	On Off	
MiniGC_autocorrect4span [on/off] correct following measuring results according to the last span	On Off	
MiniGC_autocorrect4zero [on/off] correct following measuring results according to the last zero	On Off	
MiniGC_wrong_cal_to_status [on/off] status fail on calibration values enabled	On Off	
CaliOnMiniGC [on/off] Zero/Span values are computed, enables automatic calibration cycles	On Off	
MiniGC_IgnorCalStatus [on/off] Values are averaged even with status wrong calibration on	On Off	
		Save .
Calibration Timing		
MiniGC_CaliInterval [hours] 0 disables automatic calibration check	2	
CaliNextAutoStartMiniGC_1 [datetime] next calibration cycle starts at:	2016 - Jun - 15 12 : 15	= 2016-06-15 12:15:00
MiniGC_ZeroDuration [sec] duration of active zero valve	900	
MiniGC_ZeroPurgeIn [sec] purge in time with zero air, data are not sampled	600	



MiniGC_DurationPurgeOut [sec] purge in time with sample, data are not sampled to averages	900
MiniGC_IndependentSpanTiming [on/off] independend timing for span check	On / Off (Off selected)
MiniGC_CaliIntervalSpan [hours] 0 disables automatic span calibration check	0
CaliNextAutoSpanStartMiniGC_1 [datetime] next span calibration cycle starts at:	2015 - Aug - 1 00 : 00 = 2015-08-01 00:00:00
	Save ..
Calibration Setpoints	
MiniGC_SetpointSpan_1 setpoint for calculation of automatic function check	5
MiniGC_SetpointSpan_2 setpoint for calculation of automatic function check	5
MiniGC_SetpointSpan_3 setpoint for calculation of automatic function check	5
MiniGC_SetpointSpan_4 setpoint for calculation of automatic function check	5
MiniGC_SetpointSpan_5 setpoint for calculation of automatic function check	5
	Save ..
Typical Configuration	
miniGC_1_1FileName File Name	benzene
miniGC_1_2FileName File Name	toluene
miniGC_1_3FileName File Name	ethylbenzene
miniGC_1_4FileName File Name	xylene
miniGC_1_5FileName File Name	
	Save ..
Aux Configuration	
miniGC_1_1Name Name	Benzene
miniGC_1_1Unit unit	ppb
miniGC_1_2Name Name	Toluene
miniGC_1_2Unit unit	ppb
miniGC_1_3Name Name	Ethylbenzene
miniGC_1_3Unit unit	ppb
miniGC_1_4Name Name	Xylene
miniGC_1_4Unit unit	ppb
miniGC_1_5Name Name	
miniGC_1_5Unit unit	ppb
	Save ...
Save	

FIGURE 16: CONFIGURATION OF THE BTEX-GC

7- Calibration of the device

Like on all other gas modules in the Recordum airpointer, calibration is very important. The user should choose the calibration interval depending on the local regulation, or whenever a drift that exceeds a defined threshold is visible in the automatic zero or span check. Recordum recommends a calibration at least once a year even if the automatic zero and span checks show stable readings. If the BTEX-GC module was purchased with the optional Internal Span Module (ISM), automatic zero and span checks can easily be performed daily. Please be aware that a calibration of the instrument needs to be performed with an external gas source to fulfil the needs of quality assurance. Technically, a calibration can also be done with the built-in ISM when installed. In this case, flow, temperature and permeation rate of the permeation tube need to be checked and taken into account to calculate the concentration.

The signals of the BTEX-GC module are computed with the calibration factors the following way:

Concentration = Conc * Factory factor * Slope – Offset

The calibration philosophy is identical to the one described in 'Chapter 7.6 - Calibration' of the airpointer manual, but for the BTEX module, the slope and offset values must be entered manually in the user interface. For this, go to setup/configuration/Pyxis BTEX/, as shown in *Figure 16*.

Factory Factor

The Factory factor is set during the first calibration with span gas. If the slope factor is more than 25% away from 1.0, a factory factor should be defined and put into the system and should stay the same during the instrument's life. It is calculated the same way as the slope but rounded up to the nearest tenth (e-g: if your result is 2.53 use a factory factor of 2.5).

Calibration at span and zero level

Span Calibration:

Supply the span gas to the calibration inlet, making sure there is an excess of gas. The airpointer sampling system will act as a bypass (vent). The typical span concentration for ambient air monitoring should be around 10ppb. Let the instrument run for at least one complete analysis cycle. We recommend running more 4 samples and to discard the first one. Check on the chromatograms that the peaks are clearly separated and unambiguously identified. When this is the case, find the actual value by averaging the concentration values of the last 3 samples and use the following formula to calculate the new slope:

New Slope = Setpoint / Actual value * Old slope

Example:

Setpoint: 10 ppb

Actual: 9,5 ppb

Slope: 1,12

New Slope = 10 / 9,5 * 1,12 = 1,179

Zero Calibration:

Because of the measuring method, an offset value is not really needed. We added it nonetheless because some customers want to have it. In most situations, the value shown on zero shows the quality of the zero air and less the real zero value. The recommendation is to use an offset factor of 0.

If you need to do a zero calibration, supply zero air to the calibration inlet, making sure there is an excess of gas. We recommend running more 4 samples, discarding the first one, and averaging the last 3. Once you obtained the actual values, you can calculate the new offset value using the following formula:

New Offset = Actual value + Old Offset

Glossary:

Function check: Typically, automatic performed zero and span check with the internal zero and span source. When the results are out of limit, a calibration should be performed.

Calibration: Manually performed zero and span check with an external gas source. Slope and offset factors are updated to match the set points.

Factory factor: Coarse adjustment to reach a Slope of 1

Slope: Multiplicative calibration factor

Offset: Additive calibration factor

Calibration of the retention times

The retention times are factory settings, optimized by Recordum before delivery of the device, and under normal conditions, the retention times should be stable. If they aren't, it is most probably due to a flow problem and the BTEX module needs maintenance. Therefore, the user normally doesn't need to modify the retention times.

However, after several years of operation, the retention times may drift slightly. Also, the user's needs may change, and they may want to analyze different compounds.

In these cases, a retention time recalibration is needed. This can be done in Setup/Configuration/Pyxis Timings, as shown in *Figure 17*. The meaning of the different timing parameters is explained in *Figure 11.*

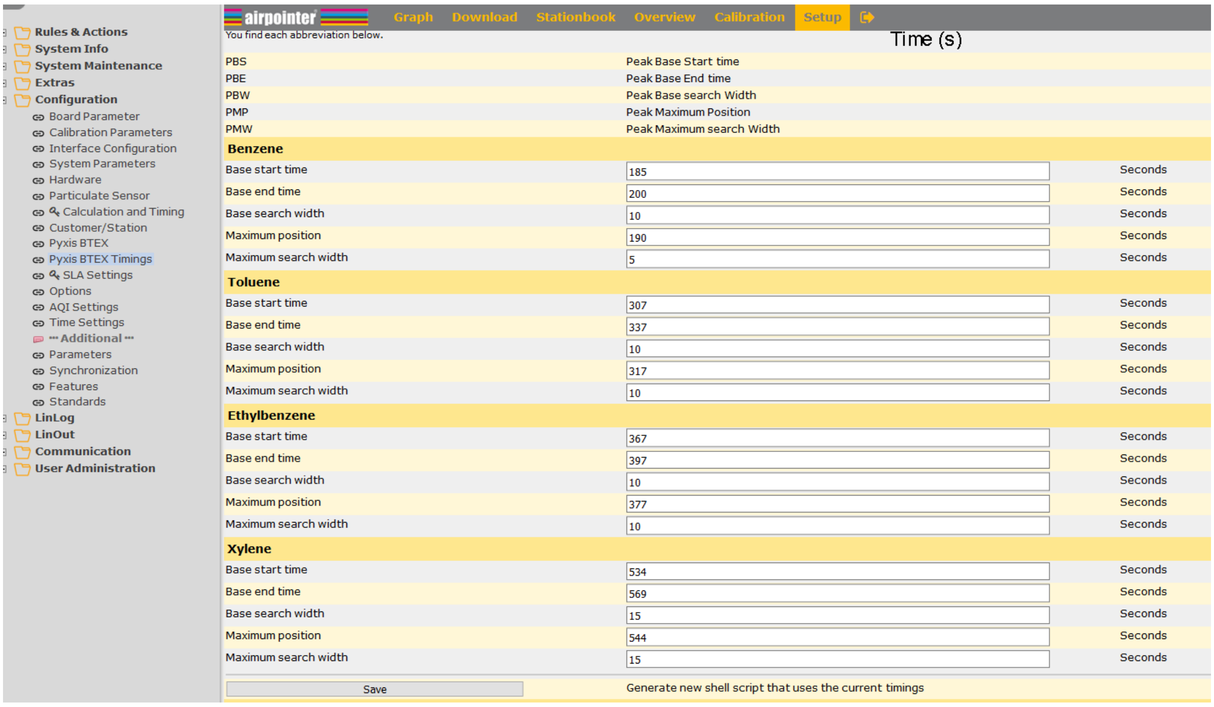


FIGURE 17: SETTING THE RETENTION TIMES

8- Troubleshooting

Sensitivity loss: perform a leak check, check that the perm tube is not empty or broken, clean the lamp, replace the lamp (see chapter 9 for indications on how to replace the PID lamp).

Sample flow: check that the air filter and the sample filter are not polluted, check with an external calibrated flowmeter (with a working range of 0-1000 ml/min) that the flow reading from the "sample" flow sensor is accurate.

Sensor flow: check that the air filter and the sample filter are not polluted, check with an external calibrated flowmeter (with a working range of 0-100 ml/min) that the flow reading from the "sensor" flow sensor is accurate.

Sample press: check with an external calibrated barometer that the pressure sensor reading is accurate.

PermT: check with an external calibrated thermometer that the temperature sensor is working, check that the fans are working and clean.

9- Maintenance

Maintenance schedule

air zero adsorbent	Once to twice a year
air zero filter	Once to twice a year
lamp change	Once to twice a year
Capillary inspection	every year
Perm tube change	every year

Maintenance procedures

Maintenance procedures can be viewed or downloaded using the links below:

https://www.airpointer.tech/wp-content/uploads/2019/02/BTEX1-Replacing-the-GC.pdf

https://www.airpointer.tech/wp-content/uploads/2019/02/BTEX2-Replacing-the-PID-lamp.pdf

https://www.airpointer.tech/wp-content/uploads/2019/02/BTEX3-Replacing-the-carrier-gas-filter.pdf