

Landesanstalt für Umwelt Baden-Württemberg



# Type test of the gas chromatograph

# GCX55-601 (PID) for Benzene

# Synspec B.V.

₩ LUBW-Report number: SYN143-E01/22E



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EMAS GEPRÜFTES UMWELTMANAGEMENT

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# **1** Summary of the test results

# **1.1** Tabular summary of the test results

Minimum requirement	Requirement	Test result	Req. fulfilled	Test date
Measuring value display	Measuring value display shall be availa- ble.	Measured value display is available.	Yes	2022
Easy maintenance	Maintenance shall be easy and possi- ble, preferably from outside of the de- vice.	The device does not require any maintenance to be carried out regularly by the user.	Yes	2022
Functional check	Equipment that is necessary for the de- vice operation shall be considered as a part of it and shall be applied in the cor- responding sub-tests.	The tested devices do not have test gas generator, so internal function control is not provided. Test is not ap- plicable.		2022
Set-up and run-in time	Set-up and run-in time shall be specified in the operating manual.	The operation manual contains de- tailed description of the commission- ing. The set-up and run-in times are specified in the operation manual.	Yes	2022
Design of the measuring device	The operating manual shall include in- formation about the design of the meas- uring device.	Information on the design is available in the manual operation.	Yes	2023
Unauthorized adjustment	It shall be possible to secure the meas- uring device against the unintended ad- justment.	The measuring device has a pass		2022
Data output	The measuring device shall provide dig- ital and/or analogue data output.	Measuring signals, status signals and error signals can be communicated over a serial port.	Yes	2022
Digital interface	The digital interface shall allow the transmission of output data, status signals and information like instrument type, measurement range, measured component and unit. The digital interface shall be described fully in respective standards and guidelines.	ion of output data, status sig- nformation like instrument usurement range, measured nt and unit. The digital inter- be described fully in respec-During the field test, the devices were connected to the LUBW's monitoring network center. The respective oper- ating states as well as error mes- sages were transmitted telemetrically.		2022
Data transmission protocol	The measuring device shall contain at least one data transmission protocol.			2022
Measuring range	The upper end of the measuring range shall be greater or equal to the upper limit of the certification range.	- Under end of the measuring range		2022
Negative output signals	Negative output signals shall not be dis- abled (live zero point).	The device is not equipped with a live zero point.		2022

Minimum requirement	Requirement	Test result	Req. fulfilled	Test date
Power failure	Uncontrolled discharge of operating and calibration gases shall be prevented. The device parameters shall be secured by buffering. The measurement shall continue when the power is restored.	d. cured all boots the operating system and starts		2022
Operating modus	Monitoring by telemetrically transmitta- ble data shall be possible.	Status signals and error messages can be transmitted via a serial interface.	Yes	2023
Switch-over	Switching between measurement and functional test and/or calibration shall be possible.	The device has no facilities for calibra- tion gas generation, so an internal function control is not provided.		
Device software	The measuring device shall be able to display the software version.	The current software versions for the control of the gas chromatograph can be displayed: <u>Software for GC control:</u> Version 6.4.2.19	Yes	2023
Short-term drift (Requirement of DIN EN 14662-3)	The short-term drift $D_{s,s}$ at test value 2 under laboratory conditions and over a drift period of 12 h shall be less than or equal to 2.0 µg/m <sup>3</sup> .	$\begin{array}{ll} \mbox{The short-term drift } D_{s,s} \mbox{ at test value 2} \\ \mbox{was determined to:} \\ \mbox{GC 80001:} & D_{s,s} = 0.32 \ \mbox{\mug/m^3} \\ \mbox{GC 80004:} & D_{s,s} = 0.43 \ \mbox{\mug/m^3} \end{array}$	Yes	2022
Repeatability standard devia- tion at test value 1 (Requirement of DIN EN 14662-3)	The repeatability standard deviation $s_{r,z}$ at test value 1 shall be less than or equal to 0.20 µg/m <sup>3</sup> .	$\label{eq:standard} \begin{array}{ll} \mbox{The repeatability standard deviation $s_{r,z}$} \\ \mbox{at test value 1 was determined to:} \\ \mbox{GC 80001:} & $s_{r,z}$ = 0.01 \ \mu g/m^3$ \\ \mbox{GC 80004:} & $s_{r,z}$ = 0.01 \ \mu g/m^3$ \\ \end{array}$	Yes	2022
Repeatability standard devia- tion at limit value (Requirement of DIN EN 14662-3)	The repeatability standard deviation $s_{r,ct}$ at limit value shall be less than or equal to 0.25 µg/m <sup>3</sup> .	The repeatability standard deviation $s_{r,ct}$ at limit value was determined to: GC 80001: $s_{r,ct} = 0.02 \ \mu g/m^3$ GC 80004: $s_{r,ct} = 0.02 \ \mu g/m^3$	Yes	2022
Linearity (Lack-of-fit) (Requirement of DIN EN 14662-3)	The residual $r_z$ at zero shall be less than or equal to 0.5 µg/m <sup>3</sup> . The maximum relative residual $r_{max}$ from the linear regression function shall be less than or equal to 5 %.	The residual $r_z$ at zero was determined to: GC 80001: $r_z = 0.5 \ \mu g/m^3$ GC 80004: $r_z = 0.3 \ \mu g/m^3$ The maximum relative residual $r_{max}$ was determined to: GC 80001: $r_{max} = -2.1 \ \%$ GC 80004: $r_{max} = -3.0 \ \%$	Yes	2022
Sensitivity coefficient of sam- ple gas pressure (Requirement of DIN EN 14662-3)	The sensitivity coefficient of sample gas pressure $b_{gp}$ at test value 2 shall be less than or equal to 0.10 (µg/m <sup>3</sup> )/kPa.	The sensitivity coefficient of sample gas pressure $b_{gp}$ at test value 2 was determined to: GC 80001: $b_{gp} = 0.07 \ (\mu g/m^3)/kPa$ GC 80004: $b_{gp} = 0.06 \ (\mu g/m^3)/kPa$	Yes	2022

Minimum requirement	Requirement	Test result	Req. fulfilled	Test date
Sensitivity coefficient of surrounding temperature (Requirement of DIN EN 14662-3)	The sensitivity coefficient of surrounding temperature $b_{st}$ at test value 2 shall be less than or equal to 0.08 (µg/m <sup>3</sup> )/K.	The sensitivity coefficient of surround- ing temperature $b_{st}$ at test value 2 was determined to: GC 80001: $b_{st} = 0.03 (\mu g/m^3)/K$ GC 80004: $b_{st} = 0.06 (\mu g/m^3)/K$	Yes	2022
Sensitivity coefficient of supply voltage (Requirement of DIN EN 14662-3)	The sensitivity coefficient of supply voltage $b_V$ at test value 2 shall be less than or equal to 0.08 (µg/m <sup>3</sup> )/V.	The sensitivity coefficient of supply voltage $b_V$ at test value 2 was deter- mined to: GC 80001: $b_V = 0.001 (\mu g/m^3)/V$ GC 80004: $b_V = 0.002 (\mu g/m^3)/V$	Yes	2022
Influence of water vapor (Requirement of DIN EN 14662-3)	The sensitivity coefficient $b_{H2O}$ at limit value and H <sub>2</sub> O concentration of approx. 19 mmol/mol shall be less than or equal to 0.015 (µg/m <sup>3</sup> )/(mmol/mol).	H <sub>2</sub> O concentration of approx. mol shall be less than or equal $b_{120} = 0.012 (\mu g/m^3)/(mmol/mol)$		2022
Influence of organic compounds (Requirement of DIN EN 14662-3)	The contribution of organic compounds $\Delta x_{OC}$ to the measurement of benzene concentration at limit value shall be less than or equal to 0.25 µg/m <sup>3</sup> .	to the measurement of benzenepounds $\Delta x_{oc}$ was determined to:ncentration at limit value shall be lessGC 80001: $\Delta x_{oc} = -0.09 \ \mu g/m^3$		2022
Memory effect (Requirement of DIN EN 14662-3)The benzene concentration of the first zero gas analysis following the analysis of the test gas at test value 3 shall be less than or equal to 1.0 $\mu$ g/m³.The mean value c <sub>m</sub> of the first zero gas measurement was determined to: GC 80001: c <sub>m</sub> = 0.1 $\mu$ g/m³ GC 80004: c <sub>m</sub> = 0.3 $\mu$ g/m³		Yes	2022	
Long-term drift (Requirement of DIN EN 14662-3)	(Requirement of $D_{l,z}$ at zero shall be less than or equal to $C = 00004$ . $D_{l,z} = -0.02 \mu g/m$		Yes	2023
Reproducibility standard devi- ation under field conditions (Requirement of DIN EN 14662-3)	The reproducibility standard deviation der field conditions $s_{r,f}$ of two measur- g devices shall be less than or equal 0.25 µg/m <sup>3</sup> . The reproducibility standard deviation under field conditions $s_{r,f}$ of the two measuring devices was determined to: $s_{r,f} = 0.17 \mu g/m^3$		Yes	2023
Maintenance interval	At least 14 days, preferably three months.	The maintenance interval is three months.	Yes	2023

Minimum requirement	Requirement	Test result	Req. fulfilled	Test date
Availability	At least 90 %.	The availability was determined to:GC 80001:100 %GC 80004:100 %	Yes	2023
Expanded uncertainty	The determined value must not exceed the specifications of the EU directives on air quality. For benzene, the relative expanded uncertainty must be less than or equal to 25%.	The determined value for the relative expanded measurement uncertainty W is: GC 80001: W = 15.1 % GC 80004: W = 12.5 %	Yes	2023

- Test value 1: 1/10 of the annual limit value =  $0.5 \ \mu g/m^3$
- Test value 2: 70 80 % of the maximum of the certification range
- Test value 3: 90 % of the maximum of the certification range
- Limit value: Annual limit value of benzene =  $5 \mu g/m^3$

# **1.2** Announcement proposal

Based on the positive results obtained, the following recommendation is made for the announcement as a type tested measuring device:

#### Measuring device:

Gas chromatograph GCX55-601 Version PID

# Manufacturer:

Synspec B.V., Groningen, the Netherlands

#### **Approval:**

For the continuous detection of benzene immission concentrations in ambient air in stationary use.

#### Measuring ranges during the performance test:

Component	Certification area	Unit
Benzene	0 – 50	$\mu g/m^3$

# Software version:

6.4.2.19

# Limitations:

None

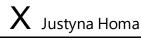
#### Notes:

- 1. The measuring device is not equipped with a live zero point.
- 2. The performance test report can be viewed on the internet at www.qall.de.

# **Test report:**

Landesanstalt für Umwelt Baden-Württemberg (LUBW), Karlsruhe Report no. SYN143-E01/22E from 05 April 2023 Testing and reporting

11.04.2023



Dr. Justyna Homa

Signiert von: Homa (LUBW)

Release of the test report

11.04.2023

X Michael Maban

Michael Maban Sachgebietsleiter / Fachlich Verantwortlicher Signiert von: Maban (LUBW)

# 2 Introduction

In a letter, dated 31-08-2020 the company Synspec B.V. give an order to the LUBW to test the gas chromatograph GCX55 version PID for its suitability for measuring ambient concentrations of benzene.

Due to the corona pandemic, the tests could be started first in August 2022. Synspec B.V. provided two gas chromatographs with the following serial numbers:

- GC 1: 80001 (hereinafter referred to as GC 80001)
- GC 2: 80004 (hereinafter referred to as GC 80004).

The tests were carried out according to the specifications of the following guidelines and standards:

#### VDI-Guideline 4202 part 1 (April 2018)

Automatic measuring systems for air quality monitoring – Performance test, declaration of suitability and certification of point-related measuring systems for gaseous air pollutants.

#### DIN EN 14662-3 (February 2016)

Ambient air - Standard method for the measurement of benzene concentrations - Part 3: Automated pumped sampling with in situ gas chromatography.

#### **Measuring device:**

Gas chromatograph GCX55-601 version PID (serial no. 80001 and 80004)

Fig. 2.1 and Fig. 2.2 show the front and rear views of the GCX55.



Fig. 2.1: Front view of the gas chromatograph



Fig. 2.2: Rear view of the gas chromatograph

# **3** Working principle of the gas chromatograph

The GCX55 gas chromatograph is the successor of the GC955. It was developed for the quasi-continuous measurement of organic air pollutants. It allows the detection of low ppb concentrations of e.g. benzene, toluene, ethylbenzene, m/p-xylene and o-xylene in the ambient air.

The GCX55 has a sample pre-concentration of the pollutants to be monitored. At the beginning of each sampling, the sample gas lines are purged by a bypass pump. The sample is then drawn onto an enrichment tube by sampling cylinder. As soon as there is enough sample, the tube is heated and the components are thermally desorbed. The carrier gas (nitrogen) flows through the tube in countercurrent. The organic compounds enter a separation column consisting of two parts: a stripping column and an analysis column. In the stripping column the components start to separate - the components with the lowest boiling points enter the analytical column first. After all components of interest have left the stripping column, the column is purged of components of no interest by switching the 10-way valve in the opposite direction. In this way, a short analysis time can be realized (15 minutes) because it is not necessary to wait for the elution of all compounds.

The organic compounds are quantified by a photoionization detector (PID). In the photoionization detector, electrons are excited by radiation energy during the measurement until they are separated from their atom or molecule. The energy source for the ionization is a discharge lamp filled with inert gas under low pressure, which generates a stable monochromatic energetic light flux of photons. The UV lamp is installed gas-tight in the detector. The light source is separated from the carrier gas flow by a window through which UV light can pass. The measurement signals are registered with an UV detector. The signals are amplified, digitized and stored.

The GCX55 gas chromatograph is equipped with a standard industrial PC with a Windows operating system. All data are stored on the internal hard disk and can be retrieved at any time. The GCX55 is controlled by the GCX55.exe software. The current chromatogram can be followed in real time on the LCD screen. The peak area, peak height and concentration are calculated and displayed for the components. Furthermore, the saved chromatograms can be called up in a menu and the settings of the control program can be changed.

The GCX55 has electronic sensors for monitoring e.g. the carrier gas flow, the oven temperature and the pressure for the valve circuit. The values measured are continuously displayed on the screen and stored in the chromatogram data.

# Technical data:

General:	
Tested software	6.4.2.19
Housing	19"-rack
Height	5 HE (224 mm)
Depth (without handles)	552 mm
Width (housing)	430 mm
Width (front panel)	483 mm
Weight	19.5 kg
Operating temperature	0 °C to 30 °C
Humidity	0 % to 95 % relative humidity, non-condensing
Instrument supply:	
Voltage	230 VAC, 115 VAC (on request)
Power consumption	500 VA (commissioning, 1 Minute), 300 VA (operation)
Carrier gas	Nitrogen, 4 to 5 bar, 15 ml/min
Gas ports	Swagelok 1/8" fittings
	Calibration gas BTX, pressureless, $\geq$ 30 ml/min
	Optional: zero air for calibration, 4 to 5 bar, 50 ml/min
Detector:	
Туре	PID – Photoionization detector
Commilia or	
Sampling:	
Sampling	Sampling cylinder
Flow rate control	Thermal mass flow controller
	Via sampling cylinder, with pressure and temperature correction
Cycle time	15 min
Flow rate	max. 30 ml/min (flushing of the internal sample line)
Communication and the second	15 ml/min (operation)
Sample volume	35 ml
Sample pre-concentration:	
Technology	Enrichment tube with Graphsphere 2016
Enrichment temperature	Room temperature, $\leq 40$ °C
Desorption temperature	300 °C
Heating rate	25 °C/s
-	

Specifications:	
Gas line switching	10-port valve
Analytical oven	Isothermally set to 70 $^{\circ}$ C
Communication:	
Interfaces	4x RS232
PC ports	4x USB 2.0, 2x Ethernet 1 Gb/s, VGA
Analog outputs	7 digital outputs (including one potential-free relay)
	4 analog outputs
	Optional: E/A-board
Protocols	ASCII (Synspec own protocol)
	Bayern-Hessen, Gesytec
	Modbus

# 4 Test program

# 4.1 Laboratory tests

The two gas chromatographs from Synspec B.V. (serial no. 80001 and 80004) were installed in a 19" rack in the LUBW test laboratory. To carry out the laboratory tests, a gas mixing system (according to the specifications of DIN EN ISO 6145-6 "Gas analysis - Preparation of calibration gas mixtures using dynamic-volumetric methods - Part 6: Critical orifices") and a catalytic zero air generator from Headline-Filters (Air Purifier CAP 60) were used. Using the gas mixing system, a BTX gas from a traceable test gas cylinder (NPL, No.: NPL-D711530) with a benzene concentration of 16.87 mg/m<sup>3</sup> was diluted with zero air to a concentration range of  $1.3 - 54.3 \mu g/m^3$ . The desired dilution was set by varying the two volume flows (zero air and benzene gas). The exact determination of these volume flows was performed with a certified Volumeter from DryCal.

The zero air generator was used to produce the zero air (purified compressed air) for the gas mixing system and some of the measurements.

Before starting the laboratory tests, an auto-linearization was performed. This function is specifically for the PID and runs for several hours. A concentration of approx. 25  $\mu$ g/m<sup>3</sup> was specified. Several concentration levels are automatically generated to achieve a linearization of the PID. Subsequently, a calibration of the gas chromatographs was carried out. The calibration function was determined with five calibration points in a concentration range from 0  $\mu$ g/m<sup>3</sup> to 54.3  $\mu$ g/m<sup>3</sup> and stored in the instruments by the manufacturer.

To carry out the temperature tests, the two test instruments were installed in a climate chamber Clime-Event C/340/40/3 from Weisstechnik.

# 4.2 Field test

To carry out the field test, the two gas chromatographs were installed and calibrated in an air-conditioned measuring station ( $T = 22^{\circ}C \pm 3^{\circ}C$ ) on the test site opposite the LUBW building close to a road with considerable traffic volume (motorway feeder) (see Fig. 4.1).



Fig. 4.1: Measuring station at the LUBW test site

The devices were installed in a 19" rack and connected to the sampling system (sampling tube with a standardized sampling head on the roof) of the station. This ensured representative measurements of the same sample air volume.

For the function check, which shall be carried out by manual switching between zero air and test gas, a gas mixing system was installed.

The average benzene concentrations measured on the test site are between 1 and 3  $\mu$ g/m<sup>3</sup>. To determine the reproducibility standard deviation or to measure higher benzene concentration, a capillary dosing device (glass container with a capillary opening) filled with BTX was fixed below the sampling head of the sampling system. Depending on the ambient temperature and air pressure, a certain amount of BTX was emitted, which was randomly added to the sample airflow depending on the wind direction. In this way, benzene concentrations up to 20  $\mu$ g/m<sup>3</sup> could be measured.

For data transfer to the LUBW monitoring network center, the instruments were connected to the station computer via the internal interface RS-232, which is equipped with a data acquisition system. For the communication between the gas chromatographs and the station computer, the "Bayern-Hessen Protocol" was used.

# 5 Reference measurement methods

The test gases used for the performance test of the gas chromatographs must be traced back to the reference standards.

For this performance test, a certified test gas cylinder from NPL (National Physical Laboratory, national reference laboratory in England) with a volume of 10 l was used. The concentrations of benzene, toluene, ethylbenzene and o-xylene were around 5-ppm and the concentration of m/p-xylene was around 10-ppm.

The NPL test gas cylinder was connected to a gas mixing system, which is a dynamic and continuously working dilution system, based on DIN EN ISO 6145. By its use, different test gas concentrations can be produced by mixing a small flow rate of the certified test gas  $Q_{PG}$  (approx. 0.5 - 8 ml/min) with a zero air flow  $Q_{GG}$  (approx. 2.3 - 6.7 l/min).

)

The concentration of the generated test gas is then calculated as follows:

$$\boldsymbol{c} = \boldsymbol{c}_{\boldsymbol{P}\boldsymbol{G}} * \frac{\boldsymbol{Q}_{\boldsymbol{P}\boldsymbol{G}}}{\boldsymbol{Q}_{\boldsymbol{P}\boldsymbol{G}} + \boldsymbol{Q}_{\boldsymbol{G}\boldsymbol{G}}} \tag{Eq. 5.1}$$

Where:

c generated test gas concentration, in  $\mu g/m^3$ ;

c<sub>PG</sub> concentration of the certified test gas cylinder, in mg/m<sup>3</sup>;

Q<sub>PG</sub> flow rate of the certified test gas, in Nml/min;

Q<sub>GG</sub> flow rate of the zero air, in Nml/min.

Varying the two flow rates  $Q_{PG}$  and  $Q_{GG}$  is done by changing the corresponding upstream pressure in front of the two critical orifices using precision pressure regulators. The procedure is carried out according to the specifications of the quality management system in the laboratory according to DIN EN ISO/IEC 17025 (Technical Instructions TA 5-6-AM1-207 "Determination of the concentration of test gases for aromatic and chlorinated hydrocarbons").

# 6 Test results

# 6.1 Measured value display

# 6.1.1 Minimum requirement (VDI 4202 part 1 chapter 8.3.1)

The measuring system shall have a measured value display.

# 6.1.2 Equipment

No additional equipment is required.

#### 6.1.3 Testing

It shall be checked whether the measuring device has a measured value display.

#### 6.1.4 Evaluation

The operability of the measured value display shall be checked.

# 6.1.5 Assessment

Minimum requirement met? yes

#### 6.1.6 Documentation

The gas chromatograph is equipped with a standard industrial PC with a Windows operating system. A complete PC structure is available for processing the measurement results. All data are stored to the internal SSD and can be recalled at any time. On an LCD screen, the current or the saved chromatogram are displayed.

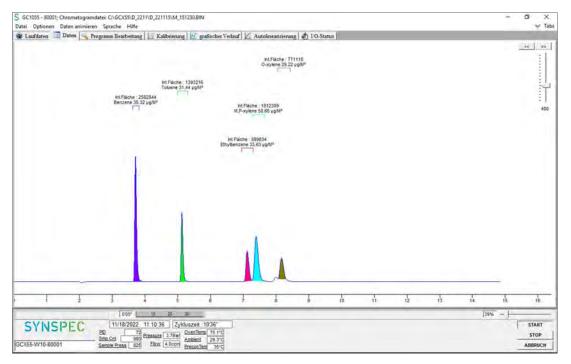


Fig. 6.1: Display of a chromatogram on the LCD screen

# 6.2 Calibration inlet

# 6.2.1 Minimum requirement (VDI 4202 part 1 chapter 8.3.2)

The measuring system may have a test gas inlet separate from the sample gas inlet.

#### 6.2.2 Equipment

No additional equipment is required.

#### 6.2.3 Testing

It shall be checked whether the measuring device has a test gas inlet separate from the sample gas inlet.

#### 6.2.4 Evaluation

See Chap. 6.2.5

#### 6.2.5 Assessment

The tested device does not have a separate test gas inlet for the application of the calibration gas.

Minimum requirement met? not applicable

#### 6.2.6 Documentation

Not applicable

# 6.3 Easy maintenance

#### 6.3.1 Minimum requirement (VDI 4202 part 1 chapter 8.3.3)

The necessary maintenance of the measuring system shall be performed in accordance with the operation manual and be possible without larger effort, preferably from outside of the instrument.

# 6.3.2 Equipment

No additional equipment is required.

#### 6.3.3 Testing

It shall be checked whether the necessary maintenance work on the measuring device is possible without larger effort.

#### 6.3.4 Evaluation

The measuring device does not require any maintenance to be carried out regularly by the user. Furthermore, a complete maintenance by the manufacturer is recommended once a year.

#### 6.3.5 Assessment

Minimum requirement met? yes

#### 6.3.6 Documentation

Not applicable

# 6.4 Functional check

# 6.4.1 Minimum requirement (VDI 4202 part 1 chapter 8.3.4)

See Chap. 6.4.5

# 6.4.2 Equipment

No additional equipment is required.

# 6.4.3 Testing

It shall be checked whether the test gas generator can be controlled telemetrically.

#### 6.4.4 Evaluation

See Chap. 6.4.5

#### 6.4.5 Assessment

The measuring device does not have a test gas generator, so internal function control is not provided.

Minimum requirement met? not applicable

#### 6.4.6 Documentation

Not applicable

# 6.5 Set-up and run-in times

# 6.5.1 Minimum requirement (VDI 4202 part 1 chapter 8.3.5)

The set-up and run-in times of the measuring system shall be specified in the operation manual.

#### 6.5.2 Equipment

Testing requires:

• Clock

#### 6.5.3 Testing

The device shall be commissioned according to the operation manual. The required set-up and run-in times shall be measured separately.

# 6.5.4 Evaluation

The required set-up and run-in times shall be documented.

#### 6.5.5 Assessment

Minimum requirement met? yes

#### 6.5.6 Documentation

Set-up time:

The set-up time is about 1 hour

Before starting a measurement, the gas chromatograph must be installed in a 19" rack and the carrier gas  $N_2$  (quality 5.0) must be connected. The connections are made via Swagelok 1/8" fittings at the marked point on the rear of the instrument. A pressure of approximately 4 - 5 bar must be set for the connected carrier gas.

#### Run-in time:

#### The run-in time is about 1 hour

When the device is switched on, the operating system and the chromatography software start automatically. During start-up, a self-test of the electronic is performed and the oven is heated up to the base temperature. Since the sample gas must first be preconcentrated on the enrichment tube, the first measurement results are available after the second measurement cycle.

# 6.6 Instrument design

### 6.6.1 Minimum requirement (VDI 4202 part 1 chapter 8.3.6)

The operation manual shall include information on the design of the measuring device.

#### 6.6.2 Equipment

Testing requires:

- AC meter
- Balance

#### 6.6.3 Testing

The design of the measuring devices shall be compared with the description in the operation manual. Furthermore, the installation position, safety requirements and dimensions as well as the weight including accessories shall be determined. The specified energy consumption shall be determined over 24 h during the field test.

### 6.6.4 Evaluation

The characteristics found shall be compared with the description in the operation manual.

#### 6.6.5 Assessment

Minimum requirement met? yes

#### 6.6.6 Documentation

Device dimensions:

19" housing with 5 HE, width 48.3 cm, height 20.4 cm, depth (without handles) 55.2 cm

**Operating voltage:** 

230 VAC

Weight:

20 kg

Power consumption:

1,7 kWh

# 6.7 Unauthorized adjustment

# 6.7.1 Minimum requirement (VDI 4202 part 1 chapter 8.3.7)

It shall be possible to secure the settings of the measuring system against unintended or unauthorized adjustment during operation.

# 6.7.2 Equipment

No additional equipment is required.

### 6.7.3 Testing

It shall be determined whether the adjustment can be changed despite the activated device protection (e.g. password).

#### 6.7.4 Evaluation

See Chap. 6.7.6

#### 6.7.5 Assessment

Minimum requirement met? yes

#### 6.7.6 Documentation

The software for controlling the gas chromatograph is protected by a password and cannot be changed accidentally.

# 6.8 Data output

# 6.8.1 Minimum requirement (VDI 4202 part 1 chapter 8.3.8)

The measuring signals shall be provided digitally (e.g. RS 232) and/or analog (e.g. 4 mA to 20 mA).

#### 6.8.2 Equipment

Testing requires:

• Computer for data transmission to the LUBW monitoring network center

# 6.8.3 Testing

It shall be checked whether the operating states and output signals are correctly recognized and registered by the evaluation system.

#### 6.8.4 Evaluation

An evaluation system shall be connected to the measuring device. The respective operating states and output signals are to be recorded and compared with the set values.

#### 6.8.5 Assessment

Minimum requirement met? yes

#### 6.8.6 Documentation

The output signals could be transmitted by "Bayern-Hessen-protocol". This was checked during the field test. There was a match between the transmitted data and the data stored in the measuring device (see Tab. 6.1).

 Tab. 6.1: Comparison between the data transmitted to the LUBW monitoring network center (MNZ) and the data stored in the gas chromatographs

Data/	Concentra-	GC-d	isplay	Data ii	n MNZ
Time	tion	GC 80001	GC 80004	GC 80001	GC 80004
03.01.2023 11:45		34.8 µg/m <sup>3</sup>	34.9 µg/m <sup>3</sup>	34.8 µg/m <sup>3</sup>	34.9 µg/m <sup>3</sup>
03.01.2023 12:00		34.5 µg/m <sup>3</sup>	35.6 µg/m <sup>3</sup>	34.5 µg/m <sup>3</sup>	35.6 µg/m <sup>3</sup>
03.01.2023 12:15	35.1 μg/m <sup>3</sup>	34.6 µg/m <sup>3</sup>	36.0 µg/m <sup>3</sup>	34.6 µg/m <sup>3</sup>	36.0 µg/m <sup>3</sup>
03.01.2023 12:30		34.7 µg/m <sup>3</sup>	36.2 µg/m <sup>3</sup>	34.7 µg/m <sup>3</sup>	36.2 µg/m <sup>3</sup>

# 6.9 Digital interface

#### 6.9.1 Minimum requirement (VDI 4202 part 1 chapter 8.3.9)

The digital interface shall allow the transmission of output signals, status signals and information like instrument type, measuring range, measured component and unit. The digital interface shall be described in the relevant standards and guidelines.

#### 6.9.2 Equipment

See Chap. 6.8 and Chap. 6.14

# 6.9.3 Testing

See Chap. 6.8 and Chap. 6.14

#### 6.9.4 Evaluation

See Chap. 6.8 and Chap. 6.14

#### 6.9.5 Assessment

Minimum requirement met? yes

#### 6.9.6 Documentation

See Chap. 6.8 and Chap. 6.14

# 6.10 Data transmission protocol

# 6.10.1 Minimum requirement (VDI 4202 part 1 chapter 8.3.10)

If the output signals of the device are transmitted digitally, it shall be checked whether the device has at least one data transmission protocol.

#### 6.10.2 Equipment

See Chap. 6.8 and Chap. 6.14

#### 6.10.3 Testing

See Chap. 6.8 and Chap. 6.14

#### 6.10.4 Evaluation

See Chap. 6.8 and Chap. 6.14

#### 6.10.5 Assessment

Minimum requirement met? yes

#### 6.10.6 Documentation

See Chap. 6.8 and Chap. 6.14

# 6.11 Measuring range

#### 6.11.1 Minimum requirement (VDI 4202 part 1 chapter 8.3.11)

The upper end of the measuring range of the measuring system shall be greater or equal to the upper limit of the certification range.

#### 6.11.2 Equipment

No additional equipment is required.

#### 6.11.3 Testing

The upper end of the measuring range shall be determined.

#### 6.11.4 Evaluation

Not applicable

#### 6.11.5 Assessment

Minimum requirement met? yes

#### 6.11.6 Documentation

The manufacturer does not define the upper limit of the measuring range. During the test, concentrations up to  $50 \ \mu g/m^3$  were abandoned.

# 6.12 Negative output signals

# 6.12.1 Minimum requirement (VDI 4202 part 1 chapter 8.3.12)

Negative measuring signals or measured values may not be suppressed (live zero).

#### 6.12.2 Equipment

No additional equipment is required.

#### 6.12.3 Testing

It shall be checked whether the displayed zero point is a live zero point and the device can display both positive and negative signals.

#### 6.12.4 Evaluation

See Chap. 6.12.5

# 6.12.5 Assessment

The device is not equipped with a live zero.

Minimum requirement met? not applicable

#### 6.12.6 Documentation

Not applicable

# 6.13 Failure in the mains voltage

# 6.13.1 Minimum requirement (VDI 4202 part 1 chapter 8.3.13)

In case of malfunction of the measuring system or failure in the mains voltage, uncontrolled emission of operation and calibration gas shall be prevented. The instrument parameters shall be secured by buffering against loss caused by failure in the mains voltage. When mains voltage returns, the instrument shall automatically reach the operation mode and start the measurement according to the operating manual.

#### 6.13.2 Equipment

No additional equipment is required.

#### 6.13.3 Testing

A simulated power failure shall be used to check whether the measuring device remains undamaged and is ready for a measurement again when the power is restored.

#### 6.13.4 Evaluation

The trouble-free operation of the device after power recovery shall be determined by comparing the device parameters before and after the power failure.

#### 6.13.5 Assessment

Minimum requirement met? yes

#### 6.13.6 Documentation

During the field test, a 10-minute power failure occurred. After power recovery, the computer automatically boots the operating system and starts a measurement. The supply of the operating gas  $(N_2)$  is stopped during a power failure.

# 6.14 **Operating states**

# 6.14.1 Minimum requirement (VDI 4202 part 1 chapter 8.3.14)

The measuring system shall allow the monitoring of its essential functions by telemetrically transmittable status signals.

# 6.14.2 Equipment

Testing requires:

• Evaluation or data recording system

#### 6.14.3 Testing

An evaluation or data recording system shall be connected to the measuring device. The corresponding operating states (normal operation, maintenance and malfunction) of the measuring system shall be set. A modem shall be connected to the measuring system. The status signals of the measuring system shall be determined by remote data recording.

#### 6.14.4 Evaluation

It shall be checked whether operating states set at the measuring system are correctly determined and recorded by the connected evaluation or data recording system.

#### 6.14.5 Assessment

Minimum requirement met? yes

#### 6.14.6 Documentation

During the field test, the two instruments were connected with the network monitoring center of LUBW. At the end of the field test, the respective operating states (operation and maintenance) were selected on the measuring devices. The instrument shows a plurality of operating states and error messages. Several functional errors (sampling, detector, temperature, flow, pressure, etc.) were simulated. Telemetric transmission of these operating states is guaranteed.

# 6.15 Switch-over

# 6.15.1 Minimum requirement (VDI 4202 part 1 chapter 8.3.15)

Switching between measurement and functional check and/or calibration shall be possible telemetrically or manually at the measuring system.

#### 6.15.2 Equipment

No additional equipment is required.

#### 6.15.3 Testing

It shall be checked whether manually or telemetrically switching between measurement and functional check and/or calibration is possible.

#### 6.15.4 Evaluation

See Chap. 6.15.5

#### 6.15.5 Assessment

The tested devices do not have a test gas generator, so internal function control is not provided.

Minimum requirement met? not applicable

#### 6.15.6 Documentation

Not applicable

# 6.16 Instrument software

#### 6.16.1 Minimum requirement (VDI 4202 part 1 chapter 8.3.16)

The measuring system shall be able to display the version of the instrument software.

#### 6.16.2 Equipment

No additional equipment is required.

#### 6.16.3 Testing

It shall be checked whether the version of the device software can be displayed. The version of the device software shall be documented.

#### 6.16.4 Evaluation

See chap. 6.16.3

#### 6.16.5 Assessment

Minimum requirement met? yes

#### 6.16.6 Documentation

By clicking on "Help" and "About" in the menu bar of the program, a window with the current software version for the control of the gas chromatograph appears (see Fig. 6.2).

- Software for GC control and communication with external devices: GCX55.exe
- Version 6.4.2.19

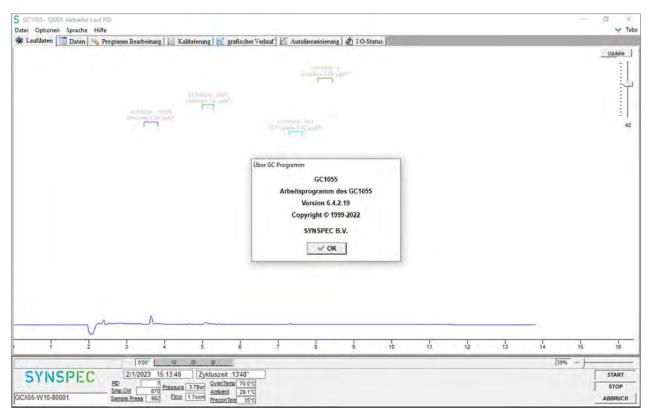


Fig. 6.2: Software version display

# 6.17 Short-term drift

# 6.17.1 Minimum requirement (DIN EN 14662-3: 2016-02 chapter 8.4.3)

The short-term drift  $D_{s,s}$  at test value 2 (approx. 70 – 80 % of benzene concentration of 50  $\mu$ g/m<sup>3</sup>) under laboratory conditions and over a drift period of 12 h shall be less than or equal to 2.0  $\mu$ g/m<sup>3</sup>.

# 6.17.2 Equipment

Testing requires:

• Test standards (e.g. zero air and test gas of suitable concentration)

#### 6.17.3 Testing

Test gas at test value 2 is applied to the device under laboratory conditions. The mean value is calculated from four successive measurements. After 12 hours of measuring the ambient air, the measurements are repeated at test value 2.

### 6.17.4 Evaluation

The short-term drift at test-value 2 is calculated as follows:

$$D_{s,s} = (x_{s,2} - x_{s,1})$$
(Eq. 6.1)

Where:

 $D_{s,s}$  the 12-h-drift at test value 2, in  $\mu g/m^3$ ;

 $x_{s,1}$  the mean value of four measurements at the beginning of the drift period, in  $\mu g/m^3$ ;

 $x_{s,2}$  the mean value of four measurements at the end of the drift period, in  $\mu g/m^3$ .

The result of the short-term drift is not included in the expanded measurement uncertainty.

#### 6.17.5 Assessment

Minimum requirement met? yes

#### 6.17.6 Documentation

To calculate the short-term drift under laboratory conditions, a concentration of approx.  $36.2 \ \mu g/m^3$  was offered to the two gas chromatographs over a period of 75 minutes (five measuring cycles). A mean value was calculated based on four measurements; the result of the first measurement was discarded. After 12 h the measurement was repeated and a mean value was again determined based on four measurements (see Tab. 6.2).

	GC 80001	GC 80004
$x_{s,1}$ , in $\mu g/m^3$	35.80	35.68
$x_{s,2}$ , in $\mu g/m^3$	35.48	35.25
D <sub>s,s</sub> , in µg/m <sup>3</sup>	0.32	0.43

**Tab. 6.2:** Calculated values for the short-time drift D<sub>s,s</sub>

# 6.18 Repeatability standard deviation

# 6.18.1 Minimum requirement (DIN EN 14662-3: 2016-02 chapter 8.4.4)

The repeatability standard deviation  $s_{r,z}$  at test value 1 (10 % of the annual limit value = 0.5 µg/m<sup>3</sup>) shall be less than or equal to 0.20 µg/m<sup>3</sup> and the repeatability  $s_{r,c(t)}$  at the annual limit value (= 5µg/m<sup>3</sup>) shall be less than or equal to 0.25 µg/m<sup>3</sup>.

#### 6.18.2 Equipment

Testing requires:

- Gas mixing system
- Test standards (e.g. test gas of suitable concentration)

### 6.18.3 Testing

Eleven measurements are performed with a test gas concentration at test value 1 and at the annual limit value.

# 6.18.4 Evaluation

From the last ten results, the repeatability standard deviation is calculated according to the following equation:

$$s_{r,c_t} = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}}$$
 (Eq. 6.2)

Where:

 $s_{r,ct}$  the repeatability standard deviation at  $c_t$ , in  $\mu g/m^3$ ;

 $x_i$  the result of the i-th measurement, in  $\mu g/m^3$ ;

 $\overline{\mathbf{x}}$  the mean value of the 10 measurements;

n the number of measurements, n = 10.

The standard measurement uncertainty of the repeatability standard deviation u<sub>r</sub> is calculated as:

$$u_r = \frac{s_r}{\sqrt{m}} \tag{Eq. 6.3}$$

and

$$s_r = \frac{l_a}{c_t} * s_{r,c_t} \tag{Eq. 6.4}$$

Where:

(105	A
s <sub>r,ct</sub>	the repeatability standard deviation at $c_t$ , in $\mu g/m^3$ .
ct	the test gas concentration in the range of the annual limit value, in $\mu g/m^3$ ;
la	the annual limit value, in $\mu g/m^3$ ;
m	the number of measurements performed in order to obtain the value s <sub>r</sub> ;
s <sub>r</sub>	the repeatability standard deviation at the annual limit value, in $\mu g/m^3$ ;
	value, in µg/m <sup>3</sup> ;
u <sub>r</sub>	the standard measurement uncertainty of the repeatability standard deviation at the annual limit

#### 6.18.5 Assessment

Minimum requirement met? yes

#### 6.18.6 Documentation

To calculate the repeatability standard deviation, a test gas in the range of test value 1 (10% of the annual limit value) and then a test gas in the range of the annual limit value were offered with 11 repetitions. The last ten results were used for the calculation (see Tab. 6.3 and Tab. 6.4).

	GC 80001	GC 80004
Repeatability standard deviation $s_{r,z}$ , in $\mu g/m^3$	0.01	0.01
Benzene concentration, in µg/m <sup>3</sup>	0.53	0.53

Tab. 6.2: Repeatability standard deviation at the annual limit value

	GC 80001	GC 80004
Repeatability standard deviation $s_{r,ct}$ , in $\mu g/m^3$	0.02	0.02
Standard measurement uncertainty $u_r$ , in $\mu g/m^3$	0.00*	0.01*
Benzene concentration, in µg/m <sup>3</sup>	5.3	5.3

\*Values are used to calculate the expanded measurement uncertainty

# 6.19 Lack of fit of linearity of the calibration function

#### 6.19.1 Minimum requirement (DIN EN 14662-3: 2016-02 chapter 8.4.5)

The residual  $r_z$  at zero point shall be less than or equal to 0.5  $\mu$ g/m<sup>3</sup> and the largest relative residual  $r_{max}$  from the linear regression function shall be less than or equal to 5 %. If one or more relative residuals do not meet the criterion, the analyzer does not pass the linearity test.

#### 6.19.2 Equipment

Testing requires:

- Gas mixing system
- Test standards (e.g. zero gas and test gas of suitable concentration)

# 6.19.3 Testing

A linear regression function is determined based on the measurement results and the corresponding abandoned concentrations. The residual at each concentration level is equal to the difference between the mean value of the measured signal, expressed as a concentration, and the concentration given by the linear regression function. The linear regression function and the residuals are calculated in accordance with Annex A of DIN EN 14662-3: 2016-02.

The linearity of the analyzer shall be tested in the range between 0 % and 90 % of the maximum of certification range with at least six concentrations (including 0  $\mu$ g/m<sup>3</sup>).

#### 6.19.4 Evaluation

The residuals of the mean values for each calibration point (including the zero point) are calculated according to Annex A.2 of DIN EN 14662-3: 20016-02 using the following equations:

$$(y_a)_c = \frac{\sum (y_i)_c}{m}$$
 (Eq. 6.5)

Where:

 $(y_a)_c$  the mean y-value at concentration level c;

(y<sub>i</sub>)<sub>c</sub> the individual y-value at concentration level c;

m the number of repetitions at one and the same concentration level c.

The residual of each mean value (r<sub>c</sub>) at each concentration level is calculated according to:

$$r_c = (y_a)_c - (A + B \cdot c)$$
 (Eq. 6.6)

Where:

- A the intercept of the calibration function;
- B the slope of the calibration function.

The relative deviation (in %) of each mean value at the associated concentration c is calculated according to:

$$r_{c,rel.} = \frac{r_c}{c} * 100$$
 (Eq. 6.7)

The largest value of  $r_{c, rel.}$  is indicated as  $r_{max}$  and is used for the uncertainty calculation.

The standard measurement uncertainty  $u_l$  of the deviation from linearity at the annual limit value is calculated according to:

$$u_l = \frac{r_{max}}{100} * \frac{l_a}{\sqrt{3}}$$
(Eq. 6.8)

Where:

- $u_1$  the standard uncertainty  $u_1$  of the deviation from linearity at the annual limit value, in  $\mu g/m^3$ ;
- $r_{max}$  the maximum residual from a linear regression function, calculated according to Annex A, in %;
- $l_a \qquad \qquad \text{the annual limit value, in } \mu g/m^3.$

# 6.19.5 Assessment

Minimum requirement met? yes

#### 6.19.6 Documentation

Using a gas mixing system, five different test gas concentrations and zero gas were applied to the two gas chromatographs in the order: 50%, 10%, 30%, 5%, 90% and 0% of the maximum value of the certification range of benzene (=  $50 \ \mu g/m^3$ ).

Each concentration was applied for 60 minutes (4 measuring cycles of 15 minutes). The first measured value at each concentration level was discarded as a run-in value, i.e. only the last three measured values were used for the determination of the mean values. A regression calculation was used to calculate the calibration function (see Tab. 6.5).

Tab. 6.3: Calibration function of the gas chromatographs GC 80001 and GC 80004

	GC 80001	GC 80004
Calibration function	y = 1.0013 x - 0.4728	y = 0.9967 x - 0.2375

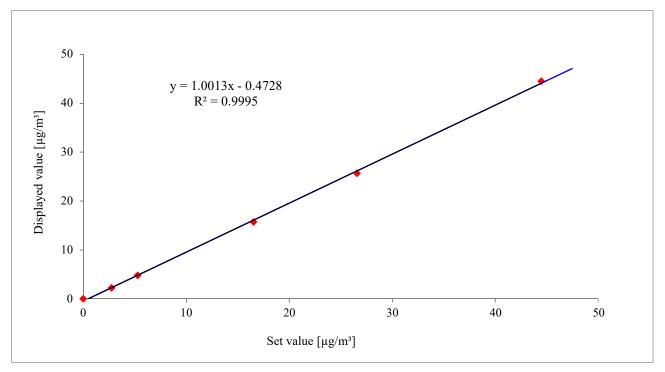


Fig. 6.3 and Fig. 6.4 show the calibration functions of the two gas chromatographs.

Fig. 6.1: Calibration function of the GC 80001

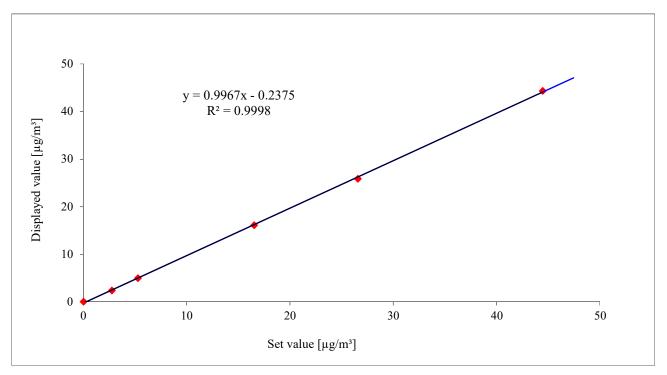


Fig. 6.2: Calibration function of the GC 80004

	c, in μg/m³	(ya)c, in µg/m <sup>3</sup>	(A + B *c)	rc, in μg/m³	rc,rel., in %
50 % of MR	26.57	25.67	26.1	-0.5	-1.7
10 % of MR	5.27	4.77	4.8	0	-0.7
30 % of MR	16.52	15.73	16.1	-0.3	-2.1
5 % of MR	2.75	2.22	2.3	-0.1	-2.1
90 % of MR	44.47	44.47	44.1	0.4	0.9
0 % of MR	0	0.02	-0.5	0.5	-

Tab. 6.4: Residuals of the mean values for each calibration point (including zero) for GC 80001

#### The results for GC 80001

Standard uncertainty $u_l$ , in $\mu g/m^3$	-0.06*
Residual at zero rz, in µg/m <sup>3</sup>	0.5
Largest residual r <sub>max</sub> , in %	-2.1

\*Values are used to calculate the expanded measurement uncertainty

<b>Fab. 6.5:</b> Residuals of the mean values for each calibration point (including zero) for GC 80004					
	c, in μg/m³	(y <sub>a</sub> ) <sub>c</sub> , in μg/m <sup>3</sup>	(A + B *c)	rc, in μg/m³	rc,rel., in %
50 % of MR	26.57	25.88	26.2	-0.4	-1.4
10 % of MR	5.27	5.01	5.0	0	-0.2
30 % of MR	16.52	16.14	16.2	-0.1	-0.6
5 % of MR	2.75	2.42	2.5	-0.1	-3.0
90 % of MR	44.47	44.35	44.1	0.3	0.6

-0.2

0.3

\_

0.05

# The results for GC 80004

0% of MR

Standard uncertainty u <sub>1</sub> , in µg/m <sup>3</sup>	-0.09*
Residual at zero $r_z$ , in $\mu g/m^3$	0.3
Largest residual r <sub>max</sub> , in %	-3.0

0

\*Values are used to calculate the expanded measurement uncertainty

# 6.20 Sensitivity coefficient of sample gas pressure

# 6.20.1 Minimum requirement (DIN EN 14662-3: 2016-02 chapter 8.4.6)

The sensitivity coefficient  $b_{gp}$  for the influence of the sample gas pressure at test value 2 (70 – 80% of the maximum of the certification range) shall be less than or equal to 0.10 ( $\mu g/m^3$ )/kPa.

# 6.20.2 Equipment

Testing requires:

- Gas mixing system
- Test standards (e.g. test gas of suitable concentration)
- Pressure measuring system

# 6.20.3 Testing

Five measurements shall be performed with concentrations at test value 2 and at sample gas pressures of 80 kPa ( $\pm$  0.2 kPa) and 110 kPa ( $\pm$  0.2 kPa). For each pressure, the mean value from the last four measurements shall be calculated.

# 6.20.4 Evaluation

The influence of the sample gas pressure  $b_{gp}$  is calculated according to the following equation:

$$b_{gp} = \left| \frac{(x_{p2} - x_{p1})}{(p_1 - p_2)} \right|$$
(Eq. 6.9)

The standard uncertainty  $u_{gp}$  depending on the change of sample gas pressure at the annual limit value is calculated as follows:

$$u_{gp} = \frac{l_a}{c_t} * b_{gp} * \sqrt{\frac{(p_2 - p_1)^2}{3}}$$
(Eq. 6.10)

Where:

$\mathbf{b}_{\mathrm{gp}}$	the sensitivity coefficient to sample gas pressure, in $(\mu g/m^3)/kPa$ ;
X <sub>p1</sub>	the mean value of the measurements at sample gas pressure $p_1$ , in $\mu g/m^3$ ;
X <sub>p2</sub>	the mean value of the measurements at sample gas pressure $p_2$ , in $\mu g/m^3$ ;
$\mathbf{p}_1$	the lowest sample gas pressure, in kPa;
<b>p</b> <sub>2</sub>	the highest sample gas pressure, in kPa;
u <sub>gp</sub>	the standard uncertainty depending on the change of sample gas pressure at the annual limit value, in $\mu$ g/m <sup>3</sup> ;
la	the annual limit value, in µg/m <sup>3</sup> ;
Ct	the test gas concentration in the range of test value 2 (70-80% of the maximum of the certification range), in $\mu g/m^3$ .

# 6.20.5 Assessment

Minimum requirement met? yes

# 6.20.6 Documentation

The two gas chromatographs were supplied via a T-piece with a test gas in excess. To set a sample gas overpressure, the excess line was constricted with a needle valve. The correct pressure was monitored with a Vacuubrand DVR 2 pressure measuring system. To set a sample gas vacuum, the needle valve was installed on the sample gas side of the T-piece. An additional small pump was used to generate vacuum in the sampling line. The correct pressure was monitored with a Vacuubrand DVR 2 pressure measuring system.

For the calculation of the sensitivity coefficient  $b_{gp}$  of the sample gas pressure and the associated standard measurement uncertainty  $u_{gp}$ , five successive measurements with concentrations at test value 2 were carried out at a pressure of 85 kPa and at a pressure of 110 kPa. For each pressure, the mean values of the last four measured values were used for the evaluation (see Tab. 6.8).

	GC 80001	GC 80004
Pressure p1, in kPa	80.0	80.0
Pressure p <sub>2</sub> , in kPa	110.0	110.0
Concentration $x_{p1}$ , in $\mu g/m^3$	34.4	34.8
Concentration $x_{p2}$ , in $\mu g/m^3$	36.5	36.5

**Tab. 6.6:** Measured data of  $p_1$ ,  $p_2$ ,  $x_{p1}$  and  $x_{p2}$ 

The following values of sensitivity coefficient  $b_{gp}$  and the associated standard uncertainty  $u_{gp}$  were calculated (see Tab. 6.9).

Tab. 6.7: Calculate values of bgp, ugp

	GC 80001	GC 80004
Sensitivity coefficient $b_{gp}$ , in ( $\mu g/m^3$ )/kPa	0.07	0.06
Standard uncertainty $u_{gp}$ , in $\mu g/m^3$	0.16*	0.13*

\*Values are used to calculate the expanded measurement uncertainty

# 6.21 Sensitivity coefficient of surrounding temperature

# 6.21.1 Minimum requirement (DIN EN 14662-3: 2016-02 chapter 8.4.7)

The sensitivity coefficient  $b_{st}$  for the influence of surrounding temperature at test value 2 (70 – 80% of the maximum of the certification range) shall be less than or equal to 0.08 ( $\mu g/m^3$ )/K.

# 6.21.2 Equipment

Testing requires:

- Test standards (e.g. test gas of suitable concentration)
- Climate chamber

# 6.21.3 Testing

The influence of the surrounding temperature on the measured value is determined by measurements at a minimum temperature of 0°C and a maximum temperature of 30°C (or within manufacturer's specifications). Five measurements with concentrations at test value 2 shall be performed at each temperature. The mean value of the last four measurements shall be calculated.

#### 6.21.4 Evaluation

The influence of the surrounding temperature b<sub>st</sub> is calculated according to the following equation:

$$b_{st} = \left| \frac{(x_2 - x_1)}{(T_{s,2} - T_{s,1})} \right|$$
(Eq. 6.11)

The standard uncertainty  $u_{st}$  depending on the change of surrounding temperature at the annual limit value is calculated as follows:

$$u_{st} = \frac{l_a}{c_t} * b_{st} * \sqrt{\frac{(T_{S,2} - T_{S,1})^2}{3}}$$
(Eq. 6.12)

Where:

b <sub>st</sub>	the sensitivity coefficient for the influence of surrounding temperature, in $(\mu g/m^3)/K$ ;
<b>X</b> 1	the mean value of the measurements at $T_{s,1}$ , in $\mu g/m^3$ ;
<b>X</b> <sub>2</sub>	the mean value of the measurements at $T_{s,2}$ , in $\mu g/m^3$ ;
$T_{S,1}$	the lowest surrounding temperature, in K;
$T_{S,2}$	the highest surrounding temperature, in K;
u <sub>st</sub>	the standard uncertainty depending on the change of surrounding temperature at the annual
	limit value, in µg/m <sup>3</sup> ;
la	the annual limit value, in $\mu g/m^3$ ;
c <sub>t</sub>	the test gas concentration in the range of test value 2, in $\mu g/m^3$ .

#### 6.21.5 Assessment

The sensitivity coefficient  $b_{st}$  of the influence of surrounding temperature does not exceed the minimum requirement.

Minimum requirement met? yes

#### 6.21.6 Documentation

To determine the influence of the ambient temperature on the measured value, the devices were placed in a climate chamber. The sensitivity coefficient was determined at a minimum temperature of 0°C and a maximum temperature of 30°C. Humidity was set to near 50 %. The following temperature program was set:

 $20^{\circ}\mathrm{C} \rightarrow 0^{\circ}\mathrm{C} \rightarrow 30^{\circ}\mathrm{C} \rightarrow 20^{\circ}\mathrm{C}$ 

At each temperature setting, the two gas chromatographs were supplied with test gas concentration in the range of test value 2. After an equilibration time of the climatic chamber (approx. 2 h), the last four measurement cycles at each temperature level were used for evaluation (see Tab. 6.10).

Tab. 6.8: Results of the ambient temperature dependence

	GC 80001	GC 80004
Benzene concentration C, in µg/m <sup>3</sup>	38.7	38.7
Lowest surrounding temperature T <sub>S,1</sub> , in K	273.0	273.0
Highest surrounding temperature T <sub>S,2</sub> , in K	303.0	303.0
Sensitivity coefficient $b_{st}$ , in (µg/m <sup>3</sup> )/K	0.03	0.06
Standard uncertainty $u_{st}$ , in $\mu g/m^3$	0.06*	0.12*

\*Values are used to calculate the expanded measurement uncertainty

# 6.22 Sensitivity coefficient of electrical voltage

#### 6.22.1 Minimum requirement (DIN EN 14662-3: 2016-02 chapter 8.4.8)

The sensitivity coefficient  $b_V$  for the influence of the electrical voltage at the test value 2 (70 – 80% of the maximum of the certification range) shall be less than or equal to 0.08 ( $\mu$ g/m<sup>3</sup>)/V.

#### 6.22.2 Equipment

Testing requires:

- Test standards (e.g. test gas of suitable concentration)
- Transformer with a controlled range for mains voltages from 210 V to 245 V

#### 6.22.3 Testing

The voltage dependence shall be tested at the minimum and maximum voltage range specified by the manufacturer. Five measurements with concentration at the test value 2 shall be performed at each voltage. The mean value of the last four measurements shall be calculated.

# 6.22.4 Evaluation

The influence of the electric voltage  $b_V$  is calculated according to the following equation:

$$b_V = \left| \frac{(x_{V,2} - x_{V,1})}{(V_2 - V_1)} \right|$$
(Eq. 6.13)

The standard uncertainty  $u_V$  depending on the change of electric voltage at the annual limit value is calculated as follows:

$$u_V = \frac{l_a}{c_t} * b_V * \sqrt{\frac{(V_2 - V_1)^2}{3}}$$
(Eq. 6.14)

Where:

$b_{\rm V}$	the sensitivity coefficient for the influence of electric voltage, in ( $\mu g/m^3$ )/V;
<b>X</b> <sub>V,1</sub>	the mean value of the measurements at $V_1$ , in $\mu g/m^3$ ;
<b>X</b> V,2	the mean value of the measurements at $V_2$ , in $\mu g/m^3$ ;
$V_1$	the lowest electric voltage, in V;
$V_2$	the highest electric voltage, in V;

- $u_V$  the standard uncertainty depending on the change of electric voltage at the annual limit value, in  $\mu g/m^3$ ;
- $l_a$  the annual limit value, in  $\mu g/m^3$ ;
- $c_t$  the test gas concentration in the range of test-value 2, in  $\mu g/m^3$ .

# 6.22.5 Assessment

The sensitivity coefficient  $b_V$  of the influence of electric voltage does not exceed the minimum requirement.

Minimum requirement met? yes

#### 6.22.6 Documentation

To test the influence of electric voltage, the mains voltage was varied using a programmable AC voltage source (AC voltage source ACS-4600-PS). The following settings were made:

 $230 \text{ V} \rightarrow 210 \text{ V} \rightarrow 245 \text{ V} \rightarrow 230 \text{ V}$ 

At each voltage setting, the two gas chromatographs were supplied with test gas concentration in the range of test value 2. The last four measurement cycles at each voltage level were used for evaluation (see Tab.6.11).

	GC 80001	GC 80004
Benzene concentration C, in $\mu g/m^3$	36.2	36.2
Lowest electric voltage V <sub>1</sub> , in V	210	210
Highest electric voltage V <sub>2</sub> , in V	245	245
Sensitivity coefficient $b_V$ , in ( $\mu g/m^3$ )/V	0.001	0.002
Standard uncertainty $u_V$ , in $\mu g/m^3$	0.002*	0.006*

\*Values are used to calculate the expanded measurement uncertainty

# 6.23 Interferents

The instrument response to certain interferents, which are to be expected to be present in ambient air, shall be tested. The cross-sensitivity to water vapor and to organic compounds shall be determined at test gas concentration in the range of the annual limit value (=  $5.0 \ \mu g/m^3$ ).

### 6.23.1 Water vapor

#### 6.23.1.1 Minimum requirement (DIN EN 14662-3: 2016-02 chapter 8.4.9.2)

The influence of water vapor  $b_{\rm H2O}$  shall be tested in the range of the annual limit value and shall be less than or equal to 0.015 ( $\mu g/m^3$ )/(mmol/mol).

#### 6.23.1.2 Testing

The influence of water vapor is tested at a  $H_2O$ -concentration of 19 mmol/mol. The test shall be performed at a benzene concentration  $c_t$ , which is similar to the annual limit value.

#### 6.23.1.3 Evaluation

The influence of water vapor  $b_{H2O}$  at a benzene concentration  $c_t$  is calculated according to the following equation:

$$b_{H_2O} = \frac{(X_{+W} - X_{-W})}{c_W}$$
(Eq. 6.15)

The standard measurement uncertainty  $u_{H2O}$  due to the interference by water vapor at the annual limit value is calculated as follows:

$$u_{H_2O} = \frac{l_a}{c_t} * b_{H_2O} * \sqrt{\frac{(c_{H_2O,max}^2 + c_{H_2O,max} * c_{H_2O,min} + c_{H_2O,min}^2)}{3}}$$
(Eq. 6.16)

Where:

b <sub>H2O</sub>	the sensitivity coefficient to water vapor, in (µg/m <sup>3</sup> )/(mmol/mol);
$\mathbf{X}$ +W	the mean value of the measurements at $c_t$ with water vapor, in $\mu g/m^3$ ;
X-W	the mean value of the measurements at $c_t$ without water vapor, in $\mu g/m^3$ ;
$c_{W}$	the concentration of water vapor in the test gas, in mmol/mol;
u <sub>H2O</sub>	the standard measurement uncertainty due to the disturbance caused by water vapor, in $\mu g/m^3$ ;
la	the annual limit value, in µg/m <sup>3</sup> ;
c <sub>t</sub>	the test gas concentration in the range of the annual limit value, in $\mu g/m^3$ ;
C <sub>H2O,max</sub>	the highest concentration of water vapor (= 21 mmol/mol), in mmol/mol;
C <sub>H2O,min</sub>	the lowest concentration of water vapor (= 6 mmol/mol), in mmol/mol.

#### 6.23.1.4 Assessment

Minimum requirement met? yes

#### 6.23.1.5 Documentation

To determine the cross-sensitivity, test gas in the range of the annual limit value (=  $5.0 \ \mu g/m^3$ ) with interfering component was offered to the measuring devices. For this purpose, the dilution air was passed over a Nafion membrane, which was moistened with distilled water in countercurrent. A defined moisture content was set via the temperature of the water bath. The same measurement was carried out in the absence of the interfering component. To calculate the deviation between the measured value in the "dry" and "wet" test gas, the measured value of the "wet" test gas was corrected using the volume fraction of water vapor in the dilution air (see Tab. 6.12).

	GC 80001	GC 80004
Concentration of water vapor in the test gas cw, in mmol/mol	19.0	19.0
Minimum water vapor concentration c <sub>H2O,min</sub> , in mmol/mol	6	6
Maximum water vapor concentration c <sub>H2O,max</sub> , in mmol/mol	21	21
Benzene concentration $c_t$ , in $\mu g/m^3$	5.3	5.3
Sensitivity coefficient $b_{H2O}$ , in (µg/m <sup>3</sup> )/(mmol/mol)	0.012	-0.006
Standard uncertainty $u_{H2O}$ , in $\mu g/m^3$	0.163*	-0.081*

\*Values are used to calculate the expanded measurement uncertainty

#### 6.23.2 Organic compounds

#### 6.23.2.1 Minimum requirement (DIN EN 14662-3: 2016-02 chapter 8.4.9.3)

The contribution of the mixture of organic compounds  $\Delta x_{OC}$  to the measurement signal of benzene at a concentration in the range of the annual limit value (= 5.0 µg/m<sup>3</sup>) shall be less than or equal to 0.25 µg/m<sup>3</sup>.

#### 6.23.2.2 Testing

A possible cross-sensitivity due to organic compounds is tested by adding a gas mixture of different organic compounds with concentrations of about 5  $\mu$ g/m<sup>3</sup> to the test gas with a benzene concentration in the range of the annual limit value (= 5.0  $\mu$ g/m<sup>3</sup>).

#### 6.23.2.3 Evaluation

The interference test with the organic compounds is a test to ensure that the contributions of organic compounds that may co-elute with benzene to the signal of benzene are insignificant. The results of the test shall meet the minimum requirement.

$$\Delta x_{OC} = x_{+} - x_{-} \tag{Eq. 6.17}$$

Where:

$\Delta x_{\rm OC}$	the contribution of the mixture of organic compounds to the signal of benzene, in $\mu g/m^3$ ;
X+	the mean value of the measured signal for benzene at the concentration ct (at the annual limit
	value) in the presence of interfering components, in $\mu g/m^3$ ;
Х-	the mean value of the measured signal for benzene at the concentration ct (at the annual limit
	value) in the absence of interfering components, in $\mu g/m^3$ .

#### 6.23.2.4 Assessment

Minimum requirement met? yes

#### 6.23.2.5 Documentation

To determine the cross-sensitivity, test gas in the range of the annual limit value (=  $5.0 \ \mu g/m^3$ ) with 11 organic interfering components was offered to the measuring devices. The organic components were added to the benzene test gas using a thermal mass flow controller (MFC). Since the test gas volume flow was considerably higher than the volume flow of the added interfering components (about a factor of 3300), a correction of the

initial benzene concentration was not necessary. The same measurement was then carried out in the absence of the interfering components.

The following 11 interfering organic components were used: methylcyclopentane (5.1  $\mu$ g/m<sup>3</sup>); 2,2,3-trimethylbutane (5.0  $\mu$ g/m<sup>3</sup>); 2,4-dimethylpentane (4.9  $\mu$ g/m<sup>3</sup>); 2,2,4-trimethylpentane (5.1  $\mu$ g/m<sup>3</sup>); cyclohexane (5.3  $\mu$ g/m<sup>3</sup>); 2,3-dimethylpentane (5.0  $\mu$ g/m<sup>3</sup>); 2-methylhexane (4.9  $\mu$ g/m<sup>3</sup>); 3-ethylpentane (5.0  $\mu$ g/m<sup>3</sup>); trichloroethene (5.3  $\mu$ g/m<sup>3</sup>); n-heptane (5.0  $\mu$ g/m<sup>3</sup>); 1-butanol (5.2  $\mu$ g/m<sup>3</sup>).

The results of the tests on the cross-sensitivity to organic compounds are shown in Tab. 6.13.

Tab. 6.11: The	e cross-sensitivity to	organic co	mpounds
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	GC 80001	GC 80004
Contribution interference component $\Delta x_{OC}$ , in $\mu g/m^3$	-0.09	-0.03

# 6.24 Memory-effect

# 6.24.1 Minimum requirement (DIN EN 14662-3: 2016-02 chapter 8.4.10)

The benzene concentration of the first zero gas analysis following the analysis at test value 3 (90 % of the maximum value of the certification range of benzene) shall be less than or equal to  $1.0 \ \mu g/m^3$ .

## 6.24.2 Equipment

Testing requires:

• Test standards (e.g. zero gas and test gas of suitable concentration)

## 6.24.3 Testing

The influence of a memory effect caused by retention of benzene in the measuring system due to unsuitable materials or a large dead volume shall be tested. Two measurements shall be performed at test value 3, after which the gas supply is switched to zero air. The result of the first measurement of zero air is used to calculate the concentration of benzene  $c_m$  resulting from memory effect. The test sequence shall be repeated three times.

## 6.24.4 Evaluation

The first zero gas analysis immediately following the high benzene concentration analysis (90% of the maximum of certification range) shall not exceed a measured benzene concentration of  $1.0 \ \mu g/m^3$ .

The standard uncertainty  $u_m$  due to memory effect is calculated according to:

$$u_m = \frac{l_a}{c_r} * c_m * \sqrt{3}$$
 (Eq. 6.18)

Where:

u <sub>m</sub>	the standard uncertainty due to memory effect, in $\mu g/m^3$ ;
c <sub>m</sub>	the influence of memory effect, in $\mu g/m^3$ ;
Ct	the test concentration of benzene (around 90 % of the maximum of the certification range), in $\mu g/m^3$ ;
$l_a$	the annual limit value, in $\mu g/m^3$ .

## 6.24.5 Assessment

Minimum requirement met? yes

#### 6.24.6 Documentation

To determine the memory effect, two measurements were carried out at a concentration corresponding to approximately 90% of the maximum value of the certification range. Then the gas supply was switched to zero air. This procedure was repeated three times. The mean value from each of the first three zero gas values was used to determine the memory effect.

Table 6.14 shows the result of the determination of the memory effect.

Tab. 6.12: Results of the memory effect test

	GC 80001	GC 80004
Influence of memory effect $c_m$ , in $\mu g/m^3$	0.1	0.3
Standard uncertainty $u_m$ , in $\mu g/m^3$	0.01*	0.02*

\*Values are used to calculate the expanded measurement uncertainty

# 6.25 Long-term drift

## 6.25.1 Minimum requirement (DIN EN 14662-3: 2016-02 chapter 8.5.4)

In the field test, over a drift period of 14 days, the largest value of long-term drift at zero point  $D_{l,z}$  shall be less than or equal to 0.5  $\mu$ g/m<sup>3</sup> and the largest value of long-term drift at test value 2 (70 - 80 % of the maximum value of the certification range)  $D_{l,s}$  shall be less than or equal to 10 %.

## 6.25.2 Equipment

Testing requires:

• Test standards (e.g. test gas of suitable concentration)

## 6.25.3 Testing

During the first 14 days of the field test, measurements at zero point and at test value 2 shall be performed at least every two days. During the rest of the field test, these measurements shall be performed at least every two weeks. Five successive measurements shall be carried out. The long-term drift is then determined from the mean values of the last four measurements. During the field test, no adjustments of the zero point and the reference point shall be carried out.

## 6.25.4 Evaluation

The long-term drift at the zero point  $D_{l,z}$  is calculated using the following equation:

$$D_{l,z} = (x_{z,1} - x_{z,0}) \tag{Eq. 6.19}$$

The long-term drift at test value 2  $D_{l,s}$  is calculated using the following equation:

$$D_{l,s} = \frac{(x_{s,1} - x_{s,0}) - D_{l,z}}{x_{s,1}} * 100$$
 (Eq. 6.20)

The standard uncertainty  $u_{d,l,la}$  due to the long-term drift at level of the annual limit value is calculated according to:

$$u_{d,l,l_a} = \frac{D_{l,s}}{100} * \frac{l_a}{\sqrt{3}}$$
(Eq. 6.21)

#### Where:

D <sub>l,z</sub>	the long term drift at zero point, in $\mu g/m^3$ ;
X <sub>z,0</sub>	the mean value of the measurements at the zero point at the beginning of the drift period, in $\mu g/m^3$ ;
X <sub>z,1</sub>	the mean value of the measurements at the zero point at the end of the drift period, in $\mu g/m^3$ ;
D <sub>l,s</sub>	the long-term drift at test-value 2, in %;
X <sub>s,0</sub>	the mean value of the measurements at test value 2 at the beginning of the drift period, in $\mu g/m^3$ ;
X <sub>s,1</sub>	the mean value of the measurements at test value 2 at the end of the drift period, in $\mu g/m^3$ ;
$\mathbf{u}_{\mathrm{d,l,la}}$	the standard measurement uncertainty due to the long-term drift at the annual limit value, in $\mu g/m^3$ ;
la	the annual limit value, in μg/m <sup>3</sup> .

#### 6.25.5 Assessment

Minimum requirement met? yes

#### 6.25.6 Documentation

During the first two weeks of the field test, five measurements cycles of zero air and test gas (benzene concentration of approx.  $35.1 \ \mu g/m^3$ ) were performed every two or three days. During the rest of the field test these measurements were perform every one or two weeks. For evaluation of the long-term drift four measurement cycles were used in each case. The first measurement cycle was discarded.

The results of the long-term drift test are shown in Tab. 6.15 and Tab. 6.16.

#### Tab. 6.13: Long-term drift $D_{l, z}$ at zero point

	GC 80001	GC 80004
	D <sub>1,z</sub> , in µg/m <sup>3</sup>	D <sub>1,z</sub> , in µg/m <sup>3</sup>
Mean value	-0.01	-0.01
Minimum value	0.00	0.00
Maximum value	-0.01	-0.02

**Tab. 6.14:** Long-term drift  $D_{l,s}$  at test value 2 (set point: 35.1  $\mu$ g/m<sup>3</sup>)

	GC 8	30001	GC 80004				
	D <sub>1,s</sub> , in %	u <sub>d,l,la</sub> , in µg/m <sup>3</sup>	D <sub>1,s</sub> , in %	u <sub>d,l,la</sub> , in µg/m <sup>3</sup>			
Mean value	-1.78	-	-0.28	-			
Minimum value	-0.15	-	0.10	-			
Maximum value (after 3 month)	-7.76	-0.22*	-4.64	-0.13*			

\*Values are used to calculate the total measurement uncertainty

The specified drift limit was not exceeded by both gas chromatographs in the field test (see Fig. 6.5 and Fig. 6.6). Therefore, three months are set as the maintenance interval.

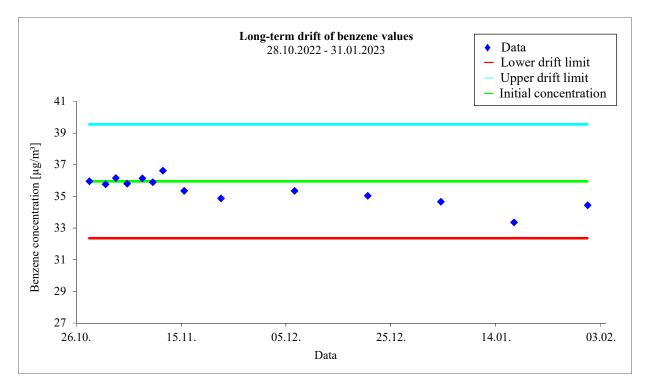


Fig. 6.3: Long term drift  $D_{l,s}$  at test value 2 (set point: 35.1  $\mu$ g/m<sup>3</sup>) - GC 80001

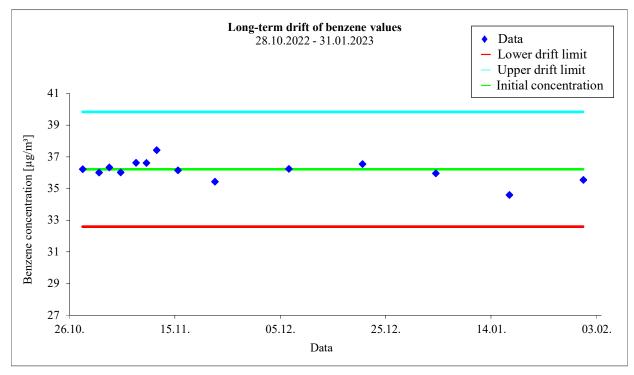


Fig. 6.4: Long term drift  $D_{l,s}$  at test value 2 (set point: 35.1  $\mu$ g/m<sup>3</sup>) - GC 80004

# 6.26 Reproducibility standard deviation under field conditions

# 6.26.1 Minimum requirement (DIN EN 14662-3: 2016-02 chapter 8.5.5)

The reproducibility standard deviation under field conditions  $s_{r,f}$  of the two devices shall be less than or equal to 0.25  $\mu$ g/m<sup>3</sup>.

# 6.26.2 Equipment

Testing requires:

• Test standards (e.g. test gas of suitable concentration)

# 6.26.3 Testing

The standard deviation under field conditions is determined during the three-month field test. The simultaneous, continuous measurements with two identical measuring devices shall be performed at the same site as a double determination. For taking the air samples, a common sampling system may be used. The sampling system shall allow both measuring devices to make a representative measurement of the same ambient air sample. This reduces systematic errors caused by spatial and/or temporal inhomogeneities of the mass concentration distribution of the measured objects in the ambient air. The output signals of the measuring systems shall be recorded with electronic data storage as single measured values.

## 6.26.4 Evaluation

The reproducibility standard deviation under field conditions is calculated from the measured values of the three-month period.

The difference  $\Delta x_{f,i}$  for each (i-th) parallel measurement is calculated according to:

$$\Delta x_{f,i} = x_{f,1,i} - x_{f,2,i} \tag{Eq. 6.22}$$

The reproducibility standard deviation under field conditions  $s_{r,f}$  is calculated as follows:

$$s_{r,f} = \sqrt{\frac{\sum_{i=1}^{n} \Delta x_{f,i}^2}{2n}}$$
 (Eq. 6.23)

The standard uncertainty  $u_{r, f}$  due to the reproducibility under field conditions at the annual limit value is calculated according to:

$$u_{r,f} = s_{r,f} \tag{Eq. 6.24}$$

Where:

$\Delta x_{\mathrm{f},i}$	the i-th difference in a parallel measurement, in $\mu g/m^3$ ;
$\mathbf{X}_{\mathrm{f},1,\mathrm{i}}$	the i-th measurement result of device 1, in $\mu g/m^3$ ;
X <sub>f,2,i</sub>	the i-th measurement result of device 2, in $\mu g/m^3$ ;
$\mathbf{S}_{r,f}$	the reproducibility standard deviation under field conditions, in $\mu g/m^3$ ;
n	the number of parallel measurements.

## 6.26.5 Assessment

Minimum requirement met? yes

## 6.26.6 Documentation

All values of the ambient air measurements during the field test were used for evaluation. The data for functional check with test gas and zero gas were not included.

The determined data used to calculate the standard deviation are summarized in Tab. 6.17.

	GC 80001	GC 80004	
Number of parallel measurements n	87	82	
Mean difference $\Delta x_f$ , in $\mu g/m^3$	-0.13		
Mean benzene concentration, in µg/m <sup>3</sup>	0.98	1.11	
Maximum benzene concentration, in $\mu g/m^3$	17.53	19.53	
$s_{r,f} = u_{r,f}$ , in $\mu g/m^3$	0.1	7*	

Tab. 6.15: Data used to calculate the reproducibility standard deviation under field conditions

\*Values are used to calculate the expanded measurement uncertainty

Fig. 6.7 shows the data obtained from the two gas chromatographs during the field test.

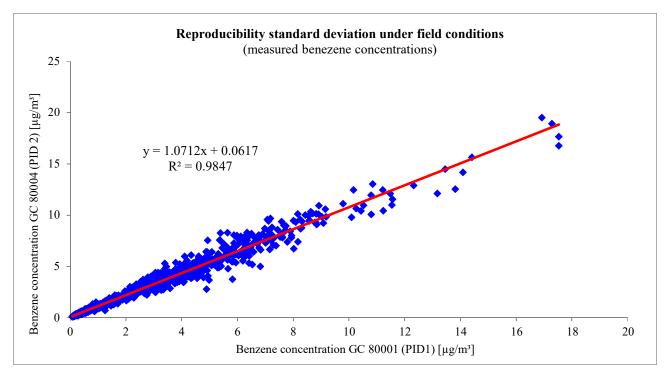


Fig. 6.5: Benzene concentrations measured during the field test

## 6.27 Period of unattended operation

#### 6.27.1 Minimum requirement (DIN EN 14662-3: 2016-02 chapter 8.5.6)

The control interval is either the period of time in which the drift is within the performance criterion for longterm drift or the manufacturer's specified maintenance interval, whichever is shorter.

#### 6.27.2 Equipment

Testing requires:

• Test standards (e.g. zero gas and test gas of suitable concentration)

#### 6.27.3 Testing

During the field test, it shall be determined which maintenance work is required at which intervals to ensure the proper functioning of the measuring system As far as no maintenance work at shorter intervals is necessary in terms of equipment, the maintenance interval essentially results from the drift behavior of the measuring device (see Chap. 6.25).

If one of the measuring instruments malfunctions during the field test, these shall be determined and corrected. The field test shall be restarted if the malfunction has an effect on the determination of the maintenance interval.

# 6.27.4 Evaluation

The respective measured values are compared with the start values. The deviations are determined for the zero point and the reference point. The end of the maintenance interval is reached when the deviations exceed the permissible drift.

A shorter maintenance interval than the one determined by the drift analysis is given if other maintenance work is required at shorter intervals.

## 6.27.5 Assessment

No maintenance work was required on the measuring devices during the three-month field test. The carrier gas consumption (nitrogen 5.0) is approx. 15 ml/min per device. This results for a 50-liter compressed gas cylinder in a service life of more than one year.

The maintenance interval is determined only by the drift behavior of the measuring devices. (see Chap. 6.25). During the measurement period of 95 days, the specified drift limit was not exceed by both gas chromatographs. For this reason, three months are set as the maintenance interval.

Minimum requirement met? yes

# 6.27.6 Documentation

See Chap. 6.25.

# 6.28 Availability

# 6.28.1 Minimum requirement (DIN EN 14662-3: 2016-02 chapter 8.5.7)

The availability of the measuring system shall be determined during the field test and shall be at least 90 %.

## 6.28.2 Equipment

No additional equipment is required.

## 6.28.3 Testing

The availability of the measuring system shall be determined in the field test. The start and end time of the field test shall be documented. Furthermore, all interruptions of the test, e.g. due to malfunctions or maintenance work, shall be documented with the respective start and end times.

## 6.28.4 Evaluation

From the difference between the end time and the start time, the operating time shall be calculated as the reference time of availability. Furthermore, the times in which the device did not provide any usable measured values shall be determined.

The total time period with useable measuring data is the period during which valid measuring data of the ambient air concentrations are obtained. The time needed for calibration, conditioning of sample lines and filters and maintenance shall not be considered.

The availability of the device shall be calculated according to the following equation:

$$A = \frac{t_{\rm u}}{t_{\rm t}} \cdot 100 \ \% \tag{Eq. 6.25}$$

#### Where:

*A* the availability of the device;

- $t_{\rm u}$  the total time period with valid measured values, in h;
- $t_t$  the total time period of the field test minus time for calibration, conditioning and maintenance, in h.

#### 6.28.5 Assessment

Minimum requirement met? yes

#### 6.28.6 Documentation

The field test was carried out in the period from 28.10.2022 to 31.01.2023 at the LUBW test site. During this time, a function check was carried out by externally connecting zero gas and test gas at intervals of two or three days during the first two weeks. After the first two weeks, the function check was carried out every one or two weeks.

The results for calculating availability are summarized in Table 6.18.

		GC 80001	GC 80004		
Start of the field test	f the field test		28.10.2022; 10:30 AM		
End of the field test		31.01.2023;	03:00 PM		
Total time $t_E^*$	h:min	2238:00	2238:00		
Calibration time $t_K$	h:min	42:30	42:30		
Failure time t <sub>A</sub>	h:min	0:00	0:00		
Maintenance time t <sub>w</sub>	h:min	0:00	0:00		
Availability A.	%	100	100		

Tab. 6.16: Data needed for the determination of the availability of GC 80001 and GC 80004

With:  $t_u = t_E - t_A - t_K - t_W$  and  $t_t = t_E - t_K - t_W$ 

\*During the field test, various maintenance work had to be carried out at the station and on the sampling system. Furthermore, there were problems with the dosing of benzene. Since this led to an impairment of the measurements, the corresponding measured values were not taken into account in the evaluations. This resulted in a failure of 46 h30°min. The field test was accordingly extended by three days.

# 6.29 Expanded Uncertainty

# 6.29.1 Minimum requirement (DIN EN 14662-3: 2016-02 chapter 8.6 und annex E)

The expanded measurement uncertainty of the device shall be determined. The determined value shall not exceed the requirements of the EU daughter directives for air quality (data quality requirement: for benzene, a maximum value of 25% for the relative expanded uncertainty is allowed for continuous measurements).

## 6.29.2 Equipment

No additional equipment is required.

#### 6.29.3 Testing

The expanded uncertainty of the measured values shall be compile together for the determined performance characteristics during the performance test of the instruments.

#### 6.29.4 Evaluation

The type approval of the analyzer consists of the following steps:

- a) The value of each individual performance characteristic tested in the laboratory shall fulfil the criterion stated in Table 1 of the DIN EN 14662-3: 2016-02.
- b) The expanded measurement uncertainty calculated from the standard uncertainties based on the values of the specific performance characteristics determined in the laboratory tests shall fulfil the criterion of 25 % (for fixed measurements) given in Annex I to Directive 2008/50/EC. This criterion is the maximum permissible uncertainty of individual measurements for continuous measurements at the limit value.
- c) The value of each of the individual performance characteristics tested in the field shall fulfil the criterion stated in Table 1 of the DIN EN 14662-3: 2016-02.
- d) The expanded measurement uncertainty calculated from the standard uncertainties based on the values of the specific performance characteristics determined in the laboratory and field tests shall fulfil the criterion of 25 % (for fixed measurements) given in Annex I to Directive 2008/50/EC. This criterion is the maximum permissible uncertainty of individual measurements for continuous measurements at the limit value.

The instrument can be type approved when all four requirements are met.

The combined standard uncertainty uc shall be calculated according to:

$$u_c = \sqrt{\sum u_i^2} \tag{Eq. 6.26}$$

The absolute expanded uncertainty U shall be calculated according to:

$$U = k * u_c \tag{Eq. 6.27}$$

The relative expanded uncertainty W shall be calculated according to:

$$W = \frac{U}{l_a} * 100\%$$
 (Eq. 6.28)

Where:

- $u_c$  the combined standard uncertainty, in  $\mu g/m^3$ ;
- $u_i$  the partial contribution to the combined standard uncertainty, in  $\mu g/m^3$ ;
- U the absolute expanded uncertainty, in  $\mu g/m^3$ ;
- k the expansion factor (here k=2) with a confidence level of 95 %;
- W the relative expanded uncertainty, in %;
- $l_a$  the annual limit value, in  $\mu g/m^3$ .

The uncertainties listed below are to be included in the calculation of the expanded measurement uncertainty after the laboratory tests (Tab. 6.19) and after the laboratory and field tests (Tab. 6.20), respectively.

 Tab. 6.17: Standard uncertainties included in the calculation of the expanded measurement uncertainty after the laboratory tests

Standard uncertainty due to	Symbol
Repeatability standard deviation at annual limit value	<i>Ur</i>
Deviation of linearity at annual limit value	ul
Change of the sample gas pressure at annual limit value	u <sub>gp</sub>
Change of the surrounding temperature at annual limit value	u <sub>st</sub>
Change of the electrical voltage at annual limit value	$u_V$
Interfering component H <sub>2</sub> O (at 19 mmol/mol) at annual limit value	u <sub>H2O</sub>
Memory effect	u <sub>m</sub>
Difference sample/calibration port	<i>u</i> <sub>Asc</sub>
Uncertainty of the test gas	ucg

 Tab. 6.18: Standard uncertainties included in the calculation of the expanded measurement uncertainty after the laboratory and field tests

Standard uncertainty due to	Symbol
Repeatability standard deviation at annual limit value <sup>a</sup>	<i>u</i> <sub>r</sub>
Deviation of linearity at annual limit value	ul
Change of the sample gas pressure at annual limit value	u <sub>gp</sub>
Change of the surrounding temperature at annual limit value	u <sub>st</sub>
Change of the electrical voltage at annual limit value	uv
Interfering component H <sub>2</sub> O (at 19 mmol/mol) at annual limit value	u <sub>H2O</sub>
Memory effect	u <sub>m</sub>
Reproducibility standard deviation under field conditions <sup>a</sup>	<i>U</i> <sub>r,f</sub>
Long-term drift at annual limit value	U <sub>d,l,la</sub>
Difference sample/calibration port	<i>u</i> <sub>Dsc</sub>
Uncertainty of the test gas	u <sub>cg</sub>

<sup>a</sup> For the calculation of the combined standard uncertainty, either the uncertainty due to the repeatability standard deviation at the annual limit value or the uncertainty due to the reproducibility standard deviation under field conditions has to be taken, whichever is greater.

#### 6.29.5 Assessment

Minimum requirement met? yes

#### 6.29.6 Documentation

The calculation of the expanded uncertainty was carried out according to the requirements of Annex E of the DIN EN 14662-3: 2016-02. Tab. 6.21 and Tab. 6.22 show the expanded measurement uncertainties of the laboratory tests and the laboratory and field tests, respectively, which were calculated using the process parameters determined in the laboratory and field tests.

#### Tab. 6.19: Expanded measurement uncertainties for GC 80001

Rel. expanded uncertainty laboratory + field

Parameter	Minimum requirement		Value		benzene- nc. C <sub>t</sub>	Sen	sitivity coefficient	1	K <sub>min</sub>	)	K <sub>max</sub>		u	u <sup>2</sup>	
Repeatability standard deviation	≤ 0,25 µg/m³	0,02	µg/m³	5,3	µg/m³							0,005	µg/m³	0,00002	(µg/m³)
Largest deviation of linearity	≤ 5,0 %	-2,15	%	2,7	µg/m³	1		1	÷.,			-0,06	µg/m³	0,004	(µg/m³)
Change of the sample gas pressure	≤ 0,1 (µg/m³)/kPa	0,07	(µg/m³)/kPa	36,2	µg/m³	0,07	(µg/m³)/kPa	80	kPa	110	kPa	0,16	µg/m³	0,027	(µg/m³)
Change of the surrounding temperature	≤ 0,08 (µg/m³)/K	0,03	(µg/m³)/K	38,7	µg/m³	0,03	(µg/m³)/K	273	к	303	к	0,06	µg/m³	0,003	(µg/m <sup>3</sup> ) <sup>2</sup>
Change of the electrical voltage	≤ 0,08 (µg/m³)/V	0,001	(µg/m³)/V	36,2	µg/m³	0,00	(µg/m³)/V	210	٧	245	V	0,002	µg/m³	0,000003	(µg/m³) <sup>a</sup>
Interfering component H <sub>2</sub> O (19 mmol/mol)	≤ 0,015 (µg/m³)/(mmol/mol)	0,012	(µg/m³)/(mmol/mol)	5,3	µg/m³	0,0121	(µg/m³)/(mmol/mol)	0,6	Vol-%	2,1	Vol-%	0,162	µg/m³	0,02615	(µg/m³)
Carry over (memory-effect)	≤ 1,0 µg/m³	0,10	µg/m³	44,5	µg/m³	1.00		12			1	0,01	µg/m³	0,00004	(µg/m³)²
Reproducibility standard deviation under field conditions	≤ 0,25 µg/m³	0,17	µg/m³	1				1				0,17	µg/m³	0,03	(µg/m³) <sup>s</sup>
Long term drift at span-value	≤ 10,0 %	-7,8	%	35,1	µg/m³					) [		-0,22	µg/m³	0,050	(µg/m³)
Short term drift at span-value	≤ 2,0 µg/m³	0,32	µg/m³	36,2	µg/m³	1		1							
Difference sample/calibration port*	≤ 1,0 %	k. A.	%												1
Maintenance interval	14 Tage	3	Monate	1		1		1.1							
Availability	≥ 90 %	100,00	%												
test gas	k. A.	2,1	%	1 = = =		1		1		1		0,05	µg/m³	0,003	(µg/m <sup>3</sup> ) <sup>2</sup>
Benzene annual limit value	5,0	µg/m³		*no sep	parate cal	ibration (	port	-			-	2			
Combined standard uncertainty laboratory	0,25	ppb								X <sub>min</sub>	Minimu	m value (	of the influ	lence quan	tities
Relative expanded uncertainty laboratory	10,1	%								Xmax	Maximu	m value	of the infl	uence quar	ntities
Comb. standard uncertainty laboratory + field	0,38	ppb													

15,1

%

#### Tab. 6.20: Expanded measurement uncertainties for GC 80004

Parameter	Minimum requirement		Value		enzene- nc. C <sub>t</sub>	Sen	sitivity coefficient	)	( <sub>min</sub>	)	K <sub>max</sub>		u	u <sup>2</sup>	
Repeatability standard deviation	≤ 0,25 µg/m³	0,02	µg/m³	5,3	µg/m³	5		1.1				0,01	µg/m³	0,00003	(µg/m³)²
Largest deviation of linearity	≤ 5,0 %	-2,97	%	2,7	µg/m³	(		1.1	- 1	1.1	1.11	-0,09	µg/m³	0,007	(µg/m³)
Change of the sample gas pressure	≤ 0,1 (µg/m³)/kPa	0,06	(µg/m³)/kPa	36,2	µg/m³	0,06	(µg/m³)/kPa	80	kPa	110	kPa	0,13	µg/m³	0,018	(µg/m³)
Change of the surrounding temperature	≤ 0,08 (µg/m³)/K	0,06	(µg/m³)/K	38,7	µg/m³	0,06	(µg/m³)/K	273	к	303	к	0,12	µg/m³	0,02	(µg/m³)
Change of the electrical voltage	≤ 0,08 (µg/m³)/V	0,002	(µg/m³)/V	36,2	µg/m³	0,00	(µg/m³)∕V	210	V	245	٧	0,006	µg/m³	0,0000	(µg/m³)
Interfering component H <sub>2</sub> O (19 mmol/mol)	≤ 0,015 (µg/m³)/(mmol/mol)	-0,006	(µg/m³)/(mmol/mol)	5,3	µg/m³	-0,0060	) (µg/m³)/(mmol/mol)	0,6	Vol-%	2,1	Vol-%	-0,080	µg/m³	0,00643	(µg/m³) <sup>a</sup>
Carry over (memory-effect)	≤ 1,0 µg/m³	0,29	µg/m³	44,5	µg/m³	1		110			1.1	0,02	µg/m³	0,000	(µg/m³) <sup>2</sup>
Reproducibility standard deviation under field conditions	≤ 0,25 µg/m³	0,17	µg/m³					T.				0,17	µg/m³	0,03	(µg/m³)
Long term drift at span-value	≤ 10,0 %	-4,6	%	35,1	µg/m³	1		11.00				-0,13	µg/m³	0,018	(µg/m³)
Short term drift at span-value	≤ 2,0 µg/m³	0,43	µg/m³	36,2	µg/m³	1.								1	
Difference sample/calibration port*	≤ 1,0 %	k. A.	%	1.000											
Maintenance interval	14 Tage	3	Monate	1		1		-							- 1
Availability	≥ 90 %	100,00	%	1.		ĭ. =					1	[			
test gas	k. A.	2,1	%			1				-		0,05	µg/m³	0,003	(µg/m³) <sup>2</sup>
Benzene annual limit value	5,0	µg/m³		*no sep	arate cal	ibration	port								
Combined standard uncertainty laboratory	0,22	ppb	2.4.4							Xmin	Minimu	m value o	of the influ	ience quan	tities
Relative expanded uncertainty laboratory	9,0	%								Xmax	Maximu	m value (	of the influ	uence quar	tities
Comb. standard uncertainty laboratory + field	0,31	ppb													
Rel. expanded uncertainty laboratory + field	12,5	%													

# AED

# SYNTECH SPECTRAS

1.1 Order data		
Serial number	80001	
Model	GCX55	
Туре	601	
Orderd by	Synspec BV	Customer nr.:
Order number		Our order nr.:
Order date		
Delivery date	1-1-2021	
1.2 GC description	1	Serial number
Application	BTEX	
Detector 1	PID	P302
Detector 2	-	
Valve 1	AFP ELDV2	147823007
Valve 2		
Pump	KNF-NMP830 KNDC	16.12509264
Sample system	Steursma V02	
Sample loop (ml)		

Sample loop (ml)		
Precon system	Graphsphere 2016	
MFC	Bronkhorst IQ+	M20209146G
Oven	Isothermal	
Analysis column type	CP70003	1025589
Analysis column packing	SY-1	
Analysis column length (m)	26	
Backflush column type	CP70003	
Backflush column packing	SY-1	
Backfush column length (m)	4	

# 1.3 Hardware and software data

Industrial computer type	ASRock	SBC-210			
Processor type	Intel® Ba	ytrail /Baytr	ail-I SoC Pro	cessor	
Memory	4Gb				
Hard disk type	InnoDisk	Corp mS	ATA 3ME4 A	TA Device	
Hard disk size	64GB				
Oparating system	Windows	10 Enterpri	se LTSC 20	19	
Oparating system service pack					QualityMasters
Oparating system version	GCX55-V	V10-64B-V0	1		0
Oparating system language	English (	United State	es)		
Watch dog version	WDAUTO	C			
GC software version	6.4.2.19				ISO 9001
GC software language	Duits				
MX board version	9				
PIC software versions	010B	0204	0305	1.9	(F
Options	temperat	ure controlle	er board firm	ware V3 2018	

1.4 General data

# AED

# SYNTECH SPECTRAS

1.1 Order data						
Serial number	80004					
Model GCX55						
Туре	601					
Orderd by	Synspec BV	Customer nr.:				
Order number		Our order nr.:				
Order date						
Delivery date	1-1-2021					
1.2 GC description		Serial number				
Application	BTEX					
Detector 1	PID					
Detector 2	-					
Valve 1	AFP ELDV2					
Valve 2	-					
Pump	KNF-NMP830 KNDC	16.12509294				
Sample system	Steursma V02	2430				
Sample loop (ml)						
Precon system	Graphsphere 2016					
MFC	Bronkhorst IQ+					
Oven	Isothermal					
Analysis column type CP70003		1025736				
Analysis column packing	SY-1					
Analysis column length (m)	26					
Backflush column type	CP70003					
Backflush column packing	SY-1					

#### 1.3 Hardware and software data

4

Backfush column length (m)

ASRock SBC-210					
Processor type	Intel® Ba	ytrail /Baytr	ail-I SoC Pro	cessor	
Memory	4Gb				
Hard disk type	InnoDisk	Corp mS	ATA 3ME4 A	TA Device	
Hard disk size	64GB				
Oparating system	Windows	10 Enterpri	se LTSC 201	19	
Oparating system service pack					QualityMasters
Oparating system version	GCX55-V	V10-64B-V0	1		
Oparating system language	English (I	United State	es)		
Watch dog version	WDAUTO	)			
GC software version	6.4.2.19				ISO 9001
GC software language	Duits				
MX board version	9				
PIC software versions	010B	0204	0305	1.9	(F
Options	Temperature controller board firmware V3 2018				

1.4 General data



# Manual for the Synspec GCX55 analyser

25-01-2023 Version 1.1



Zernike Campus De Deimten 1 9747 AV Groningen The Netherlands <u>info@synspec.nl</u>



Manual for the Synspec GCX55 analyser 25-01-2023 Version 1.1 ©2021 Synspec

See our sales conditions on our website www.synspec.nl

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# 1. The GCX55 analyser

This chapter presents a Record of revision, an overview of the GCX55 configurations, the location of the components, the gas- and electrical connections.

# **1.1** Record of revision

Version	Revised date	Page	Content	
1.0	2021-01-01		First release	by JWI
1.1	2023-01-24	4, 29	Introduction; Addition of information for time needed to setup the GC and the time for warming up.	HBA

#### **1.2** Introduction

This document is the manual of the GCX55. This manual will be improved and updated without notice. The latest version can be requested at Synspec. The GCX55 is the successor of the GC955. Contact us at <u>info@synspec.nl</u> or call us on <u>+31505266454</u>, or look up your local distributor on our website on <u>www.synspec.nl</u>. Our office hours are Monday to Friday 08:00 to 17:00 CET (GMT +1/UTC+1).

## **1.3** Different series configurations

The gas chromatograph GCX55 is available in 7 different types. These types are optimized for the different requirements of compounds that would be measured, the field conditions, and the different customer's requirements.

A basic GCX55 includes:

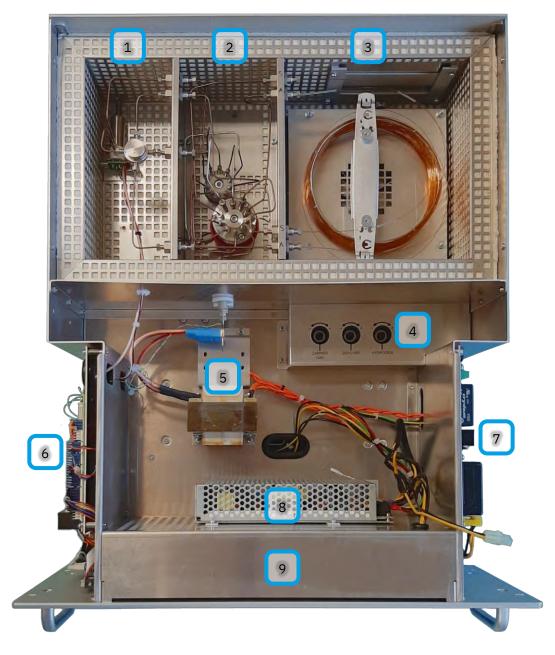
- 1. Sampling and injection unit for gaseous samples: either with a sample loop or with a preconcentration tube, at ambient temperature or cooled.
- 2. Column: a packed column or a capillary column.
- 3. Oven for the temperature regulation of the column: isothermal or temperature programmed.
- 4. One or two detector(s) (PID, FID, TCD, ECD etc.)
- 5. Integrated industrial computer.

An overview of the series biggest differences, where "GCX55-815" belongs to the 800 series.

	Series						
Includes	100	200	300	400	500	600	800
Sample loop	<ul> <li>✓</li> </ul>		<b>~</b>		<b>~</b>		
Pre-concentration tube		<b>~</b>		<b>~</b>		<b>~</b>	
Packed column	<b>~</b>	<b>~</b>					
Isothermal oven	<b>~</b>	<b>~</b>	<	<b>~</b>			
Capillary column			<b>~</b>	<b>~</b>	<b>~</b>	<b>~</b>	
Temperature programmed oven					<b>~</b>	<	
Cooled pre-concentration tube							<b>~</b>

# 1.4 Overview

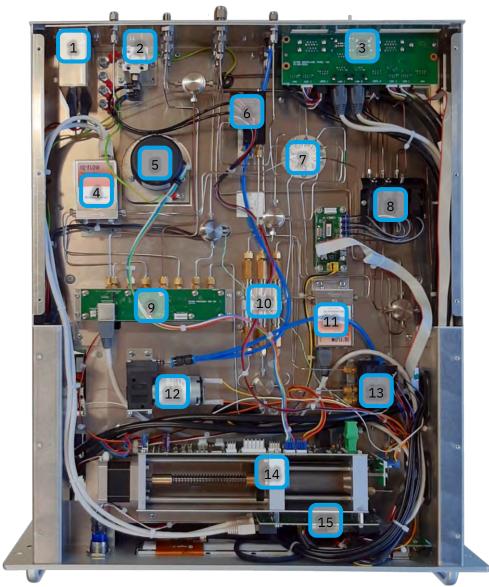
#### 1.4.1 Top view of the GCX55



#### Figure 1 Opened top overview

- 1. Oven left detector(s) compartment with PID and or FID.
- 2. Oven middle valve compartment.
- 3. Oven right column compartment.
- 4. Mass Flow Controllers(s)
- 5. Pre-concentration tube and its power supply.
- 6. MX8 main electronics board (on the left side).
- 7. Power distribution board (on the right side).
- 8. PC Power supply
- 9. Computer (inside)

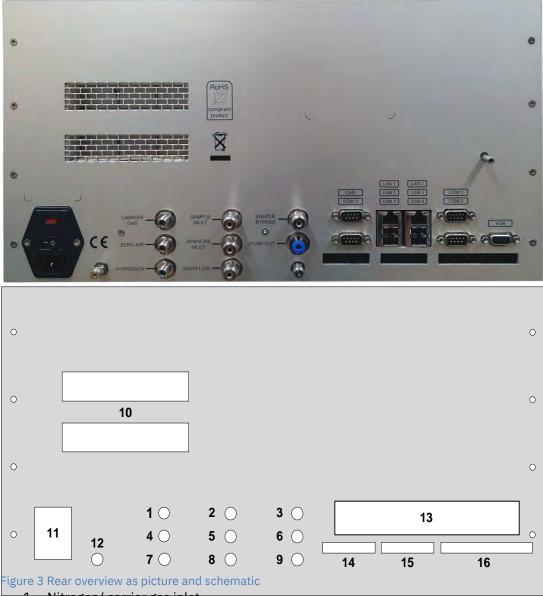
#### 1.4.2 Bottom view of the GCX55



#### Figure 2 Opened bottom overview

- 1. Mains power connection.
- 2. Oven door cylinder.
- 3. Computer and external connector board.
- 4. Mass flow controller(s) for Zero air and Hydrogen
- 5. Oven motor.
- 6. Solenoid block 1 to control the oven door cylinder and 10p valve
- 7. Oven pass though.
- 8. Solenoid block 2 to control the switchbox
- 9. Pressure sensors on a PCB
- 10. Filter(s)
- 11. Mass flow controller for Nitrogen
- 12. Pump
- 13. Solenoid block 3 to control the bypass pump and the sample cylinder or sample pump
- 14. Sample cylinder with control board and sample pressure- and temperature board.
- 15. Mass flow controller board (USB to 4x RS232)

#### 1.4.3 Rear of the GCX55

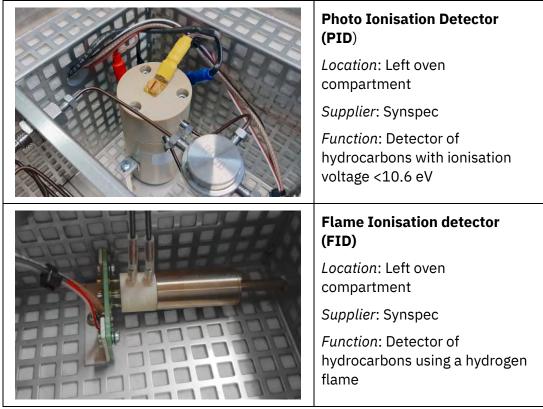


- 1. Nitrogen/ carrier gas inlet.
- 2. Sample inlet.
- 3. Sample bypass.
- 4. Zero air inlet.
- 5. Span gas inlet.
- 6. Pump outlet.
- 7. Hydrogen inlet.
- 8. Overflow outlet.
- 9. Detector outlet
- 10. Oven door ventilation holes.
- 11. Power inlet mains, this includes fuses.
- 12. Stripper outlet
- 13. Backplane panel with computer connections
- 14. Analog input
- 15. Analog out (Voltage/Current)
- 16. Digital outputs (Relays)

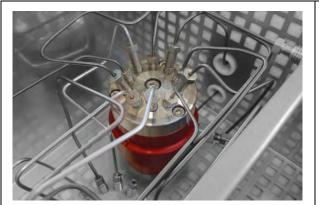
# 1.5 Main elements

#### 1.5.1 Detectors

The GCX55 can be supplied with one (or two) of the following detectors:



# 1.5.2 Other main parts

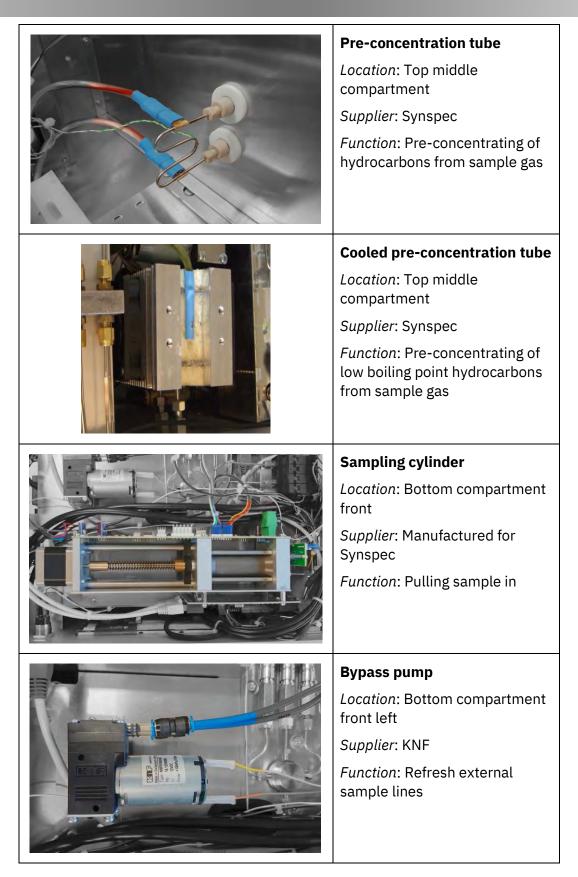


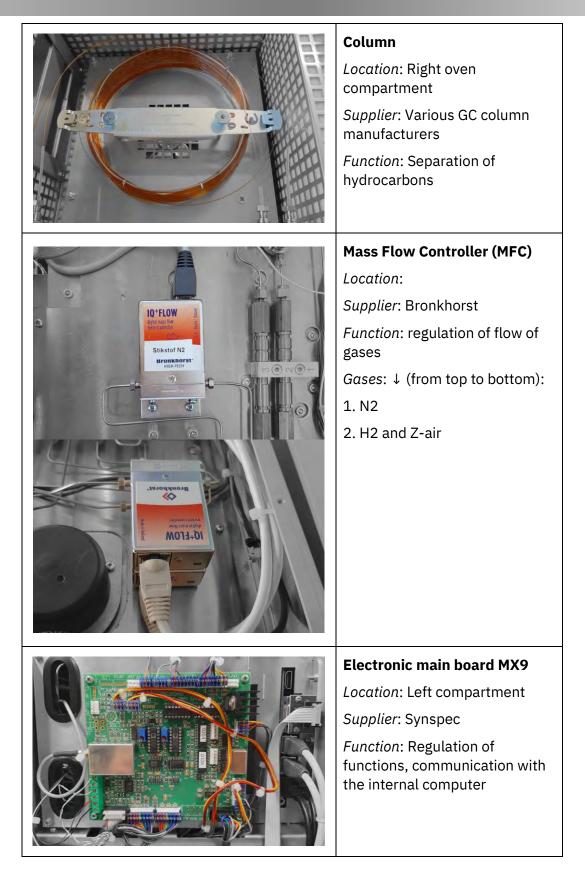
#### **10** port valve

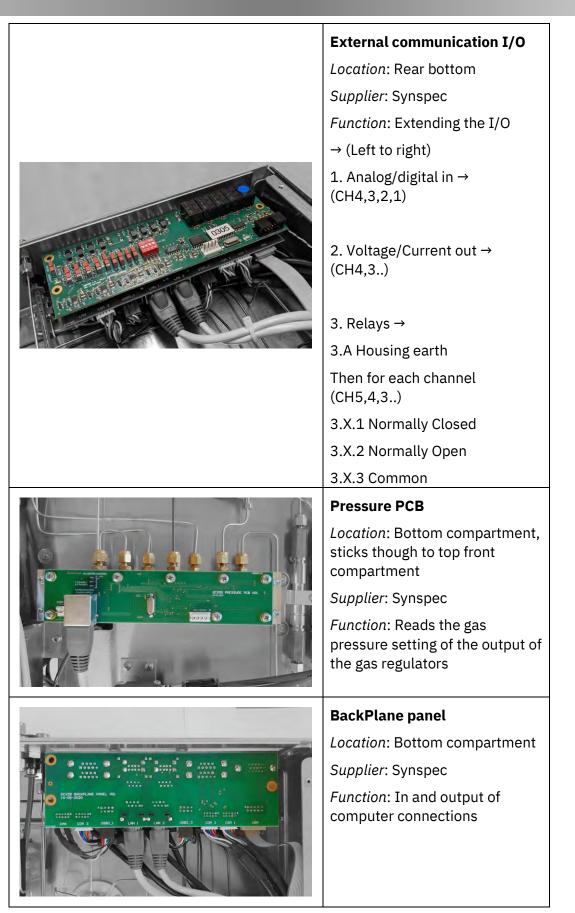
*Location*: Middle oven compartment

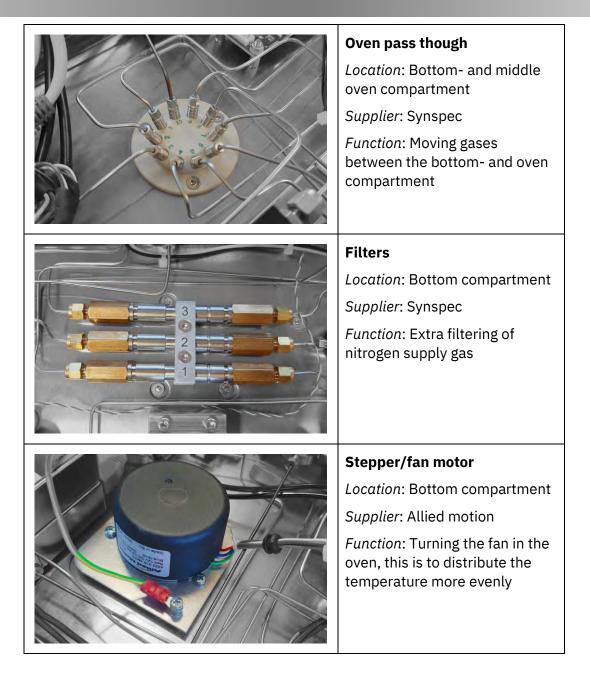
Supplier: AFP

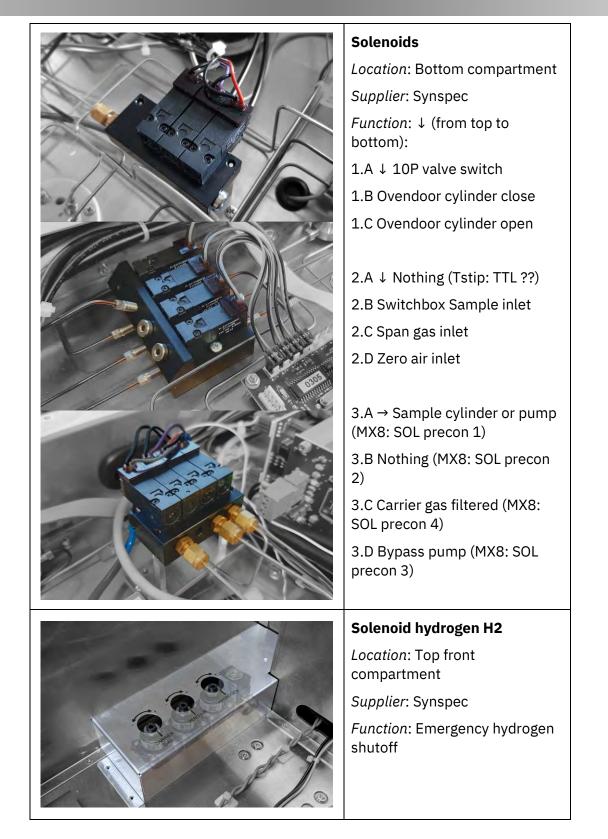
*Function*: switching gas streams











Elle Options Language Help	
W Rundata [1] Filedata [1] Program Edr. [1] Gabbeaton [1] Gabbeaton [1] Autoinnousiation 1000 - 1000 - 1000 -	<i>Location</i> : Shortcut to application on desktop
8000 - 5000 - 4000 -	Supplier: Synspec
3000 - 3000 - 100 - 0 - 1102 <sup>0</sup>	<i>Function</i> : control of the gas chromatograph, communicat
SYNSPEC         90         20         100% (100% 3           SYNSPEC         1977 09-11         06-11.19         00         00           SREpti         2         2         00         00         00           SREpti         2         3         0         00         00         00         00           SREpti         3         3         0         0         00	with external equipment, use interface

#### 1.5.3 Back flush cleaning system

The separation column consists of two parts: a stripper column and an analysis column. The goal is to prevent a long wait for the end of the analysis. With a single column this is caused by the necessity of waiting for elution of the highest boiling compound.

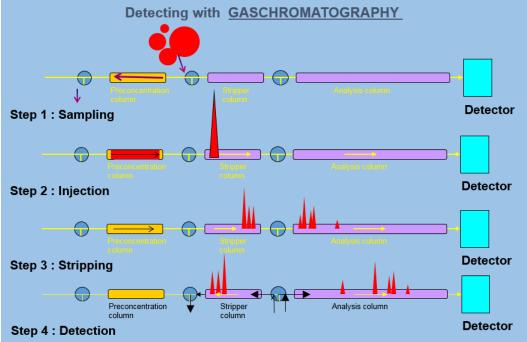


Figure 4 Gas chromatographic separation with a sample loop and stripper/analysis column.

The backflush stripping principle is as follows:

- 1. The air is sampled into the pre-concentration trap.
- 2. Once enough is sampled, the trap is heated, the compounds desorb, after which the whole sample is brought onto the stripper column.
- 3. The compounds start to separate, the lowest boiling compound passes first to the analytical column. Then the higher boiling ones follow. The 10-port valve switching time is set for the slowest eluting compound of interest to get through the analytical column. After this time the flow in the stripper column is reversed by switching the 10-port-valve.
- 4. The lower boiling compounds are separated on the long analysis column. They will be quantified by the detector. The higher boiling compounds are at the same time flushed back.

# 1.5.4 Sampling system

The sampling system consist of a pre-concentration trap, where the sample gas is sucked through by means of a sample cylinder or pump, In the case of a sample cylinder a bypass pump is used to flush the external sampling lines.

### **1.6** Flowchart gases

Note that *Figure 5* is a combination of multiple models of the GCX55. The colours indicate the sections that apply only to the type listed in the middle of that section (e.g. *<Only with PID>*) Note that the "Oven passthrough" is the passthrough from the bottom of the machine into the oven, this does not mean the pre-concentration tube is located in the oven, it only means that is it on that side of the oven passthrough.

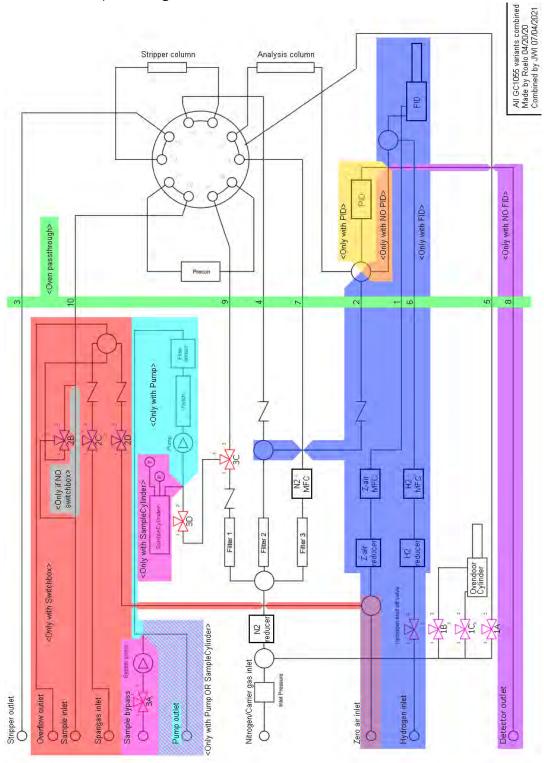


Figure 5 Flow chart

### 2. Commissioning of the GCX55

### 2.1 Safety notice

The instrument has been built for semi-continuous online measurement of hydrocarbons in air and other gases.



### WARNING:

The following risks must be considered when working with the instrument:

- The instrument is only to be used by trained personnel.
- A relevant selection of the gases nitrogen, helium, hydrogen and air at a pressure from 4 to 5bar will be connected to the instrument. This gas is provided from compressed gas bottles, the mixtures may be explosive if the correct procedures are not followed. Personnel must be schooled in working safely with these gases. Always fix bottles to a wall before opening them!
- The instrument works with a 230Vac or 115Vac power supply. Inside the instrument there is a high voltage supply of 2 kV in a protective housing.
- The pre-concentration unit will be at a temperature of 100 to 300°C during desorption. The oven will be heated up to a maximum of 180°C, the cooled pre-concentration can reach temperatures below 0°C.
- Detectors must be switched off before opening the analyser, some will emit UV light.

The instrument has been tested for accordance to the EMC directives:

EN 55011:2009 (Including Amd 1:2010) EN 61000-3-2:2014 EN 61000-3-3:2013 EN 61000-6-2:2005 Part 6-2 EN 61000-6-3:2007 Part 6-3 (including Amd 1:2011) IEC 61326-2014 (EN 61326-1: 2013)

### 2.2 Warranty

There is a warranty on the instrument of one year after delivery. The warranty does not include filters, pre-concentration tubes and capillary columns. For a PID lamp there is a two-year warranty.

### A value of the second s

- Loss of data is at own risk; frequent backups are recommended, only the physical drive has the 2 year warranty.
- Inspect the instrument for transport damage. In case of damage contact the supplier within 8 days after receiving the instrument.
- Read 2 before installing the instrument, and 3 before switching the instrument on. *Chapter* 3.3 provides important instructions on QAQC (Quality Assurance Quality Control) procedures.
- Execute maintenance on schedule, please refer to *Chapter 4.1* for this.
- Always disconnect the gas and power supply before opening the instrument.
- Beware of electrostatic shocks to the internal computer, take care to use appropriate equipment.
- Do not drop (metal) parts in the GC, and do not drop the GC itself.

If there are more questions contact the supplier, or Synspec at <u>info@synspec.nl</u> or on <u>+31505266454</u>.

### 2.3 Unpacking

While unpacking it is advised to check the contents of the delivery. And do a short visual inspection. Some applications require the use of 2 different systems using one application. In such case the second system has contact over an RS-232 port with the main system. The comport that should be used is set in the file "GCX55\_ID.TXT". Both systems possess their own unique serial number.

The gas connections on the back needs to be closed for transport, thus they are delivered closed from Synspec. When removing these plugs store then somewhere safe for later use, when transporting the machine again.

### 2.3.1 Contents



#### Figure 6 Synspec GCX55

The package contains:

- One analyser, or in case of a double system two analysers.
- Power cable(s).
- Keyboard / mouse.
- UTP cross cable.
- USB stick.
- RS232 cable (in case of a double system).
- A tool set with essential tools for connecting the (imperial measured) gas connections.
- This manual.
- Any spare parts ordered with the analyser.

#### 2.3.2 Visual inspection

If the analyser has been long in disuse, the analyser should be checked for obvious problems before start-up. As an example are lose parts in the oven compartment these can do much harm to the ventilator, or lose parts in the computer compartment which can create shorts. For reference pictures and diagrams are listed above, for example *Figure 1* can be used for information on the internal lay-out.

Instrument identification:

- 1. With each analyser comes a test report with serial numbers of many essential parts, this also serves as part of the warranty.
- 2. The test report identifies many manufactured parts in the instrument. If the application is extensive, the report is in the form of a FAT, a factory test report.
- 3. The GC comes with a range of settings which are saved in the GCX55id.txt, this is the analyser identification file.
- 4. It is advised to make a back-up of the original settings of the GC directory. Synspec also makes a copy of all original test data and these can, in case of loss, be supplied in form of a new hard drive with the original set-up.
- 5. The serial number and production date are mentioned on the serial number tag at the rear side of the instrument.

### 2.4 Installation

#### 2.4.1 Requirements and information

A quick overview of the requirements is handled in this section.

### SYNSPEC GCX55 SERIES 601 Benzene ANALYSER WITH FID or PID

TECHNICAL DESCRIPTION	FID or PID detector. Lowest detection level for benzene 0.1 ppbV. Range: standard 0-20 ppbV, with software adaptations up to 300 ppbV possible.
CERTIFICATES	In progress
STANDARD CALIBRATION	Standard 4 point calibration provided for BTEX in range 4 to 16 ppbV.
EXTRA COMPOUNDS AVAILABLE	The software of the analyser is prepared to measure BTEX but can measure up to 40 hydrocarbons in total. Provide the hydrocarbons of importance to add. At Synspec, the list is checked with respect to the possibilities of the existing hardware. Please contact Synspec for advice about calibration gas and peak window settings.
REPRODUCIBILITY	Typical <3% at 1 ppbV (benzene, with capillary column), temperature controlled room
GAS CONSUMPTION	Nitrogen: Quality 5.0; 4 to 5 bar; approx. 15 ml/min for PID and 50ml/min for FID. Hydrogen: Quality 5.0; 4 to 5 bar; 25 ml/min Zero-air: Quality 5.0; 4 to 5 bar; 250 ml/min + 50ml/min for zero point at calibration
DIMENSIONS	19" rack, 5 standard Height Units (224mm), With is 430cm, with frontpanel 483mm, Depth is 552mm. Weight is 19.5kg for PID and 20kg for FID analyser.
HARDWARE	Internal industrial x86 based computer, suitable for measuring and saving data up to 10 years. Hard disk, full color touchscreen, various data connection options.
COMMUNICATION	Direct control via touchscreen, keyboard or mouse. External data communication via RS232, analogue and digital outputs via TCP-IP. Standard available protocols: ASCII terminal, HessenBayern, Gesytec and MODBUS; other protocols on demand.
	Windows 10 embedded and GCX55 software. Direct control via touchscreen, keyboard or mouse via remote host (RS232/modem) or Ethernet. Software for running the analyser and demo version for data evaluation and reprocessing on desktop is included.
POWER DEMAND	230VAC 50-60Hz, 500VA at startup for approx. 1 minute and 300 VA thereafter. (GCX55 with 115VAC power supply is available at request)

#### 2.4.2 Placement of the analyser

It is advised to provide the instrument a ventilation space of 1 Standard Height Units at the bottom and top, as is it advised for it to be mounted on a rail. If the GC is placed on a metal or wooden board, please pay attention of ventilating the bottom, either by using a ventilated housing or by using a perforated board.

Do not forget to reserve a place for keyboard and mouse, preferably a board on slides, which can be placed 10 to 50 cm below the GC.

For standard applications it is not advised to use a room temperature regulation that results in temperature changes of more than 5°C per hour. Stable retention times are best achieved when the room temperature is stable, so do not place the instrument in the direct airflow of the air conditioning.

#### 2.4.3 Gas connections

The stainless-steel Tee that is delivered for the sample tubing is connected to 2 connections on the GC, and place the sample filter in the last piece of tubing before the sample Tee.

#### Gas pressure regulators and connections

- All fittings to the instrument must be 1/8" Swagelok-compatible and in stainless steel
- Pressure regulators must be of gas chromatographic quality, i.e. must be dust free and may not absorb or emit hydrocarbons. As regulators and the connections to bottles are not standardized, the best thing to do is obtain advice from a good and reliable local gas supplier
- Take care: the pressure-drop over the internal pressure reducer should be at least 0.5bar. The values advised below are based on this offset
- All pressure readings are provided in Standard atmosphere [atm] which is about ~1bar
- For the carrier gas Nitrogen, gas chromatographic quality means: use a nickel-plated bronze two stage regulator with steel membrane, range 0 to 4 or 0 to 10bar. Use bottles with a maximal pressure of 200bar
- For calibrating gases a stainless-steel regulator with high quality steel membrane is preferred
- When exchanging gas bottles: pressure of 8bar+ on the analyser will damage internal parts

#### 2.4.4 Calibration and validation gas

The Synspec GCX55 can be equipped with an internal switchbox for easy calibration. But the system can also be calibrated with external (manual) switching systems.

The machine will not be able to properly compensate for pressures outside the stated calibration gas pressure. Make sure that undiluted bottles are restricted to the desired flow, a restriction can be used for this. Synspec can also provide these upon request. In which case also make to combine it with an overflow so no pressure would build up too much above the ambient pressure.

### 2.4.5 Sample conditioning

The sample must be filtered as the internal diameter of the inside tubing is mostly 0.5 to 1.0mm. A Teflon filter membrane of 5micron is advised for this, preferably in a 1/8" housing but alternatively in a 6mm or ¼" housing with good connections to the 1/8" tubing. Disposable nylon filters with glass frit or stainless steel filters can also be used as filter.

Only use only FEP or stainless steel, even for short connecting pieces. Do not use other plastics or bronze.

Use different tubing for taking ambient air samples and calibration samples.

The sample line can be connected to central sampling systems with a substantial volume pump.

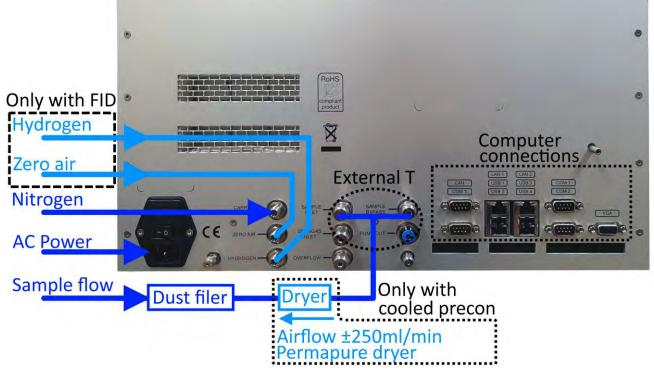


Figure 7 General connection diagram

### 2.5 The first measurements

#### 2.5.1 Turning it on

The instrument can be switched on once all the connections are made and all the panels are installed. Here is a short checklist:

- 1. Are all the gas connections made?
- 2. Are all the gas connections on the right pressure and sufficient?
- 3. Are the gas connections checked for leaks?
- 4. Are the electrical connections made?

Failing to meet these points, could block the machine from working properly.

#### 2.5.2 Starting the program

After starting up the program is by default on "Rundata" as show in *Figure 8*. It either automatically starts (when this setting is set) or it waits on a manual start to start the run. If this is the first actual run, the background zero adjust signal will be set, after which the program starts. The detectors might need some time to stabilize, so the first reset may take a minute. While in automatic mode it will start either immediately or as soon as synchronization is reached. This screen then shows the current gas chromatogram live.

#### 2.5.3 The first run

By default the GC software starts after booting up, but there is also a shortcut to this application placed on the Desktop. When the application start, a self-check is performed on the electronics, and the oven is heated to the set application base temperature. Only when all checks are successful the program will continue.

S	GCX55; Act	tual run												—			×
<u>F</u> ile	<u>Options</u>	<u>L</u> anguag	e <u>H</u> e	elp													⊻ Tabs
. 🕲	Rundata	📃 Fileda	ta 🍳	Prog	gram Edi	t 🟒	Calibr	ation	$\mathbb{M}$	Graph	ıs 🔟	Au	toline	arisatio	n	₿ I/(	D-state
	10000 - 9000 -	-														1	x1
	8000																×10 ×100 ×1000
	7000																1000
	6000	1														Up	date
	5000	1															
	4000	-															
	3000																
	2000	-															
	1000	-															
	0 -	-															
,	-1000- 1 2	3 4	5	6	7 8	9	10	11	12	13	14	15	16	17	18	19	20
		Enabling PIC	)			0.00.		10	20	3	0		100%	100%	50%	5 259	6 10%
	SYN	SDI	· C		19	77-09	-11 0	6:11:1	9							STAR	TRUN
	<u> </u>		- 1	-	<u>PID</u> Smp Cnt		0 Pr	essure	0.0	Barl –	venTe		0.0°C			STOP	RUN
GC	X55				Sample Pi	ess	0	Flow	0.00	cm -	Ambient PreconT	. –	0.0°C 0°C			ABOR	TRUN

Figure 8 The FileData mode

As shown in *Figure 8*, The program contains seven different modes, which are separated with tabs, these are:

- 1. **Rundata** is the current measurement, and shows the measured values (These are also the values on the lower part of the screen)
- 2. Filedata is where all measured chromatograms can be recalled
- 3. **Program Edit** allows changing the run-program settings
- 4. Calibration contains the tools for calibrating
- 5. **Graphs** is used for a quick view of the measured compounds
- 6. **Autolinearisation** can be used for a multi-point calibration.
- 7. **I/O-state** shows the instrument's present state of functioning.

The lower part of the screen always shows data about the current measurement, this includes -but is not limited to- for example the clock date and time, the actual measured raw value of the detector(s), ambient- and oven- and pre-concentration tube temperature.

For some columns there will be the message SOFTFLOWSTART onscreen while the system is starting, this is just informative.

**FID:** When starting, the hydrogen valve is opened. With newly connected lines, the lines are filled with air. The supply of hydrogen limits the burning, and the instrument will not start in the first run. For safety when no flame is detected for 10seconds the hydrogen is cut off, the flame is detected with temperature by a thermocouple. This error progress can repeat 5 times, after which an error message will appear, this will require a manual restart for the program to try again. (after transport it can happen that this process might need to be repeated a few times to flush the tubes, but generally it will start quickly). The burning can also be checked manually by holding a glass or mirror at the exhaust, and checking for drops.

If the instrument is set to start automatically after power failure, and the hydrogen generator is not fast enough to build up pressure again, the machine might error out. In which case it is advised to extent the waiting time for starting the machine.

A new start can only begin after the flame has been extinguished, the thermocouple must be able to see a rise in temperature.

If the hydrogen or air is cut off mid run, the hydrogen safety valve will close, and although the program will continue to run, giving a flat baseline. It is required to restart the FID by clicking "options-restart FID" in actual run.

If the hydrogen supply is not optimal, the amount of retries can be changed in the GCX55id.txt with the line "FIDRETRYMAX=nn" to a value of maximum 15.

### 2.5.4 Data file

In "Filedata: File - Load datafile" measurements can be recalled. The default format the software writes these files is "/D\_YYMM/D\_YYMMDD/M\_HHMMSS.BIN" (for instance

"/D\_2301/D\_230119/M\_031407.BIN" for 03:14:07 on January 19 2023). The X and Y-axes scale is being shared with *"Rundata"* to make comparison simpler.

GCX55; Filedata -		<				
<u>F</u> ile <u>O</u> ptions <u>A</u> nimate data <u>L</u> anguage <u>H</u> elp	⊻ Ta	abs				
🛞 Rundata 🔲 Filedata 💫 Program Edit 🔟 Calibration 📈 Graphs 🔟 Autolinearisation	I/O-state	e				
10000       Height: 11254 Int.area: #11878         900P2enet: 1594025       G-xylene 21.51 ug/m3         Width: 2.4 sec.       G-xylene 21.51 ug/m3         900P2enet 45.78 tagints: 56768       G-xylene 21.51 ug/m3         9000       Int.area: 926122         7000       Width: 2.4 sec.         Toluene 38.11 ug/m3       Height: 30508         6000       Int.area: 11101215         Width: 7.2 sec.       S000         Midth: 18075         1000       Height: 54.sec.         2000       Ethylbenzene 23.19 ug/m3         4000       Height: 54.sec.         5000       M.P-xylene 23.19 ug/m3         4000       Height: 54.sec.         2000       Ethylbenzene 24.69 ug/m3         1000       Ethylbenzene 24.69 ug/m3	>>     1     x1       2     x10     4     x100       4     x100     8     x1000	5				
1 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 1	8 19 20	-				
8 Min. 14 Sec. 0'00" 10 20 30 100% 100% 50% 25% 10%						
SYNSPEC         1977-09-11         06:11:19           PID         0         0         OvenTemp         0.0°C           Smp Cnt         0         0         Ambient         0.0°C           Sample Press         0         0.0ccm         PreconTemp         0°C	START RUN STOP RUN ABORT RUN					

Figure 9 Filedata tab screen

### 2.5.5 Stopping the program

There are three ways to stop the program. Independently of the way used to stop the program the machine makes a reset of all set parameters to the position needed for a restart. This also happens in case of any power failure.

- 1. The correct way is to click the button: "STOP RUN", then the program stops at the end of the present run and this run is still saved normally. After which a new run can start again after making the optional required changes. Or the instrument can be completely shut down.
- 2. When it is required to emergency break the software the "ABORT RUN" button can be used. The program will stop immediately and the windows application can be switched off. But be warned that the next run might be contaminated by residue effects of the aborted run.
- 3. The last way is for (hardware) emergencies only: switching off the power. The last run is not saved and this way of stopping the GC is not advised, as the software has no way to save the current data.

#### 2.5.6 Switching off the equipment:

When the analyser will be stored for a long time, there are a few options:

- To let it run on without doing anything with the results: this will enable to start up again in minutes, the only problem is that it consumes power and gas.
- Stopping the run and leaving it ready for restart: this is generally better. There is less consumption of gas and power, but still a decent fast restart.
- Switching it off completely: in this case make sure that no dust or humidity comes in.

### 2.6 Time needed to setup the GCX55 for BTEX and warming up

The time to make a basic setup for the GCX55 is about 1 hours. This includes the placement of the GC in a 19" rack, connecting the tubings and the electrical cables.

Before doing any linearisation or calibration, let the GCX55-PID stabilise for three days to remove all the moisture from the column and PID. This is needed because the PID is highly sensitive to moisture. The FID needs to warm up for a half hour only, because the FID is far less sensitive to moisture.

Time to perform a linearization (PID only) is automated but takes about 10 hours. Linearisation must be done after replacing the N2 bottle and when maintenance and/or replacement of PID has been done.

Time to manually calibrate the GCX55 takes about 4.5 hours for 5 concentration points. Each concentration point shall be measured 3 times. Each BTEX run takes 15 minutes.

When just one concentration of spangas is available, the GC can be calibrated automatically using more or less amount of sample cylinder steps.

### 3. Working with the GCX55

### 3.1 Continuous measurements

A well-adjusted instrument will run for two to four weeks without needing attention. Of course, the gas supply must be sufficient and calibrations should be made following the quality standard, and it is advised to set the program for automatic restart after power failure.

### 3.1.1 Retention time and peakwindow

The calculated retention time can be enabled in "Filedata: Options - Select screenelements" as shown in *Figure 10*. The exact retention time depends on a lot of factors like temperature, column type, flow rate, etc. This has been pre determent and set by Synspec, and if these factors do not change drastically, the retention times does not much either. It is expected that the retention times shift a minimally.

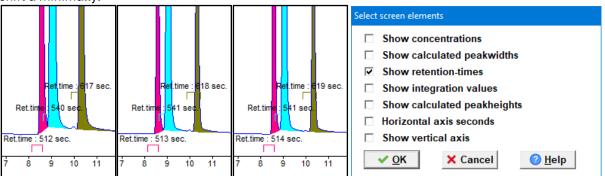


Figure 10 Retention time variations, and the window Filedata: Options - Select screenelements

Properties of the retention time windows must be set carefully in "Filedata: Options - Edit peakwindows" as shown in Figure 11, some notes about these include:

- They should not be too broad to prevent interference from other components. However, they can be narrower than the real peak, as the program calculates the peaks beginning and end
- A few seconds extra should be included for small shifts in retention time
- They should not be based purely on the first run; the machine would not have had the time to stabilize.
- Limit the change of external factors; impure sample, substantial temperature and/or humidity fluctuations, will cause the retention time to shift.

	Substance		From	To (sec.)	integration
1	Benzene	·	187	208	attributes
2	Toluene	•	296	321	attributes
3	Ethylbenzene	•	489	517	attributes
4	M,P-xylene	·	519	553	attributes
5	O-xylene	•	593	629	attributes
6	None	·	0	0	attributes
7	None	·	0	0	attributes
8	None	•	0	0	attributes
9	None	·	0	0	attributes
10	None	•	0	0	attributes

Figure 11 The window Filedata: Options - Edit peakwindows

#### 3.1.2 *Temperature graph*

The oven temperature graph can be enabled in "Filedata: Options - Select graphs for viewing" as shown in *Figure 12*. The "FileData" tab will then show these values, in this case "Set temperature" is black and "measured temperature" is red, note there is an offset in the displayed baseline. If these <u>do not look like each other and/or are not stable wait for another run until it is</u>.

File ViewMode Options Animitedata Language Help		
2	Options	
	Oven temperature	<ul> <li>Oven temperature (setpoint)</li> </ul>
L Hestocheu Dig trivit	Preconcentration temperature	Sample flow pump speed
	Ambient temperature	Sample
	Sample temperature	Ovendoor open/close
	Sample flow	Injection valve
at i .	Sample pressure	Purge
1 2 2 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20	Inlet pressure	Pump
0 10 20 20 100% 100% 2006 2006 2006 2006 2006 2006 2006 2	Column flow	-
Image: Constraint of the second sec	<u> </u>	× Cancel

Figure 12 The Oven temperature graph, and the window Filedata: Options - Select graphs for viewing

### 3.1.3 Synchronisation of measurements

Run settings					
<ul> <li>Restart at discrete time, every 15 minutes</li> <li>Restart immediately after end of RUN</li> </ul>					
✓ Automatic RUN start after software start					
Overwite old datafiles if harddisk is (almost) full					
Generate raw ASCII datafiles					
Delay validation for 1 value hours after software start					
✓ <u>O</u> K X Cancel Ø <u>H</u> elp					

Figure 13 The window Program Edit: Settings - RUN settings

It is either possible to synchronise the measurements based on time, or continue running and take as many as possible. In "Program Edit: Settings - Run settings" (see *Figure 13*) this behaviour can be selected.

- 1. In the first case the machine will wait with starting the next measurement until the computer clock reaches the right minute. If a synchronization of 10 minutes is used, then make sure that the set the cycle time under 10 minutes (minimal 15 seconds shorter). This is to allow for data saving and zero-adjustment before the next cycle begins.
- 2. In the second case, the machine will always start immediately when the run has ended and is ready.

It is advised to use "automatic restart" for when a power loss occurred, this would automate the start-up process further, and make sure no measurement opportunity would be lost.

### 3.2 Quality assurance and quality control

### 3.2.1 Quality control

Quality assurance and quality control of systems is a four-step procedure:

- 1. Production Quality Control
- 2. Standard maintenance
- 3. Technical Quality Control
- 4. Measurement Quality Assurance

At Synspec the analysers are tested according to a standard test protocol. The results of tests are provided in the copy of the original test sheet, the calibration line is printed out. For all analysers a factory acceptance (FAT) report is made.

In *chapter 4.1* there is a list proved for maintenance, it is advised to follow this for optimal performance. Synspec provides standard maintenance packages for these.

To further ensure optimal performance the following points should be observed and taken care of:

- Watch for Alarm messages (on screen)
- Visually inspect the baseline and peak shape
- Stability and correct change of flow and pressure
- Check of recorded data; the baseline, peak shape, temperature control, and sampling control as shown in *Figure 15*

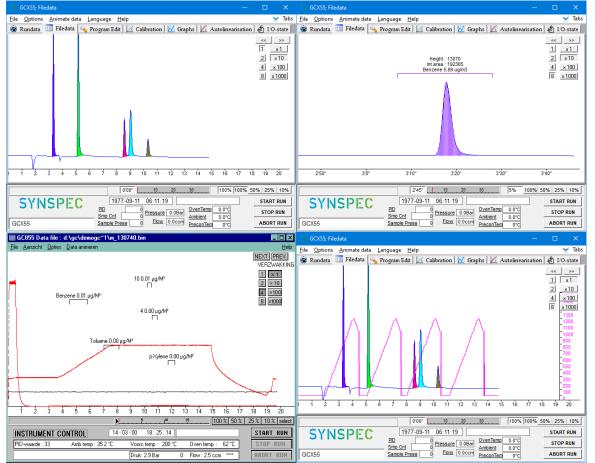


Figure 14 Check of the 4 main elements of program quality check: baseline, peak shape, temperature control, sampling control

### 3.2.2 Quality assurance

A schedule of regular checking the generated data is required for proper quality assurance. To start, check the results of the data logger on retention time stability. After which the pattern of the measurements can be checked. If these are both as expected the individual chromatograms can be inspected. In *chapter 4.1* is a list provided that states the frequency for validations, backups and such. These are advised to be followed for optional quality.

### 3.2.3 Standard Operating Procedure (SOP)

A standard operation procedure manual is necessary to achieve a standardised way of work. Every organization must make its own SOP that is adapted to the goal of the measurements, the exact configuration of the analyser, the calibration equipment, the data transmission, and reporting method.

### **3.3** Validation and Calibration

An automated and periodical validation of the calibration can be toggled in 'Rundata: Options -Activate autovalidation'. The data will then be saved to the validation directory instead of together with the normal measurements.

### 3.3.1 The principle of calibration

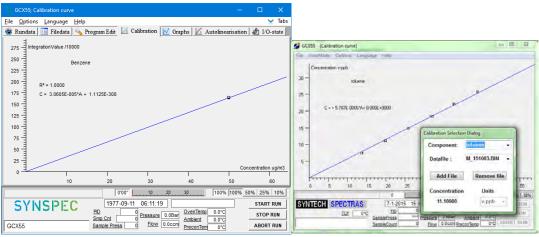


Figure 15 Calibration line for a single point and a multi-point calibration

A calibration is used to determine the conversion factor for automatic calculation of concentrations from the area values. In addition it is also used to check if the machine is functioning correctly. A validation is performed to see if the calibration is still within the set limits for the QAQC requirements. The frequency of these procedures, the number of points determined, and the number of repetitions per point depend on your quality control scheme.

#### 3.3.2 Measuring calibration mixtures

For optimal calibration the peak windows should already have been setup properly as described in *Chapter 3.1.1*.

To start the calibration apply the calibration gas to the machine, with a Switchbox this would be an automated process.

To save and use the calibration select what to do with the data in 'Rundata: Options - Set calibrationmode' There are 3 options as shown in *Figure 16*.

- Start new: Voids all old calibration data, and the present calibration is no longer active
- Add data: This can be used to add another compound, concentration, or validation point.
- Normal data: This is de default analyser mode (no calibration)

Set calibrationmode					
Calibration —					
○ Start new	calibration				
O Add data to current calibration					
Normal da	ta mode (no calibr	ration)			
✓ <u>O</u> K	× Cancel	<u> </u>			

Figure 16 The window Rundata: Options - Set calibrationmode

During the run the concentration values of the calibration mixture should be set in the software, this is done in 'Rundata: Options - Edit sample concentrations' as shown in *Figure 17*. The concentration amount is stored, and only needs to be adjusted if the calibration gas concentration is changed.

Note that this window is only accessible while in a calibration mode, and for ease of use this window also allows the unit type to be set, which is also the unit that values will be shown in after calibration.

If the calibration is done, the calibrationmode should be changed back to normal as shown in *Figure 16*, and the optional tubing or valves states should be restored if this is not done automatically.

Edit sample concentrations						
Component	USE Yes	No	Concentr.	Units		
1. Benzene	0	$\odot$	0.0	ug/m3 🔻		
2. Toluene	0	o	0.0	ug/m3 💌		
3. Ethylbenzene	0	$\odot$	0.0	ug/m3 💌		
4. M,P-xylene	0	•	0.0	ug/m3 💌		
5. O-xylene	0	$\odot$	0.0	ug/m3 💌		
6.	•	۲	0.0	µg/M³ 🔻		
7.	•	۲	0.0	v-ppb 🔻		
8.	0	۲	0.0	mg/M³ ▼		
9.	0	0	0.0	vol-%		
10.	0	0	0.0	gr/M³ ▼		
Select components > 1	10	112	0 2130	3140		
✓ <u>O</u> K X Cancel ⊘ <u>H</u> elp Load defaults						

Figure 17 The window Rundata: Options - Edit sample concentrations

### 3.3.3 Calculating the conversion factor

The measured calibration values are used to make the calibration line. The correlation coefficient of the line is calculated to indicate the linearity as shown in *Figure 18*.

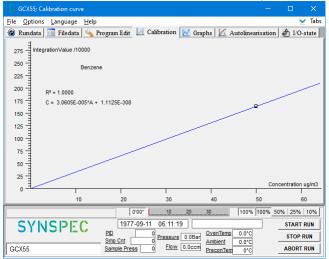


Figure 18 Calibration window

Changing the shown component can be done in 'Calibration: Options - Dataselection calibration files' as shown in *Figure 19*.

This window also allows for the addition of removal of specific files is desired, although this never should be purely done to improve the correlation coefficient, make sure there are good reasons before taking these measures.

Component:	Benzene			
Datafile:	M_19	1345.BIN		
Add Fi	le	Remove file		
Concentratio	n	Units		
50.000000		ug/m3		

Figure 19 The window Calibration: Options - Dataselection calibration files

This is done in the <u>calibration</u> mode of <u>view mode</u>.

- Go to the dialog box in <u>calibration curve</u>, click to accept the calibration files you want to use. If there are concentration values forgotten or entered wrongly, this can be corrected by going in <u>data file</u> mode, loading the chromatogram and correcting the concentration under <u>options\edit sample concentrations</u>. After closing data files which are changed, you are asked to confirm the changes.
- 2. You may remove files from the calculation set, but of course this should not be done to reach a higher "correlation coefficient" but only because there are plausible reasons to reject these measurements for the calibration curve.
- 3. From the measured values in mV the concentrations in ppb or  $\mu$ g/m3 are calculated.

Under <u>calibration\options\settings</u> the unit in which the concentration is calculated can be set. The default unit is the one used when entering the calibration concentrations, but optionally another one can be used: from the measured values in mV the concentrations in ppb or  $\mu$ g/m3 can be calculated.

Settings for calibration: choose the unit in which the data are to be saved

- 4. The calculation of the calibration line can be done in three ways. For normal use the *linear* regression with fixed base point is the best.
- 5. After this calculation has been done, this calibration is used to calculate concentrations out of raw measured data until the next time that <u>start new calibration</u> is activated.

The calculation of the conversion factor

6. Repeat all above steps for every compound.

Settings for calibration							
$_{igsymbol{ imes}}$ Use the following unit for calculation :—							
Cµg/M³ ⊙ug/m3 Cmg/M³ Cv-ppm	⊖ vol-% ⊖ gr/M³						
Standard pressure ( mBar )	1100						
Standard temperature ( °Celsius )	20						
Correction of samplevolume :							
Normal pressure ( mBar )	1013						
Normal temperature ( °Celsius )	20						
Automatic correction of samplevolum	e (P and T)						
Show formulas for calibration line							
Show R2 factor							
V OK X Cancel	() Help						
	- <u>-</u> +						
Figure 20 The window Calibration: Opt	tions - Settings						

Calculate calibrationline						
⊂Calculate baseline using :						
C Linear regression						
C Linear regression with fixed basepoint						
C Non linear regression						
✓ OK X Cancel 1 Help						
·						

Figure 21 The window Calibration: Options - Calculate calibrationline

### 3.3.4 Validation

The quality control system often requires a regular validation check. To do this it is advised to set the calibration sample duration to maximum half an hour. If the option *Add data to current calibration* (under <u>actual run\options\set calibration mode</u>) is chosen, these values will be drawn on the calibration line. The calibration itself will not change unless you recalculate the conversion factor.

### 3.3.5 Calibration overview

Action	Dialog to use	Note
Calibration start		Start the diluting system, and connect calibration gas to the GC
Check peak windows	Filedata: Options - Edit	
	peakwindows	
Take first calibration sample	Rundata	
First calibration run	Rundata: Options - Set calibrationmode	
Notate concentrations	Rundata: Options - edit sample concentrations	If concentrations are not yet known, wait for curve calculation
Repeat previous two steps		Concentration data is copied into next file if they are not changed
End of calibration	Rundata: Options - Set calibrationmode	Reconnect ambient air sampling during the last calibration run, and set the calibration mode back to normal data mode after the last run
Select runs for calibration curve	Calibration: Options - Dataselection calibration files	Check the concentrations, and optionally remove corrupted/bad runs
Calculate curve		Check the line for linearity and correlation
Set calibration factor		After acceptance of the calibration curve it will be the
active		used one until the next calibration.

### 3.4 Data registration and data handling

This chapter will describe the general idea of the data and its format, due to the huge complexity if it not feasible to explain every edge case and user variables in this chapter. For example the runtime measurement data could have its own subfolder *"Rundata/"* instead of it being saved in the main folder as described below. Please take this chapter as a simple guide instead of a rule book.

### 3.4.1 Data registration

The data structure of the generated files, based on the root directory of the software, is as follows:

Subd	irectories	Filename	What its used for
D_YYMM/	D_YYMMDD/	M_ <i>DDHHSS</i> .BIN	Measurements saved in folders per year, per month, per day (daily)
Calibdat/	C_YYMMDD/	C_DDHHSS.BIN	Calibrations (daily)
Validate/	V_YYMM/	V_DDHHSS.BIN	Validations (monthly)
Lindata/	L_YYMMDD/	L_DDHHSS.BIN	Autolinearisations
		RD <i>YYMM</i> .txt	Rundata file with results (monthly)

Placeholder	Definition
YY	Last 2 digits of the year, 0-99
MM	Month of the year, 1-12
DD	Day of the Month, 1-31
НН	Hour of the day, 0-23h
MM	Minutes, 0-59m
SS	Seconds, 0-59s

#### The Rundata text file looks as follows:

Codes: C = Calibrationdata · · · · N = Normaldata · · · · V = Validationdata · · · · B = Baddata@RM3
Code
$\longrightarrow 01 - 02 - 21 \longrightarrow 00: 00 \longrightarrow 0 \rightarrow 12.28 \longrightarrow 972456 \longrightarrow 195 \longrightarrow 18.00 \rightarrow 1471919 \longrightarrow 0304 \longrightarrow 11.61 \rightarrow 0745353333333333333333333333333333333333$
$\longrightarrow \circ 01 - 02 - 21 \longrightarrow \circ 00: 15 \rightarrow \circ \circ 0 \rightarrow \cdot 12.30 \rightarrow \circ \cdot 973857 \longrightarrow \circ \cdot 195 \longrightarrow \circ 17.98 \rightarrow \cdot 1470226 \longrightarrow \circ \cdot 304 \longrightarrow \circ 11.56 \rightarrow \circ \cdot 741983 \rightarrow \cdot 11000000000000000000000000000000000$
$\longrightarrow 01 - 02 - 21 \longrightarrow 00: 30 \longrightarrow 0 \rightarrow 12.23 \longrightarrow 968258 \longrightarrow 195 \longrightarrow 17.87 \rightarrow 1461656 \longrightarrow 0.304 \longrightarrow 11.46 \longrightarrow 0.7355$
$\longrightarrow 01 - 02 - 21 \longrightarrow 00: 45 \longrightarrow 0 \Rightarrow 12.23 \longrightarrow 968064 \longrightarrow 195 \longrightarrow 17.88 \Rightarrow 1462235 \longrightarrow 303 \longrightarrow 11.51 \Rightarrow 739053 \longrightarrow 110000000000000000000000000000000000$
$\longrightarrow \circ 01 - 02 - 21 \longrightarrow \circ 01 : 00 \longrightarrow \circ \circ 0 \\ \Rightarrow \circ 12.25 \longrightarrow \circ 970004 \longrightarrow \circ 195 \longrightarrow \circ 17.88 \\ \Rightarrow \circ 1462414 \longrightarrow \circ \cdot 304 \longrightarrow \circ 11.49 \\ \Rightarrow \circ \circ 73747 $
$\longrightarrow \circ 01 - 02 - 21 \longrightarrow \circ 01 : 15 \longrightarrow \circ 0 \\ \diamond \circ 12 . 29 \longrightarrow \circ 973099 \longrightarrow \circ \cdot 195 \longrightarrow \circ 17 . 92 \\ \rightarrow \circ 1465728 \longrightarrow \circ 303 \\ \longrightarrow \circ 11 . 54 \\ \rightarrow \circ 740563 \\ \oplus 12 . 92 \\ \oplus 1$
$\longrightarrow 01 - 02 - 21 \longrightarrow 01: 30 \longrightarrow 0 \rightarrow 12.21 \longrightarrow 966405 \longrightarrow 195 \longrightarrow 17.83 \rightarrow 1458305 \longrightarrow 303 \longrightarrow 11.48 \rightarrow 7370000000000000000000000000000000000$

Figure 22 Example Rundata text file

Each line starts with a prefix (which are defined in line 1)

- Calibration data
- Normal measurement data
- Validation data
- S a run where calibration or validation is starting
- Maintenance
- Bad this or the previous run had an essential function error like, sampling, detector, temperature, flow, pressure etc.

After the delimiter (Tab+User\_defined\_delimiter\*) the data in DD-MM-YYYY and time HH:MM is given, but all these fields are marked with the header on line 2, and should explain themselves. Each line/row is ended with a ShiftIn+CarriageReturn+LineFeed

\*The default user defined delimiter is a space, but this can be changed in "Program edit: Settings -> Rundata output text format" to ',' or ';' or '.' or '#' or back to the default space as shown in Figure 23.

Rundata output text format				
Save concentration values with : ✓ measured integration area ✓ measured retention times Use <tab> delimiters with</tab>				
Select network folder for backup				
C:\Lazarus_projects				
✓ <u>O</u> K X Cancel Ø <u>H</u> elp				

Figure 23 The window Program edit: Settings - Rundata output text format

### 3.4.2 Data handling

### As stated before in 2.1 Safety notice "Loss of data is at own risk", backup data regularly if desired

If changes have occurred or made that made the measurement unstable, regularly reprocessing the original files can catch the culprit earlier and a better overall result can be obtained. Thus is it advised after transferring the data to examine the chromatograms on following:

- Unexpected extra peaks
- Unstable baseline
- Shifting retention times
- Changes in the set parameters for temperature, pressure, and flow

This can be done at another PC with the GC demo-version, or in the animation mode.

The machine does not require the old measurement data to be there, so after a successful backup the original files can be deleted to save space on the drive.

### **3.5 Software and electronics**

For more specific info about electrical components like the 'External communication I/O' (for remote control) refer to distributor or Synspec. This info might also later be provided in a longer full manual.

### **3.6 Cooled pre-concentration**

Results with low-boiling and unstable compounds could be improved with the use of a cooled preconcentration tube.

The cooled pre-concentration tube is chilled using Peltier elements. After start-up of the GC, it needs a few minutes to reach the desired temperature. To retain this in continuous mode, the room temperature should be withing specs please read *Chapter 2.4.1* for more information about that. Generally a more stable and lower room temperature is advised for the best results.

To avoid freezing or condensation, drying of the sample gas is required. It is advised to use a Perma Pure dryer, based on Nafion<sup>®</sup>. Be aware of the limitations of this and other types of drying systems, water soluble hydrocarbons tend to permeate through the Perma Pure dryer.

Do not use pre-concentration temperatures below the freezing point in combination with such dryer, this would certainly block the system.

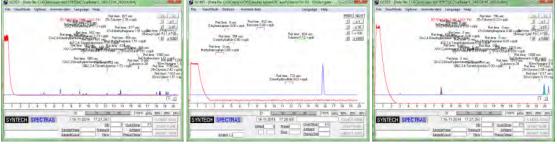


Figure 24 Left normal pre-concentration, middle and right cooled pre-concentration

### 3.7 PID detector stability compensation

The strength of a PID lamp declines slowly by age, this would make periodic calibration necessary. This effect can be followed by adding 'BKWord=TRUE' to the file GCX55id.txt, in which case the sensitivity of the PID lamp is measured on the start of each run and stored as BKword.

An automatic correction of this decline can be enabled with 'BKWADJUST=TRUE' in the GCX55id.txt. The background signal is also influenced by the concentration of hydrocarbons in the carrier gas. When you use this function it is strongly advised to recalibrate every time after changing the carrier gas.

### 3.8 Overview of system alarms

In case of an alarm, the message will appear onscreen and an entry will be made in the logfile. The data communication line registration of a general system alarm is only practical. An example of error messages onscreen is shown in *Figure 25*.

In addition to this, the error LED at the front will also light up:

- Blink in case the analyser can continue to run
- Burn constantly in case a restart is necessary

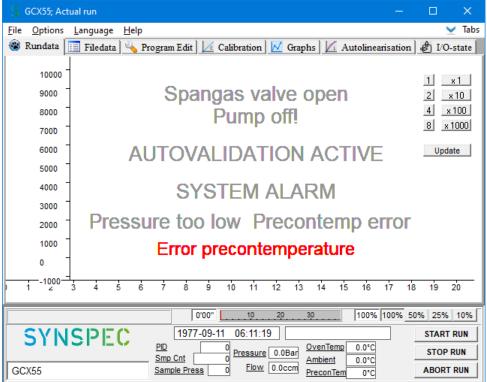


Figure 25 Error messages

Two logfiles are	present: the alar	m log can contai	n the following:
1110 1001100 010	proconti the ata		in the route ming.

Screen message	Alarm file message	comment
System alarm	System alarm	Any system alarm
PID lamp error	PID lamp error	-
FID flame fault	FID flame error	-
Precontemp alarm	Pre-concentration temperature	-
	alarm	
Oventemp alarm	Oven temperature alarm	-
Internal temperature > 40°C	Internal temperature > 40°C	Over measurement in housing
MinFlow alarm	Actual flow too low	-
Maxflow alarm	Actual flow too high	-
Low component alarm	Low component alarm	-
High component alarm	High component alarm	-
Minpressure alarm *	Actuator pressure too low	Blocks running
Maxpressure alarm *	Actuator pressure too high	Blocks running
Sample error	Sample error	
MX8 board alarm	MX8 board error	
	MX8 board error solved	Only in alarm file
Tstrip board alarm	Tstrip board alarm	
	Tstrip alarm solved	Only in alarm file
Stepper alarm	Stepper alarm	
	Stepper alarm solved	Only in alarm file
TCD alarm	TCD alarm	
	TCD alarm solved	Only in alarm file
Startup state checks Minpress	Startup state checks Minpress	
alarm*	alarm*	
Startup state checks Maxpress	Startup state checks Maxpress	
alarm*	alarm*	
Presets error	Presets error	
Hacker alarm (TCP/IP datalogger	Hacker alarm (TCP/IP datalogger	
access)	access)	
< 30 MB HD space left	< 30 MB HD space left	
< 10 MB HD space left (HD full)	< 10 MB HD space left (HD full)	Hard Drive is full
SS flow error on channel ##	SS flow error on channel ##	Only with multi-channel selector
		active
SS false flow error on channel ##	SS false flow error on channel ##	Only with multi-channel selector
		active
Low component SS channel alarm	Low component SS channel alarm	Only with multi-channel selector
		active
High component SS channel	High component SS channel alarm	Only with multi-channel selector
alarm		active
SS comms error	SS comms error	Only with multi-channel selector
		active

The file GCX55.log contains not only alarms but also reports of various actions: Start and stop of the analyser, validation actions etc. The list is not exhaustive.

Start-up and communication	Open and close of all comm ports	
	Missing communication over line	
	Manual or automatic start-up and shut down	
User activity	Maintenance activated / deactivated	
	Autolinearisation started, in addition to the report of results	
	Automatic validation started, in addition to the report of results	
Sampling problems in	Sample error: Overlapping sample strokes. Aborting current stroke	
details		
	Sample error: Purging while sampling	
	DoSampleAverage error: "Steps" is zero. Using NULL_AVERAGE' (ipv run-time error!!)	
	DoSampleAverage error: "Pressure" is zero. Using NULL_AVERAGE') (ipv run-time error!!)	
	DoSampleAverage error: "SampleCount (strokes)" is zero. Using NULL_AVERAGE') (ipv run-time!!)	
	DoSampleAverage error: "Sample pressure" is zero. Using factor 1') (ipv run-time error!!)	
Status error	ERROR: Unable to compensate detector(s)	
	Too many peaks found (max is 1000)	
	pre-concentration tube temp error	
	Oventemp error	
	Presets error	
	Problem in writing rundata and in making back-up	
	Watchdog error	

### 4. Maintenance

Before continuing make sure chapter 2.1 Safety notice has been read carefully.

### <u>The service and maintenance chapters are intended for trained service personnel only.</u> <u>damaged inflicted otherwise it at own risk.</u>

### 4.1 Maintenance frequency

Below is an overview of the advised frequency for maintenance work on a continuously used Synspec GCX55 series 600 and 800.

Frequency	Maintenance work	Estimated required time in hour(s)
every 2-4 weeks	Calibration (PID declines slowly)	0.5-3
every 2-4 weeks	Quality Assurance & Quality Control checks	
every month	Validation, either manual or automatic	
every month	Check gas supply, filter inlet, and Teflon filter blade	
every year	Remove dust from ventilator	0.1
every 2 months	Replacement of micro dust filter for sample	0.1
every year	Back-up and clean old files from the drive	0.5
every year	Checking for ovendoor shutter	0.1-1
every year	Cleaning the FID	1
every year	Cleaning the PID lamp	1
every year	Inspection of sampling piston, cleaning and greasing of piston	0.2 - 1
every year	Renew external sample tubing	1
every year	Replacement of the (cooled) Pre-concentration	1
every year	Replacement of the diaphragm	2
every year	Replacement of the internal carrier gas filters	0.5
every 2-4 years	Replacement of the analytical tubing set	1
every 2 years	Replacement of the PID head	
every 3-4 years	Replacement of the FID (Only when the signal has become bad)	
every 3-6 years	Replacement of the column	0.5

### 4.1.1 No-alarm to outputs during maintenance

When an external data logger is used example, which uses the GC Alarm function it might be useful to use the maintenance function. While the program is in Maintenance mode the alarm state will be overruled and not be send. Data while in this mode will be marked as described in chapter 0.

The maintenance mode can be activated under "Rundata – Option – Maintenance active", this will add a checkmark ( $\checkmark$ ) before that statement while also showing the message on screen with the left duration. This mode should be manual deactivated after maintenance.

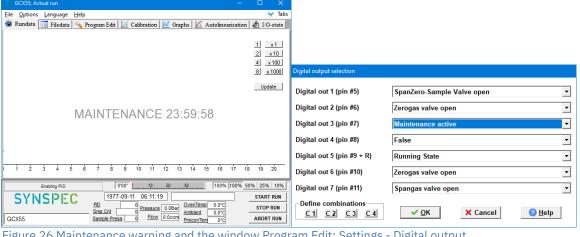


Figure 26 Maintenance warning and the window Program Edit: Settings - Digital output

### 4.1.2 Required equipment

Synspec supplies a toolbox all trainees, and with the delivery of each machine, but it can also be ordered separately. This toolbox contains the essential mechanical tools for maintenance, these tools are shown in Figure 27.

For most maintenance work a well-lighted, dust free, ESD save work area is required. And in addition a multi-meter is needed for checking electrical and electronical problems, and a flow meter is required to measure flows.



Figure 27 Minimum tools needed for standard maintenance and Synspec toolbox content list

Bended pincer 150mm

### 4.2 Opening the machine



### <u>Before continuing make sure chapter 2.1 Safety notice has been read carefully.</u> Again, before removing the cover, disconnect all connections to the machine.

The machine has been improved to be opened more easily for repair. When the top and bottom cover are removed almost all the parts are directly accessible.

### 4.2.1 Opening the top

- Do the pre-steps (shutdown, disconnect, etc)
- Remove the 2+4=6 bolts on each side of the machine as shown in *Figure 28*, (thus 12 in total)
- Open and remove the smaller top oven compartment (the top rear panel)
- Lift up and remove the whole top (including sides) panel



Figure 28 Side screws

### 4.2.2 Opening the bottom

- Do the pre-steps (shutdown, disconnect, etc)
- Remove the 12 bolts as shown in Figure 29
- Carefully remove the bottom plate, and disconnect the fan connector

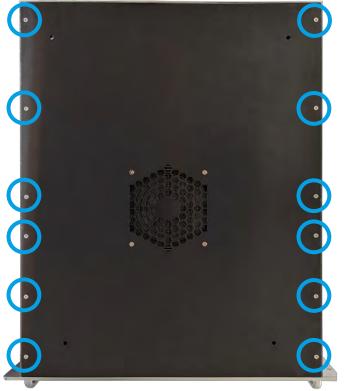


Figure 29 Bottom screws

### 4.3 Leak testing

Leak test should always be performed after maintenance on the tubing.

The leaks can mainly be placed in 2 types, leaks in the carrier gas system, or the sample system. Carrier gas are over-pressure leaks, where the sample leaks are under-pressure. With over-pressure the gas will leak out, but with under-pressure ambient air will be pulled in.

### 4.3.1 Carrier gas



### Before continuing make sure chapter 2.1 Safety notice has been read carefully. It is required to have the machine connected during parts of the maintenance, take the appropriate safety precautions.

In general the below ambient pressure is about 0.2bar below ambient. This means that room air could be sucked into the system in case of a leak, which would influence the first measurements.

This can be checked in the following simple way: you only need a 1/8in Swagelok cap:

- 1. Start the GC
- 2. Start a run, check in the program edit mode when a sampling stroke starts.
- 3. Close the inlet to the 10-port valve with the cap
- 4. Observe that the pressure drops down to a value of 700mbar and the sampling movement stops (this 700 is a setting in the GCX55id.txt file. Other values, down to 400mbar are possible for mountainous conditions.
- 5. Open the inlet again and see the normal pressure and movement come back.
- 6. If you miss a cap, you can cover the inlet shortly with a flat item that you press, in this case most likely a small movement will still be there.

In case you find a negative leak, tighten all connectors in the sampling line and retry.

- The connections of the carrier gas system come from the carrier gas connection at the rear, lead through the left compartment to the pressure and flow controllers with the carrier gas filters and then to the oven compartment.
   First check system outside of the oven with system in switched of position.
   Then check the oven compartment with care, as the system has to be powered on to do this.
   The same gas is also used to switch 3 valves.
- 2. Open the lower plate, set the analyser on the side and test for leaks with leak testing soap or foam.

Leaks on the pressure or flow regulator or on the carrier gas filters are also best detected with leak test fluid. The connections on the pressure regulator are not so easily reached, take you time to inspect and tighten them. The straight and bended tubing unions in this compartment should always be tightened by using two wrenches: be sure to hold one part still while turning the other, in order not to bend the tube instead of tightening the connection.

- 3. Careful: in this test the analyser is powered up with the oven compartment open! Check of the oven compartment: cover the column compartment with the oven lid, so that not too much heat is used. The machine is first checked in the analysis mode with leak tester soap: start up the software and click on abort run. Check all Vici type connections by using the correct 1/4in key.
- 4. The oven is cooled down by opening the slit in the rear. The piston is controlled by 2 valves. The 10-port valve is also controlled by a valve. This valve block may leak, especially if by accident tis has been pressurized to a pressure of over 5bar. The resulting leak is considerable.
- 5. A broken column appears at two moments: either directly after a transport, or a few hours later. In the first case the glass has broken, in the second case the polymer protection has been scratched off. After start-up the column may still hold for a time and then break during heating up or cooling of the tubing.
- 6. The steel tubing is rather strong. The FEP-tubing we use on other parts can only be damaged by bad handling. Replace tubing by identical pieces, contact Synspec.

The flow of the analysis column and the flow of the stripper column should be checked during maintenance: they should stay the same between services. The flow is provided in the original data at the FAT, you should make it part of your QAQC procedure. Check them in both configurations of the 10-port valve, take care not to measure at the moments the oven door is opened and shut, as the flow will then shortly vary.

### Warning: Take out the connector for the oven heating.

The flow that is indicated on the screen is the set flow of the mass flow controller. This is the flow that goes out of the mass flow controller to the analysis column. If the mass flow controller cannot reach the set flow, you can read it on the screen. However if there is a leak after the mass flow controller, the indicator will not react on this. If you suspect a leak, check with soppy fluid at the inline filters, at the oven wall, at the 10-port valve: (you can only do this with the oven cover open, to avoid too much heating, take out the connector for the oven heating from the central voltage supply board.)

The stripper flow can simply be measured at the outlet of the stripper flow at the rear of the GC. If the flow decreases sharply, a leak has occurred. This mostly occurs at the fixation points of the columns.

However, because of vibrations caused by pumps or by transport movements, other unions in the system may start to leak.

At other places leaks may be sought by using soap (Soppy or Snoop, or diluted dishwasher fluid). Retighten the union and check again.

With larger leaks it will also be apparent that the carrier gas consumption has increased. If leak free, the GC's Nitrogen pressure should drop only after 30 seconds after shutdown of the Nitrogen supply.

#### 4.3.2 Sample system

Sampling system is leaking Leaking connections in the sampling line

- 1. Check for this by putting a cap at the sample entrance: the piston strokes should now not be
  - completed. The piston should stop moving and the counter should hold. If the piston moves smoothly despite closing the sample entrance, you have a leak. Check all connectors on the line

### 4.4 **Pre-concentration tube**

Because of the environment the tube is in the filling will slowly decay, and replacement could be necessary to keep optimal performance.

As the trap is expensive due to the elements used for the cooling, Synspec provided the option to exchange the trap. Synspec will recycle the trap except for the trap volume. This recycling is a very specialised job and cannot be executed by clients.

#### *4.4.1 Maintenance steps*

Examen the baseline temperature of the pre-concentration. This can be toggled on/off in "Options – Selects graphs for viewing" as shown in *Figure 30*. The pre-concentration temperature line should have a very steep incline. And the cooldown should be a bit more gradual as shown, But it must have returned to a stable baseline before the next run.

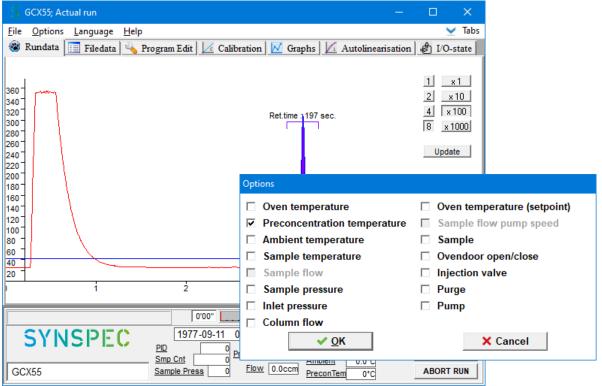


Figure 30 Pre-concentration temperature graph

If the pre-concentration tube is reporting no change in temperature, check if the solder joints have become undone. In addition if a cooled pre-concentration tube is used, check if the unit still moves heat and the heatsink is heating up.

If the rise in temperature is slow and does not reach the setpoint, water might have entered the system in idle time. A replacement would be the best option.

If everything so far seems checks out, the system can be checked on linearity and reproducibility of the system. If there are still no issues, congratulations! Then the unit would be performing optimally

### 4.4.2 Replacement

- Do the pre-steps (shutdown, disconnect, and open machine, etc)
- Locate the filters, and MX mainboard as shown in *Chapter 1.4*
- Loosen and remove the thermocouple wires from the MX mainboard
- Loosen the power wires, for the heated variant these are the 2 connections on the preconcentration tube itself. For the cooled variant follow the fan and Peltier wires, and disconnect them at their respective connections.
- Loosen the tubing on the pre-concentration tube
- Place the new pre-concentration tube in the machine,
- To mount it, reverse the steps; \*mount, connect, and close the machine again
- Check the carrier gas tubing for leaks as described in *chapter 4.3.1*

\*Optionally use some tie-wraps to prevent the delicate thermocouple from breaking off by movement.

### 4.5 Bypass pump

The KNF-pump can be used to pump the sample air up to the entrance. In optimal conditions it has a very long lifetime. But with inadequate filtering, dust may gather in the unit and the pump will get blocked.

If the sulphur dioxide content in your ambient air is high, the bumpers of the pump will become brittle and break. These can simply be replaced by any similar bumpers.

#### 4.5.1 Replacement steps

- Do the pre-steps (shutdown, disconnect, and open machine, etc)
- Locate the pump as shown in *Chapter 1.4.2*
- Disconnect the wires of the pump, take \*caution to remember what wire was connected to
- Unmount the pump
- Place the new pump and bumpers into the machine
- To mount it reverse, the steps; mount, connect, and close the machine again
- Check the whole system for leaks as described in *chapter 4.3*

\*Make sure to not swap the wires of the pump

### 4.6 Filters

To prevent pollution of the carrier gas in the system, the gas is filtered for each use case. The exact location of the filters is shown in *chapter 1.6*.

Besides the scheduled replacement as already told in *chapter 4.1*, it is also advised to replace them when the background ionisation signal becomes too high, and a restart is no longer possible because of it.

It is rather feasible to reuse the filters. To do this it is required to put them in a 200°C oven

put them in an oven for a night, at 200, while flushing them through with Nitrogen 5.0. (Heating without flushing does not work).

### 4.6.1 Replacement steps

- Do the pre-steps (shutdown, disconnect, and open machine, etc)
- Locate the filters as shown in *Chapter 1.4.2*
- Loosen the tube connections with a 5/16in wrench while holding the filter in place with a 10mm wrench
- Remove the old filters, and optionally mark them
- Place the (new and) cleaned filters into the machine
- To mount it, reverse the steps; mount, connect, and close the machine again
- Check the whole system for leaks as described in *chapter 4.3*

It is rather feasible to reuse the filters, the producer to clean the filters is

- Mount the filters in an oven
- Set the oven to 200°C\*, and let Nitrogen (at ~5bar\*) flush though them
- Leave the filters for at least 12hours
- Turn off the flow and oven, and unmount the filters.
- Cap off these cleaned filters, to prevent contamination, and optionally mark them

\*Flushing without pressure or temperature is useless

Head

O-Ring

Lamp

Clamp

Spring

### 4.7 PID

The PID is on the heart of the measurement operation, and requires quite some maintenance to be in perfect condition. All maintenance is already noted before in *chapter 4.1*.

Stable background noise is automatically filtered out by the MX mainboard, and thus the whole amplification range can be used. But reducing external factors is always a better option.

An exploded view with all the individual parts is shown in *Figure 31*.

### 4.7.1 Maintenance steps

- Disassemble the PID as described in *chapter 4.7.2*
- Replace parts that are bad, or scheduled to be replaced
- Check if the window of the lamp is clean, If the window is dirty the lamp window should be cleaned with ammonia or isopropanol
- Check the PID head for corrosion, an example is shown in *Figure 32*



Figure 32 A good and eroded head

### 4.7.2 Disassembly steps

- Do the pre-steps (shutdown, disconnect, and open machine, etc)
- Locate the PID as shown in *Chapter 1.4.1*
- Remove any tubing that is on top of the PID and is in the way
- Loosen the 2 connectors of the tubbing that go to the PID head
- Loosen and remove the 3 Philips bolts on the bottom of the apartment, this will release the PID completely
- Remove the PID from the machine to make it easier to work on
- Remove the 4 electrodes by pulling them carefully from their connectors
- Carefully Loosen and remove the input and output tubing. Do not loosen the connectors completely
- Loose the 3 long hex-bolts from the upper part of the detector and take off the upper part. Take caution that the lamp will be released and could jump out, carefully hold if back if it does so, and try to avoid touching the window
- There are another 3 hex bolts in the 3 clamp ring pieces that need to be removed. One of these clamp rings has the connector, mark the location of this connector so it can be placed back correctly in assembly

Figure 31 Exploded view of the PID

### 4.7.3 Assembly steps

- 1. Make sure to check the direction of the spring. The lamp needs to face the sharp end of this spring
- 2. Lower the spring and lamp back into its casing
- 3. Place and mount back the 3 clamp ring pieces, make sure to line up the connector with the mark, it is recommended to temporary place the 3 long hex bolts as a guidance to their right locations.
- 4. Place the new head on top of the PID
- 5. Insert and tighten the 3 long hex-bolts, be careful to not overtighten. The head can be shifted due to tolerances in the spring and lamp, do not worry about this.
- 6. Push the 4 electrodes back onto their connectors, this can be tough
  - a. Black for high voltage ground, which goes the bottom connector on the side
  - b. Red for high voltage itself, which goes on the side
  - c. Blue for signal ground, which goes on the side of the head
  - d. Yellow for signal data, which goes on top
- 7. Place the complete PID back into the oven apartment
- 8. Insert and tighten the 3 Philips bolts to hold the PID in place
- 9. Add the input and output tubing to the PID back in, be careful to not over tighten, these are not made of metal
- 10. Add the tubing that did go over the PID back in
- 11. Check the whole system for leaks as described in *chapter 4.3*
- 12. Close up the machine again

### 4.8 FID

The FID needs periodically cleaned, and needs to be replaced after its service life.

#### *4.8.1 Maintenance steps*

- 1. Disassemble the FID as described in *chapter 4.8.2*
- 2. Replace parts that are bad
- 3. Clean the FID chimney with a soft brush
- 4. Remove any contaminations from the rest of the FID with a soft brush. \*Except for the thin and fragile filament wire, which should not be touched
- 5. Put on gloves. Take a small glass beaker and fill it with diluted warm hydrochloric acid. Make this by filling the glass for 2/3 with warm water and adding ca 10% commercial hydrochloric acid. (that is a 10% solution, so the end solution is ca 1%)
- 6. Take the FID at the ceramic side and hold the metal part only not the ceramic part for 2 minutes in the hydrochloric acid
- 7. Rinse the unit with water, and let it dry
- 8. Remount all parts. Push the tubing as deep as possible in the Teflon sockets

\*The soldering on the FID and the little spiralled wire should be left untouched

The main maintenance on the FID is the cleaning, 1 to 2 times per year, of accumulated soot. When the FID has been used for 3 years, replacement is often better. However if the sensitivity and noise level is still good, do not replace it.

Clean the FID chimney with a brush. Clean carefully the other parts on the FID itself with a small brush. Take care with the thin filament wire: this material should not be touched. The FID is mounted deep in the instrument: take time to remove the connections and to remount them.

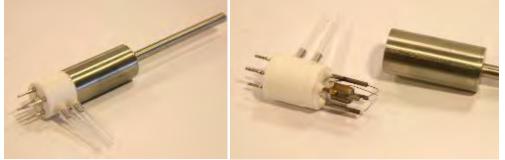


Figure 33 FID complete and taken apart

#### 4.8.2 Replacement steps

- 1. Do the pre-steps (shutdown, disconnect, and open machine, etc)
- 2. Locate the FID as shown in *Chapter 1.4.1*
- 3. Optionally remove tubing that is above or/and in the way of the PID, optionally the middle oven wall could be removed
- 4. Remove and take out the tubing to the FID
- 5. Carefully take the FID out of its socket plug
- 6. Gently move it up and backwards in the same movement and take the chimney out of the hole
- 7. Remove the spring and lay it aside

8. Gently take off the metal part from the ceramic part by turning it. To ease this step it is recommended to warm it up to hands warmth by holding it before turning

#### Procedure:

- 1. Switch off the system, remove the gas connections. Be careful with the hydrogen.
- 2. Remove the oven middle wall to have more space, especially when refitting the FID.
- 3. Take out the T on top of the FID.
- 4. Take out the tubing to the FID.
- 5. Carefully take the FID out of its socket plug.
- 6. Gently move it up and backwards in the same movement and take the chimney out of the hole.
- 7. Remove the spring and lay it aside.
- 8. Gently take off the metal part from the ceramic part by turning it. To make this a bit easier, warm it up to hand's warmth before starting to turn.
- 9. Clean the chimney with a soft brush. Remove any contaminations from the rest of the FID with a soft brush.
- 10. Put on gloves. Take a small glass beaker and fill it with diluted warm hydrochloric acid. Make this by filling the glass for 2/3 with warm water and adding ca 10% commercial hydrochloric acid. (that is a 10% solution, so the end solution is ca 1%). Take the FID at the ceramic side and hold **the metal part only not the ceramic part** for 2 minutes in the hydrochloric acid. Rinse well with water, let it dry.
- 11. The soldering on the FID and the little spiralled wire should be left untouched.
- 12. Remount all parts. Push the tubing as deep as possible in the Teflon sockets.

The FID has to be placed back in the 5 sockets of the small electronics board. This normally does not have to be replaced.

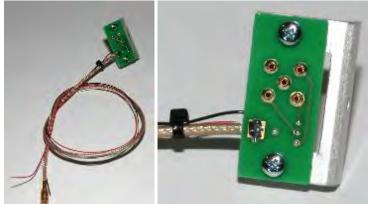


Figure 34 FID print and in detail the 5 connector pins for the signal

### 4.9 File backup

Drive capacity gets bigger every year. Nowadays, it is hard to believe that Synspec started in 1994 with a hard disk of 140MB. Still, the drive will fill up, especially with a short analysis cycles or a double system.

A backup should be made frequently, and a clean-up at least yearly. The most essential thing to copy is always the rundata.txt or the rundatamonth.txt, as this contains the ASCII file with the results of each measurement.

#### 4.9.1 Maintenance steps

- 1. Copy and backup all rundata.txt or the rundatamonth.txt and all the data files to another medium, then delete these files from the drive to free up space. These are all '\*.bin' files in the monthly subdirectories.
- 2. If the GCX55.log is larger than 0.5MB it is recommended to rename it or copy it to another medium and delete the original. A new log file will be generated automatically.
- 3. Make a safety backup of the calibration files and the functioning essentials (GCX55.exe, act\_run.prg, act\_calib.dat, GCX55id.txt) to store together with the bin-files for later reference.

### 4.10 Data communication with external data loggers

Several versions of data logging are available from our side. Also ethernet connection is possible. A problem most frequently occurs in three cases:

- 1. With data loggers: only at the end of a cycle a new value will be transmitted through the RS232 port. This is different from many other instruments that may be present at the same site. So, in many cases, the RS232 port is frequently approached by the customers data logger. If this occurs with instruments that are set to transmit many parameters of measured peaks or of status settings, the system may become very slow or fail. Please set the data logger frequency to the lowest value you can, preferably to once every cycle. If you use a modem for data logging, it should have a speed of at least 54kBps.
- 2. Some types of data logging programs, like the bus programs, have many subtle variations. The GC can in principle be set to communicate, but we need detailed information of the data logging software protocol. We will be pleased to contact the software department of the customer to exchange information.
- 3. An ethernet connection enables quick data transfer. We do not advise to log on to an office network with a central backup. This will mean that each time a new file is saved or a file is changed, a copy will be made over the network. For a standard GC this means that at the end of each cycle the bin-file and the rundata.txt must be copied. At the end of a month this may be too much for the system, which would produce an error message. Because of this message, a line would be added to the file GCX55.log. Therefore GCX55.log would also be backed up, which creates another error, and so on, and the system would get stuck. **So, logging on to a central network is only possible if you can avoid automatic backup**.

# 5. Troubleshooting

This chapter describes the most occurring problems, and their solutions. Before applying the suggested solution (s) make sure the precautions listed in the *Chapter 2.1 Safety notice* have been taken.

To apply most software changes in the GC software, a full "stop run" and "start run" is required, but for bigger changes it might also be that the machine needs to be turned off and on again.

If the problem is consistence, and cannot be solved with the solution(s) provided below, Contact your distributor or Synspec.

### 5.1 Optimalisation

Some main factors into never needing the troubleshooting guide are:

- Using QAQC procedures.
- Training staff and personal.
- Executing all maintenance work (the monthly, yearly, bi-yearly etc).
- Periodically checking the analyser for small changes in properties.

### 5.2 Troubleshooting tables

### 5.2.1 Start-up problems

Symptom	Possible suspect	Solution
Computer does not start up, Windows error message	Loose connectors	Check if the connections in the computer compartment are in place they could have become loose during transport
	Blown fuse	Replace the power fuse, and check at next start-up what happens there may be an electrical problem in the instrument
	Computer failure	Check all the computer connectors, and check if the CPU fan is spinning. In addition try to boot without hard drive or ram stick(s), this should trigger a warning if the motherboard is still functionally
	Drive failure	Check if the pc can make contact to the drive at start up
GC software does not start	Files missing (after power failure)	On the Backup section of the drive is a copy of the original set-up. Compare and copy missing files
	Bootup program changed	By default the GC application is added to the bootup sequence, and a shortcut on the desktop. Re-add the software to these places
GC software doesn't	Demo is started	If by mistake the included demo software is started. Find
show real values		the correct version and start that instead
GC error message (Fault detected by software)	General / not listed	Check the error messages, correct for them, and reboot
	Wrong comport	Check the set MX port and set it correctly. Internal it is likely to be 4 and externally between 1 and 3
	PID lamp fault	Mostly a bad contact in detector that needs to be checked
	Fault in electronic board	Check connectors and restart. Check if a function light lights up at each electronic board at start-up and goes
	Coo our alcunation	put after this. If not check with distributor for help
	Gas supply wrong	Check hydrogen and air supplies and their pressures. After a shut down the hydrogen line contains only air, so
		a slight start-up delay is expected.
		The Hydrogen line can also be check for water droplets
		(wet scrubbers), these will blow out the flame
	Too cold	A cold FID lights verry slowly, try a few more times
	Wrong burning	To start up the system it is often useful to set the
	gas ratio	hydrogen pressure a bit higher

### 5.2.2 "Ready to start" but does not start

Symptom	Possible suspect	Solution
Waiting for synchronisation	Synchronisation not met	It is not yet the set synchronisation time
Background compensation not possible	Noisy FID detector	FID detector can be noisy if contaminated and so the background is within requirements to start, check supply and perform a clean flush
Background near zero	Very weak PID signal	PIDs past their expected lifetime emit almost no light anymore, it is advised to replace within the provided time window
Setting in digital communication interferes	Software has extra settings for special start modes	If the system is connected to external loggers, the program may wait for an external status change. Disconnect this equipment, restart and check if the system does start

### 5.2.3 Measurements are discontinuous

Symptom	Possible suspect	Solution
No new run starts	Stop run has been clicked	Click "start run"
It sometimes misses a run	Cycle time too close to sync time	Set for at least 10 seconds between end of cycle and synchronising. With 30 minutes cycle use at least 15 seconds
Baseline setting to zero takes a long	Noise on baseline	A bad contact, or bad detector itself could be the culprit. Check the contacts and try replacing it
It waits several minutes before a new run	Synchronising time is set incorrectly	Check the synchronising time settings and set the timing at the desired frequency. The program will wait after each cycle for this synchronisation
Problem with writing of (log) files	Log files are substantial in size due to a report of concurring errors	Look for the communication problem in the system (listed in the log), else this will only happen again. Check the size of the log file and the data files. Make a backup and remove the original. If the problem is consistence ask the distributor for possible updates for the firmware (and note the current version)
System reboots, missing data, blue screen	Problem(s) with stability of the computer	Check log file to look for an automatic restart. Observe system to watch for a possible trigger of the stop. The computer and its components can also be checked
"Error PID, oven temperature error, flow error" keep reappearing	Communication problem	Check the commutation connections, these messages are generated by bad communication between electronics of GC and the computer. Contact the distributor for possible updates of the firmware
Stops due to sample integration overflow	Sampling piston does not finish one stroke	Check working of sampling piston, this might also be clocked or needs lubrication

### 5.2.4 No peaks, (almost) flat baseline

Symptom	Possible suspect	Solution
Only very small peaks, compared to before	Leaking connections in the sampling line	Put a cap at the sample entrance, this blocks the piston from completing the strokes, and it will stop moving which result in the step counter to stagnate. If the piston moves smoothly despite closing the sample entrance, there is a leak and all the connectors on the sampling line need to be checked.
	Detector sensitivity problem	Perform a calibration check with calibration gas
No peaks in data file	Setting of peak windows has not been saved.	Set peak windows on a chromatogram, stop and start run after end of cycle. Save the settings by saving the program under its own name in the program edit mode. The saving is required every time the window settings are changed.
No peaks, flat baseline	Sampling valve is malfunction	Check if carrier gas pressure is within the specification. Check sample valve block on left side panel
	Column is broken	Check column system
	No sampling strokes programmed	Change the program or reload older version of act_run.prg
	Injection time too short	If the injection time is set to short, nothing will arrive on the analysis column and thus nothing would be detected
	10-port valve blocked or malfunctioning	No sample is taken, OR the sample is not injected as the valve does not switch
	Pre- concentration heating does not work	If the pre-concentration heating is not working, then no hydrocarbons can be measured. However the PID should provide a dip where the air comes through and the FID should provide a small peak when the air comes through

#### 5.2.5 Unstable retention times

Symptom	Possible suspect	Solution
The peak is identified wrong, because the retention time has shifted	To hight room temperature fluctuation.	Stabilize the room temperature to be within the given machine specs, this can most of the times be achieved by moving it out of the way from airflow paths created by air- conditions. In these paths the temperature fluctuates a lot
	Malfunction oven door	The oven door is controlled with pressure with vales, these can malfunction resulting in a permanent oven door that is open or shut. This is not optimal for measuring. The oven door can also have too much friction (e.g. its bend) or is blocked (e.g. cables)
	Retention times depend on humidity of sample	Especially with Al2O3 columns. Sample must be well dried with Perma Pure dryer
	Bad or difficult type of column	Water sensitive and packed columns are more sensitive than the standard silicone column. Either choose another column or see to it, that carrier gas always stays on the column and that the sample is dry
	Defective oven thermocouple	Check the temperature with another temperature sensor and compare it with the reading

### 5.2.6 Stability of calibration result

Symptom	Possible suspect	Solution
Response is dropping	Lamp failure	If it changes more than 20% within 24 hours occurs, it is possible that the lamp is leaking
	No dust filter on sample inlet	Cleaning systems in such cases may provide an increase of sensitivity of a factor 30% to 90%. Do use inline filters after cleaning!
Response is unstable	Pressure of air or hydrogen has changed	This changes FID response. Reset to original pressures or recalibrate
Repeatability is bad	FID is dirty	Clean FID
Alarm after automatic validation	System was not calibrated	An calibrate needs to be completed before using auto validation
Alarm after automatic validation	Analyser or calibration gas unstable	Redo auto validation one more time after checking calibration gas connection
Response increases over long time	Instable calibration gas or unsuitable pressure reducer	Reproducibility needs to be tested before linearity. But if the concentration has risen over a long period of time the pressure regulator, or diluter is unsuitable
	Dirty sampling lines	Change the tubing, especially if the rise has happened over a long period of time.
System stops after multipoint calibration	No gas connected at stable concentration	Fix calibration gas supply and redo multipoint

### 5.2.7 Detector signal

Symptom	Possible suspect	Solution
Noisy PID signal	Bad connection in the PID	Disassemble the PID and check the inner and outer side of the electrical contacts. Assemble the PID properly and check the screws
Noisy FID signal	Dirty FID	The FID tends to have higher signal to noise ratio. Maintain and clean the FID. If that fails the FID might needs to be replaced
Low PID sensitivity	Dirty window, worn out detector top, or old lamp	Clean the PID, and check if the detector head needs to be exchanged. As a last step the PID lamp can be replaced
Low FID sensitivity	Ratio of air to hydrogen	If the ratio has too much hydrogen in it, the sensitivity will be too low, and either the hydrogen needs to be reduced or the air needs to be increased, But if the total flow is to high the flame would move outside the detector range, and both need to be reduced to bring the flame back to the centre of the detector chamber
Unstable PID sensitivity	Lamp expired	The lamp has a lifetime, and needs to be replaced periodically
Unstable FID sensitivity	Unstable hydrogen or zero air supply	Gas generators might cause a pressure swing, this influances the flow and thus the signal. Seek to improve this pressure stability

### 5.2.8 Linearity and reproducibility of compounds

Symptom	Possible suspect	Solution
Nonlinear multipoint calibration	Machine changes	After major changes in the equipment (new lamp, column, valve or changes of components measured) a new auto linearisation is required
	Out of linear range	There is a set amount of linear range, use non-linear multipoint calibration if a wider range is required
	Absorption effects in tubing	Clean or replace the external tubing. When this does not solve the issue, also clean or replace the internal tubing. The 10-port valve can also be cleaned with diluted ammonia and distilled water. Do not forget to dry everything before use
	Reaction effects in tubing	Reactive hydrocarbons may react with metal and polymer surfaces. With which they react depends on the compound
Bad linearity	Wrong settings	Use the GCX55id.txt and the autolin.dat belonging to the instrument. Linearity of calibration depends on the type of detector cell used. Do not copy these files or settings over from another machine
Response increases over long time	Instable calibration gas or unsuitable pressure reducer	Make sure to test reproducibility before linearity, but if this was the case the pressure regulator or dilutor may be unsuitable
	Dirty sampling line	If when calibration the concentrations rise for a long time. It is time to change the sample tubing
Dropping response	Lamp failure	If more than 20% within 24 hours occurs, it is possible that the lamp is leaking and needs to be checked or replaced
	No dust filter on sample inlet	Try using a cleaning system, this could provide an increase of sensitivity
Unstable response	Change in pressure of air or hydrogen	This changes FID response. Reset to original pressures or recalibrate
Repeatability of FID is bad	FID is dirty	Clean the FID
Response of FID is very low	Flame outside detection range	Reset to the original pressures and restart
Response of PID is very low	Low sensitivity.	Replace PID lamp and/or PID head
Calibration result is low	Leak in sampling system	Check this by blocking the sample entrance with a cap, and checking if the sample strokes cannot be completed. If the piston still moves smoothy there is a leak
	Leak in valve, column system, detector	Check the machine for leaks, including leaks on the electrical PID connections and the PID assembly

#### 5.2.9 Peak integration

Symptom	Possible suspect	Solution
Integration is not covering the area completely	Two peaks are coeluting	The second peak is already starting before first peak is back to baseline. Try using the setting that start the integration with a straight baseline, if the issue persist a different column might be required
No integration	Peak is very broad and not integrated	Change setting for the minimum slope change
Peak is split	Peak is split up into many small peaks	Check if the peak appears at the moment the oven door is opening in that case the peak is split in the column, and the time the ovendoor opens should be adjusted.
Unknown peaks	Ghost peaks from previous cycle	Ghost peaks are compounds that are injected on the analytical column but do not come out within the set cycle time. Make the injection time shorter, then they will no longer reach the analytical column. Or make the peaks come out in the run they are injected on the column system by raising the temperature, raising the flow or making the cycle time longer
Not all peaks are integrated	Long cycle time and/or noisy baseline	The software has run into the limit of number of peaks, Change the minimum peak height to lower this value

#### 5.2.10 Temperature alarm

The pre-concentration and oven are checked in multiple ways. Not only the temperature itself is observed by the machine, it also checks the time it takes to heat up. For safety if any of these are not within the expected range the machine turns them off, and the machine will wait until manual user intervention.

Symptom	Possible suspect	Solution
Temperature error at start up	To low room temperature	Make sure the room temperature is within specifications. The waiting time before this alarm could also be adjusted in the options
Pre-concentration Temperature error	No heating after maintenance Heats up slowly	A connection might be forgotten, check all heating and thermocouple connections Make sure the room temperature is within specifications, and no ventilator or air conditioning is aimed at the machine. These slow down the heating process
	Water in cooled pre-concentration unit	This is rare, but if it has occurred a replacement is recommended
	Heating up too slow	There might be a problem in the power supply or transformer. Check the input voltage, or replace the power unit(s)
Oven temperature error	Too slow, and too high room temperature	The oven heats slower when it is close the setpoint, more time could be added, or a second restart could be tried. In addition make sure the room temperature is within specifications, and lower it if possible. Raising the setpoint might also be an option.
	Heats up slowly Communication	The used heating setting could be optimised in the settings A temperature alarm will also appear if the
	problem	communication is faulty. Check for problems in pc or on MX board
	Damaged oven heating element	If the oven has been overheated, the heating coil will lose its shape and short circuit
	Malfunctioning thermocouple	Check the thermocouple connection, or replace it completely
	Oven door not working	The oven door is opened and closed with a pneumatic piston. If this break, bents, loses pressure, or has too much friction it might stop working. Check if anything is blocking the piston or tubing

## 5.2.11 Pressure and flow alarm

Symptom	Possible suspect	Solution
Pressure alarm	Pressure of carrier gas out of range	Check if the pressure has been set properly and if the supply pressure is withing the given specifications. The diameter and length of the supply tubes could also be influencing things, and it also a good idea to check for leaks
	Pressure sensor malfunctioning	The sensor is probably broken, try replacing it
Flow alarm	Problem in mass flow controller	If the flow swings around, the carrier gas pressure should be increased. If the alarm is triggered by the MFC and is completely blocking the system, contact your distributor or Synspec
	Leak in carrier gas system	Check for leaks in the carrier gas supply

### 5.2.12 Data handling and communication

Symptom	Possible suspect	Solution
No peaks in data file	Peak windows have not been saved	Set the peak windows on a chromatogram, stop and start run to save these settings. These settings can also be backed into a file in the program edit mode. It is recommended to do this every time the peak windows settings are changed
Analyser has integer overflow indication	Wrong integration attenuation setting	The current combination of the settings is responsible for this error. Reset all integration attenuation settings and retry
Integer flow error after auto linearisation	Auto linearisation was not finished successfully	Check peak window setting and stability retention time. Repeat auto linearisation and check for a stable concentration of calibration gas
Periodically (15min) peaks and dips in detector signal	Water in cooled trap	The presence of water in the trap leads to negative dips in the detector value, and high positive ones in systems with a cooled trap. It is recommended to keep within specs and avoid water in the sample, but alternatively a dryer could be used
Alarm after automatic validation	System was not calibrated	Before an auto validation the system must be calibrated
Alarm after automatic validation	Unstable analyser or calibration gas	After checking the calibration gas connections, do the auto validation one more time
Reprocessing is not working	Not all required files are present	All required information needs to be copied, follow the reprocessing steps in the manual
	Old software is used on new data	Although it would be great to live in a magical world, forwards compatibility does not exist. Old software cannot reprocess new date, use the new version instead
	New software is used on old data	Although huge care is taken for it, new software does not have flawless backwards version support. And data generated with older versions cannot always be reprocessed. If required it is advised to keep older versions of the executable, but these can also be obtained from your distributor or Synspec
Missed data and/or blue screen	Computer or power supply malfunctioning	The log file can be checked for automatic restart and its possible trigger. Furthermore observe the system for the possible problem to be fixed or replaced, in partially the system motherboard, drive and processor, Alternatively the power supply can be monitored for unstable voltage
Random irrelevant error messages	Internal communication problem	Unstable and bad communication withing the machine can generate these messages. Check the wire connectors for obvious problems

## 6. Final remarks

### 6.1 Decommissioning

The Synspec analysers series have a lifetime of over 10 years, and a great effort is put into keeping spare parts and support available. If an old analyser is unwanted or underperforming, consider exchanging it for a newer model at a discount.

The analyser should be decommissioned correctly. As the system only contains ROHS compliant parts, it can be delivered to the local electronic recycle- or waste collection centre.

### 6.2 This manual

The manual is available on paper, and one is supplied with each analyser. A digital version is stored on the analyser.

Synspec analysers are constantly being improved and adapted to meet new requirements and opportunities, there might be parts in this manual that are newer or older and might not apply, if any problem(s) occur because of this; Please contact Synspec with a description to the problem and all other relevant information such as the analyser type and ID.

Much care has been put into avoiding mistakes in this manual, but when in doubt contact Synspec. See also our sales conditions on our website for any questions on warranty and responsibility.

#### 6.3 Windows password

The GC will start-up automatically with the standard user account. Standard user account name: **GCX55** Password: **GCX55**.

To make changes to Windows settings or to install additional software a user with administrator rights is needed.

Administrator account name: GCADMIN Password: GCADMIN

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#### INTRODUCTION

The Hessen Bayern or Bayern-Hessen protocol can be used in de Synspec GC software to retrieve concentration of measured values by a datalogger.

The communication van be set over a serial RS232 connection or over an ethernet connection (TCP-IP). The GC software can handle communication over both channels simultaneous. RS232 settings can be made in the GC Software; however the TCP-IP settings are done in the GCxxx\_ID.txt file.

#### SETUP OF THE ETHERNET CONNECTION AND A CHECK WINDOW FOR RS232 OR TCP-IP

The setting of the ethernet connection must be done in the GCxxx\_ID.txt file. Add the following lines: TCPACTIVE = TRUE //datalogger ip address: TCP-IP1 = 192.168.0.144 //localhost ip address: TCP-IP2 = 127.0.0.1 //Dedicated Modbus tcp port: TCP-PORT = 9880 //To check the TCP communication: SHOWTCP = TRUE //To check the serial RS232 communication: RS232SPYWINDOW = TRUE

#### SETUP THE PROTOCOL IN GC SOFTWARE

Start the GC software. When the SHOWTCP = TRUE and RS232SPYWINDOW = TRUE is present in the GCxxx\_ID.txt file, the spy windows will pop-up and can be used to check the communication:

S TCP spy window	- [	⊐ ×	S RS232 spy window	÷ .		×
Own IP 192.168.200.104 F	Port 9880					
TCP Server activated HESSEN/BAYERN protocol active HESSEN/BAYERN protocol active HESSEN/BAYERN protocol active The IP address of the Hessen-Bayern port a shown. Port number of communication over	ddress will be of Hessen Bay	yern	Received 6 bytes at: 194046           Command received : DAU04           Answer : MD03 000 +4558+01 7E 3F 1.           Received 6 bytes at: 194048           Command received : DAU04           Answer : MD03 000 +4558+01 7E 3F 1.           Received 6 bytes at: 194050           Command received : DAU04           Answer : MD03 000 +4558+01 7E 3F 1.           Received 6 bytes at: 194052           Command received : DAU04           Answer : MD03 000 +4558+01 7E 3F 1.           Received 6 bytes at: 194054           Command received : DAU04           Answer : MD03 000 +4558+01 7E 3F 1.           Received 6 bytes at: 194054           Command received : DAU04           Answer : MD03 000 +4558+01 7E 3F 1.           Received 6 bytes at: 194058           Command received : DAU04           Answer : MD03 000 +4558+01 7E 3F 1.           Received 6 bytes at: 194058           Command received : DAU04           Answer : MD03 000 +4558+01 7E 3F 1.           Received 6 bytes at: 194054           Command received : DAU04           Answer : MD03 000 +4558+01 7E 3F 1.           Received 6 bytes at: 194050           Command received : DAU04           Answer : MD03 000 +4558+01 7E 3F 1.           Received 6 bytes at: 194100	23 000000 001 +5 23 000000 001 +5	075+01 7E 075+01 7E 075+01 7E 075+01 7E 075+01 7E 075+01 7E	2 3F 2 3F 2 3F 2 3F 2 3F 2 3F
Close Clear	Save log		Close Clea	save I	og	

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#### DATA COMMUNICATION HESSEN-BAYERN PROTOCOL

# SYNSPEC

To set the Hessen-Bayern protocol in the GC Software: Program Edit, Settings, Datalogger communication:

Datalogger Einstellung	gen						
Protokoll	seriell-	TCP:9880-	1				
Hessen/Bayern	$\odot$	•					
Gesytec II	0	0					
ASCII-terminal	0	0					
MODBUS	0	0					
Adresse:	1						
Aktuelle Port-Einstellung∉ Aktiv COM1: 1200 Baud, 7 Bit, Even Parity, 1 Stopbit.							
COM1. 1200 Da	uu, 7 Dit, 1		эторыт.				
Port Auswaehlen		COM1 (n.a.)					
Baudrate		1200	•				
Laufstatusbits de	efinieren	Fehlerstatus	bits definieren				

Set the protocol to Hessen/Bayern.

For the serial connection: the serial port must be set to Active.

The settings for the com port is automatically set to 1200, 7 bit, Even Parity and 1 stopbit.

However 9600 Baud can be used too; just be sure that the same Baud rate is set in both the GC software and the datalogger software.

When the serial port is selected, for example COM1, the GC software will try to use this port. If this succeeds, the port will be locked by the GC software and this is shown as COM1 (n.a.).

Select the desired Run status bits and Error status bits in the GC Software:

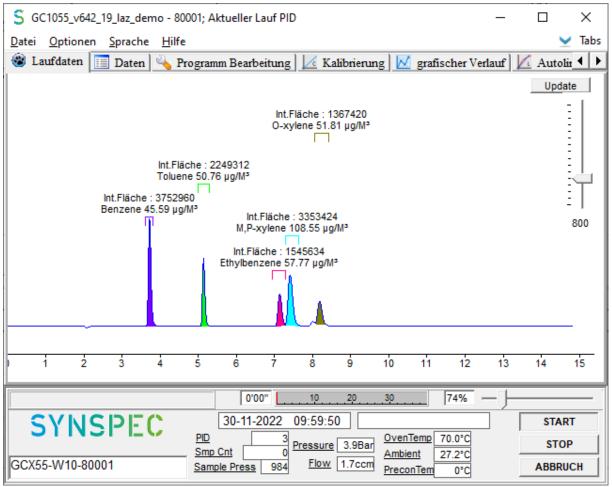
Datalogger Laufstatus-bits	Datalogger Fehlerstatus-bits				
NICHT	NICHT				
BIT 1 🗌 Steuerdruck zu niedrig	BIT 9 🗆 Lauf aktiv 💌				
BIT 2  Lauf aktiv Kalibrier Modus	BIT 10 🗆 Lauf aktiv 💌				
BIT 3  Prüfgasventil geöffnet Nullgasventil geöffnet	BIT 11 🗆 Lauf aktiv 💌				
BIT 4	BIT 12 🗆 Lauf aktiv				
BIT 5  Kalibrierdaten am Ausgang Start neuer Lauf v	BIT 13 🗆 Lauf aktiv				
BIT 6 🗆 Lauf aktiv	BIT 14 🗆 Lauf aktiv				
BIT 7 🗆 Lauf aktiv	BIT 15  Probenahme Fehler				
BIT 8 🗌 Steuerdruck zu hoch 💌	BIT 16 🗆 Fluß zu niedrig 🔽				
✓ <u>O</u> K X Cancel	✓ <u>O</u> K X Cancel Ø <u>H</u> elp				

Now, the GC will respond to serial or ethernet requests using the Hessen-Bayern protocol.

#### TESTING THE HESSEN-BAYERN COMMUNICATION WITH EASYCOM DATALOGGER

To test the Hessen-Bayern communication, send a command to the GC from the datalogger-PC. In this example, the datalogger program Easycom is used.

#### A chromatogram of the GC software:



Of the first 3 peaks, the concentration values are retrieved in Easycom datalogger software. This is the default setting in GC Software for the Hessen Bayern protocol. If more peaks must be callable, the default amount of peaks can be increased using the following line in the GC1055\_ID.txt file: for example HBMAXCOMP = 5.

EasyComp Demo Version 20011118 09:41:59 Station LASO							_			
Password	Parameter	Display Cali	ibration Gra	aphic Singl	e Measuremer	nt Language				
Name	Units	Data	Data	Data	OStat	EStat	Validity	Average	% Valid	Average
		current	corrected	checked	current	current	current	5s	5 s	1 Min
Ben	µg/m³	45,6	45,6	45,6	10000000	00000000	Valid	45,6	100	45,6
Tol	µg/m³	50,8	50,8	50,8	10000000	00000000	Valid	50,8	100	50,8
EBen	µg/m³	57,8	57,8	57,8	00000000	00000000	Valid	57,8	100	57,8
mXyl	µg/m³	0,0	0,0	0,0	00000000	00000000	Invalid	0,0	0	0,0
oXyl	µg/m³	0,0	0,0	0,0	00000000	00000000	Invalid	0,0	0	0,0

The concentration of the first 3 peaks are available in the Easycom datalogger software.

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