

Technical Report:

"BTEX ambient air analysis: sources, regulations, technologies for controls"



In collaboration with The Council for Research and Experimentation in Agriculture at the Puglia Regional Environmental Protection Agency (ARPA)



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1. BTEX ANALYSIS

1.1 Sources of BTEX pollution and health impacts

Benzene is a hydrocarbon that appears as a volatile liquid, capable of evaporating rapidly at room temperature; it is colourless and easily flammable and is the progenitor of a family of organic compounds defined as aromatic, due to the characteristic odour. Benzene is a natural component of oil (1-5% by volume) and its refining derivatives.

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In the atmosphere the most significant source of benzene is represented by vehicular traffic, mainly from the exhaust gases of petrol-fuelled vehicles, in which benzene is added to the fuel as an anti-knock (max 1%), mixed with other hydrocarbons (toluene, xylene, etc.) in place of the tetraethyl lead used up to a few years ago. In part, the dispersed benzene comes from the emissions that occur in the petrol refining, storage, and distribution cycles.

During the refuelling of vehicles, significant quantities of the toxic compound are released into the air, with exposure to the risk of personnel fuelling the vehicles.

In the industry during the past, benzene found widespread use as a solvent, especially at industrial and artisanal level (production of footwear, rotogravure printing, etc.), until the proof of its toxicity and its ability to induce tumours led to a law that drastically limits its concentration in solvents.

For the same reason, the use in open industrial cycles and in the production of consumer products (plastics, resins, detergents, pesticides, drugs, paints, adhesives, inks and adhesives) has been strongly limited and is regulated by precise regulations of the European Union.

In finished products, benzene is found in very limited quantities, also regulated by law, currently it is used mainly as a raw material for the chemical synthesis of organic compounds such as phenol, cyclohexane, styrene, and rubber in closed-cycle processing.

In part its formation is to be attributed to natural causes such as forest fires, agricultural residues, or volcanic eruptions.

Benzene is easily absorbed by the human body almost exclusively by inhalation, while penetration through skin contact is negligible. It accumulates in tissues rich in fat (adipose tissue, bone marrow, blood and liver), where it is metabolised and then quickly eliminated in urine and exhaled air.

The carcinogenic capacity of benzene has been scientifically demonstrated and has been classified by the IARC (International Agency for Research on Cancer) in class 1 as a certain carcinogen for humans. In fact, its ability to cause acute and chronic leukaemia has been ascertained, at concentrations present in the past in work environments, with a risk proportional to the cumulative dose. The carcinogenic effect appears to be linked, as with other substances, to the action of intermediate metabolites that form in the body.

At present concentrations of benzene in an urban environment, no toxic effects on blood cells have been observed.

During the risk assessment not only the concentration of benzene in the atmosphere should be considered, in consideration of the limited time of outdoor exposure, but above all the exposure in confined spaces (indoor pollution) and the introduction of the substance through ingested foods.

Exposure is subject to significant variations in relation to the seasons, outdoor physical activity, residences near busy roads or benzene point sources, but above all to both active and passive cigarette smoke.

1.2 UNI EN 14662:2015, parts 1, 2, and 3

"Air quality, standardized method for measuring C₆H₆ concentration"

The European standard EN14662:2015 specifies a semi-continuous measurement method for determining the concentration of benzene present in ambient air based on automatic sampling and analysis by gas chromatography.

The method describes the desired performance characteristics and establishes the minimum criteria required to select an appropriate automatic gas chromatograph (GC), by analysing the homologation tests. It also includes the assessment of the suitability of an analyser to be used in a given site in order to meet the data quality requirements as specified in Appendix I of Directive 20018/50/EC and the requirements during sampling, calibration, and the guarantee of quality of use.

The method is applicable to the determination of the mass concentration of benzene present in ambient air up to 50 µg/m³ (15.4 ppb) of benzene. This value represents the concentration level used for the approval test. Other concentration levels can be used, depending on the concentrations present in ambient air.

Section 7 of the standard describes the main components that a gas chromatograph must have to determine for the on field determination of benzene:

Sampling trap

A typical sampling trap is made of stainless steel or borosilicate glass. It is packaged with a sorbent or series of sorbents capable of quantitatively retaining benzene in a minimum volume of sampled air, necessary to measure benzene concentrations equal to or less than 10% of the annual limit.



Sampling device

The sampling device used may vary from instrument to instrument; in any case, it must be able to provide a known volume sample at standard temperature and pressure conditions.

Thermal desorption unit

Depending on the instrument used, the thermal desorption unit may be available at one or double benzene desorption stages from the sampling trap.

Desorption is performed by heating the trap within a short period of time while passing a gas carrier flow (typically nitrogen). Temperature, gas flow, and time are all elements that must be sufficient to quantitatively transfer the trapped benzene into a secondary trap, a pre-column or into the analytical column, depending on the instrument.

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Separation unit

The separation unit consists of an analytical column (separation) and an oven used for heating the column to separate the benzene from (potential) interferers, within a period sufficient to allow quantification at concentrations equal to or less than 10 % of annual limit value.

Detector

The detector allows the quantification of benzene at concentrations equal to or less than 10% of the annual limit value. The most commonly used detectors are flame ionization (FID) and photo-ionization (PID) detectors.

Data processing system

The analyser will be equipped with a software that allows the identification and quantification of benzene in the acquired chromatograms.

The software must allow data to be reprocessed after the acquisition. Data processing and reprocessing can be performed on field using an internal computer or external analyser.

In addition, Annex C of standard EN14662:2015 contains a summary table that indicates the main components present in the typical Benzene field analysers.

Table C.1 — Typical components of benzene analysers

Monitor	1	2	3	4	5	6
Cycle time min	15	15-30	15-30	15-30	15	60
Sorbents	Carbotrap™/ Carbosieves™ SIII	Tenax™ GR	Carbotrap™/ Carbopack™	Porapak™ SuperQ™	Tenax GR™	Carbotrap™/ Carbosieves™ SIII
Sampling	pump/nozzle	pump/MFC	internal pump	pump/MFC	piston pump	Pump / MFC
Bypass	possible	200 ml		possible	pump	no
Temperature °C	ambient	40	10-35	ambient	ambient	-30
Sample amount	500 ml	300 ml	up to 900/1 800 ml	390 ml	100 ml	800 ml
Carrier gas	H ₂	N ₂	N ₂ or H ₂	N ₂ or H ₂	N ₂	He
Desorption:						
Temp. °C	330	230	340	140	180	320
Time	60 s			2,5 min	30 s	60 s
Flow	2 ml/min	11 ml/min	0,5 ml/min			15 ml/min
Injection	350 °C					
Cryo trap	Carbopack™ B					
Stripper column		5 m		CP Sil 5CB. 12 m. 0,53 mm	1,8 m	
Analytical column	1 µm BGB-2.5 (2,5% Phenyl)	Ultimetall 1 µm CP-Wax52CB	EPA 624 type	CP Wax	1,2 µm AT-5	2 columns: Al ₂ O ₃ - Na ₂ SO ₄ (C2-C5), BP1 (C6+)
- length	9 m	45 m	10 m	25 m	13 m	60m
- diameter	0,2 mm	0,53 mm	0,22 mm	0,53 mm	0,53 mm	0,22mm
Analytical conditions °C	35-180	80	45-140	80	45-60	48-200
Detector	FID	FID	PID or FID	FID	PID 10,6 eV	FID

Table 1 - Components present in Benzene field analysers

2. INSTRUMENTAL COMPARISON: PyxisGC BTEX– CHROMATOTEC GC 866 ANALYZER PUGLIA ARPA CONTROL UNIT

2.1 PyxisGC BTEX Analyser

The PyxisGC BTEX instrument monitors in real time the volatile organic compounds such as benzene, toluene, ethyl benzene and xylene. The analysis cycle used lasts 15 minutes and the operation of the instrument can be divided into three different phases, which take place in parallel during the entire cycle:

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Figure 1 - Pre-concentrator and MEMS chromatographic gas column

Pre-concentration: the selective concentration of the reference compounds occurs through a silicon/glass MEMS device suitably filled with an adsorbent material. This phase lasts 10 minutes and takes place at temperatures below 50°C. A flow meter constantly samples the same volume of gas. Before the injection phase, the device heats up quickly above 100°C and the trapped material is desorbed and injected into the separation module

Gas chromatographic separation: the compounds desorbed during the injection phase arrive at the gas chromatographic column, also based on MEMS technology. Using ambient air filtered as gas carrier, the column separates the various compounds that elute with different times towards the final detection module.

Detection: the photo ionization detector (PID) quantifies the compounds leaving the column, based on the different retention times, creating the classic chromatographic graph. At the exit of the detector another flow meter is installed to monitor the correct functioning of the system.

PyxisGC BTEX is equipped with an internal memory, for data acquisition and chromatograms. Also, PyxisGC BTEX has a **Cloud Software (Pollution Guardian)** for the real-time monitoring and management of data acquired. Moreover, Guardian allows to remotely use the instrument.



2.2 Chromatotec GC 866

The analysis cycle of the Chromatotec GC 866 analyser is divided into:

Pre-concentration: The gas sample is taken from an external pump and passes through a trap, a thin tube containing porous substances, which holds the gas components based on their affinity with these phases; for example, permanent gases and water vapour are not retained.

Gas chromatographic separation: The trap is heated to desorb the compounds, the thermal desorption is fixed at 380°C for 60 s, the gaseous sample is subsequently introduced into the analytical column by the gas carrier. The column is located in a furnace at a programmed temperature, with a gradient that starts with the desorption of the trap (injection phase).

Detection: At the end of the column, a photo ionization detector (PID) generates an electrical signal proportional to the concentration of the sample components as they elute from the column. This electrical signal is digitized to be transferred to the CPU where the microprocessor transfers the data (integration, mass or concentration calculation, peak identification).

All files, such as the analysis results, the chromatograms and the integration reports can be transferred to the computer using an RS-232 output; the digitized signal is also available as an analogue output (0-1 V).

Quality control procedure for the BTEX parameter applied by Puglia Arpa:

For the identification of the BTEX, the analyser is subjected, automatically, to the control of the calibration drift (zero-span and span-check) by routine which, once every 24 hours, generates, through a permeation tube, a sample with a known concentration (44.7 µg/min of Benzene).

If the check is successful, no alarm is generated; otherwise an alarm is generated with consequent in-situ verification of instrumental performance and calibration through a certified gas cylinder. In any case, this check is carried out every 3 months to preventively control drifts and instrumental calibrations; everything is recorded on the server and in the cabin register.

2.3 Procedure for carrying out the tests

Test activities were carried out in the Puglia Arpa Air Quality Cabin located in Taranto, Via Alto Adige; and is classified as a traffic station.

*“**Traffic station:** station located in such a position that the pollution level is mainly influenced by emissions from neighbouring roads (Decision 2001/752/EC). Therefore, the sampling point is representative of the pollution levels mainly determined by traffic emissions from neighbouring roads, with medium-high traffic flows. These stations are located in areas with significant concentration gradients. (Ref. Guidelines for the preparation of air quality monitoring networks in Italy).”*

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The PyxisGC BTEX installation in the cabin was carried out in the presence of specialised technicians of The Council for Research and Experimentation in Agriculture at the Puglia ARPA, and the instruments were calibrated before installation.

- **PyxisGC BTEX Calibration:** calibrated at the Pollution Analytical Equipment laboratories with a certified cylinder containing BTEX at 10 ppm (for each component), connected to a dedicated dilution system to arrive at a concentration of 5.00 ppb (15.9 $\mu\text{g}/\text{Nm}^3$) of Benzene, 5.00 ppb (18.8 $\mu\text{g}/\text{Nm}^3$) of Toluene, 5.00 ppb (21.7 $\mu\text{g}/\text{Nm}^3$) of Ethylbenzene and 5 ppb (21.7 $\mu\text{g}/\text{Nm}^3$) of m,p,o-Xylenes. The calibration was performed on 02 October 2018.

No further calibrations or checks were performed during the entire test period.

- **Chromatotec GC866 calibration:** calibration carried out in the cabin with certified cylinder at a concentration of 5.00 ppb (15.9 $\mu\text{g}/\text{Nm}^3$) of Benzene, 10.00 ppb (37.6 $\mu\text{g}/\text{Nm}^3$) of Toluene, 5.00 ppb (21.7 $\mu\text{g}/\text{Nm}^3$) of Ethylbenzene, 4.80 ppb (20.8 $\mu\text{g}/\text{Nm}^3$) of Xylene-m, 4.90 ppb (21.3 $\mu\text{g}/\text{Nm}^3$) of Xylene-o, 4.80 ppb (20.8 $\mu\text{g}/\text{Nm}^3$) of Xylene-p, remainder Nitrogen. The calibration was performed on 30 January 2018. During the test period the instrument carried out a zero and span self-check through a permeation tube as per the Puglia Arpa qualitative procedure.

In order to evaluate the instrumental performance of the PyxisGC BTEX, both in terms of data quality and long-term performance quality, the test was structured according to the following steps:

a) Parallel measurement of certified cylinder gas before and after monitoring.

At the beginning and the end of the measurement campaign, a cylinder with a concentration of 5.00 ppb (15.9 $\mu\text{g}/\text{Nm}^3$) of Benzene was connected to the sample inlet of both instruments.

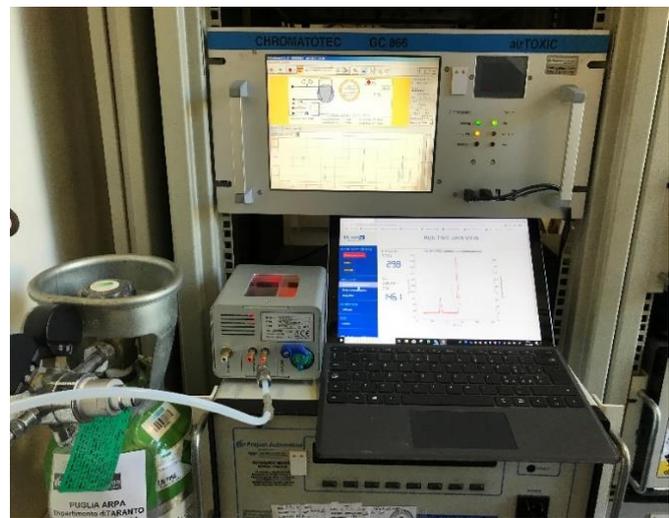
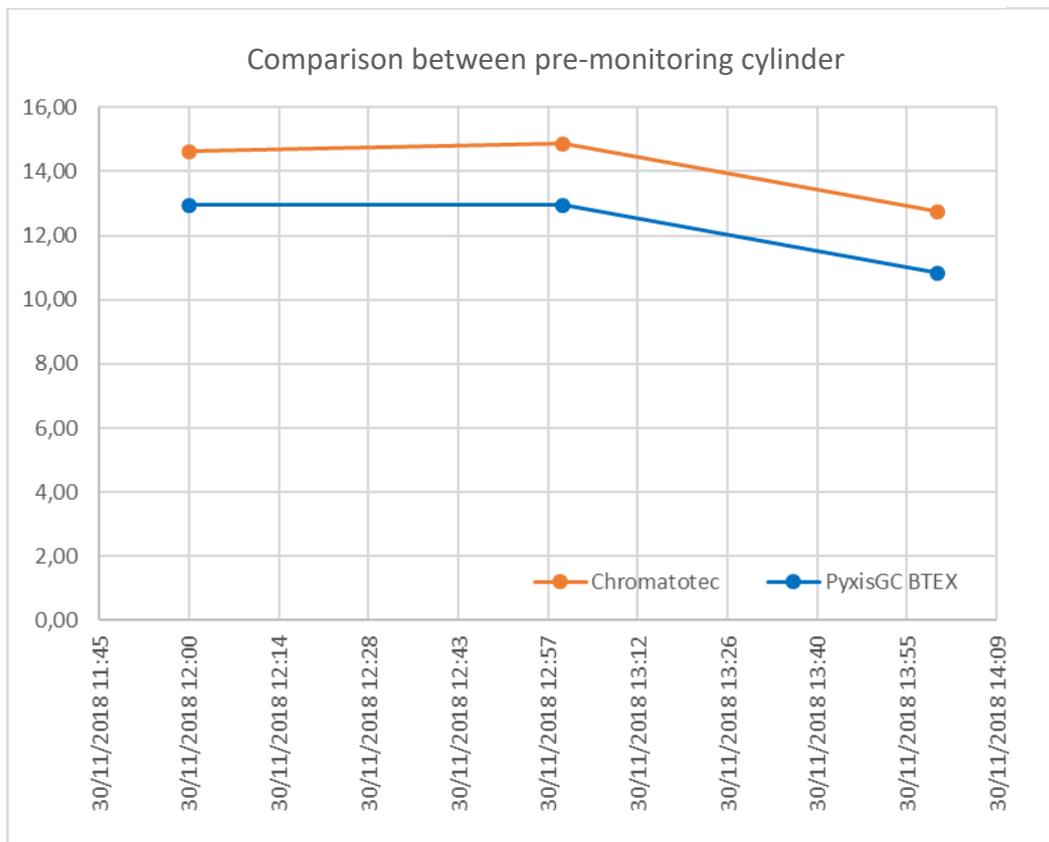
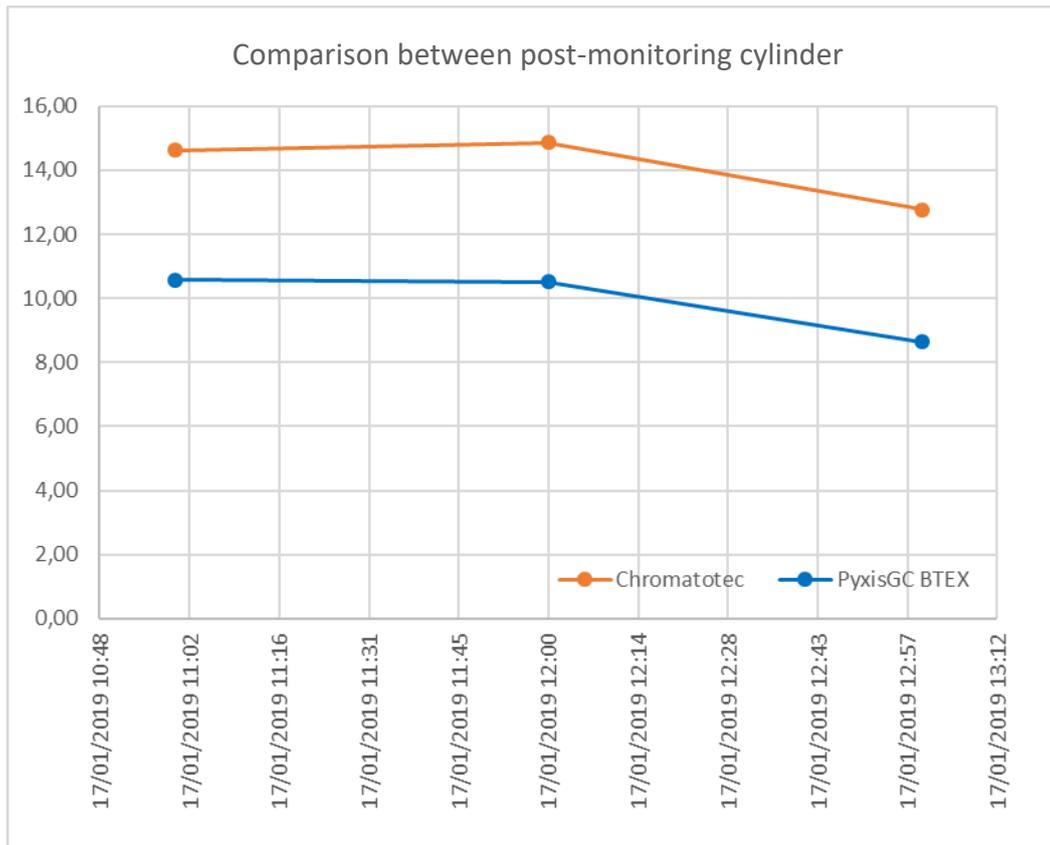


Figure 2 - Installation of the PyxisGC BTEX in the cabin



Graph 1 - Parallel measurement of benzene from certified 7-week pre-monitoring cylinder (concentrations expressed in $\mu\text{g}/\text{Nm}^3$)



Graph 2 - Parallel measurement of benzene from certified 7-week post-monitoring cylinder (concentrations expressed in $\mu\text{g}/\text{Nm}^3$)

- b) Monitoring in parallel with PyxisGC BTEX and Chromatotec GC 866, both installed inside the Puglia Arpa air quality cabin.
Duration of monitoring: 7 weeks.



Figure 3 - Air Quality Station - via Alto Adige, Taranto, Italy

2.4 Results obtained

At the end of the parallel measurement and monitoring operations, the data related to the Benzene analysed by both instruments installed in the ARPA Puglia C.R.A. control unit was compared. The data coming from the Chromatotech instrument was validated by Arpa and then compared with those extracted from the PyxisGC BTEX analyser to assess its equivalence.

For the assessment of instrumental drift over the long term, no self-calibration system was voluntarily applied to the PyxisGC BTEX, in order to assess the extent of the instrumental drift and subsequently identify the best technological solution for correcting this factor.

To cancel the effect of the different initial calibration of the two instruments, it was decided to proceed with the insertion of a response factor of the PyxisGC with respect to the certified instrument, calculated on the average deviation between the two instruments of all the measurements of the first week. This method is the same that the laboratories of the European Joint Research Center (JRC-Ispra VA) have used for previous Pollution technology inter-comparisons as can be seen from their Test Report within the ENV56 KEY-VOCs - Metrology for VOC indicators in air pollution and climate change project (report available on request).

The indices used to make the comparison between the instrumental results are:

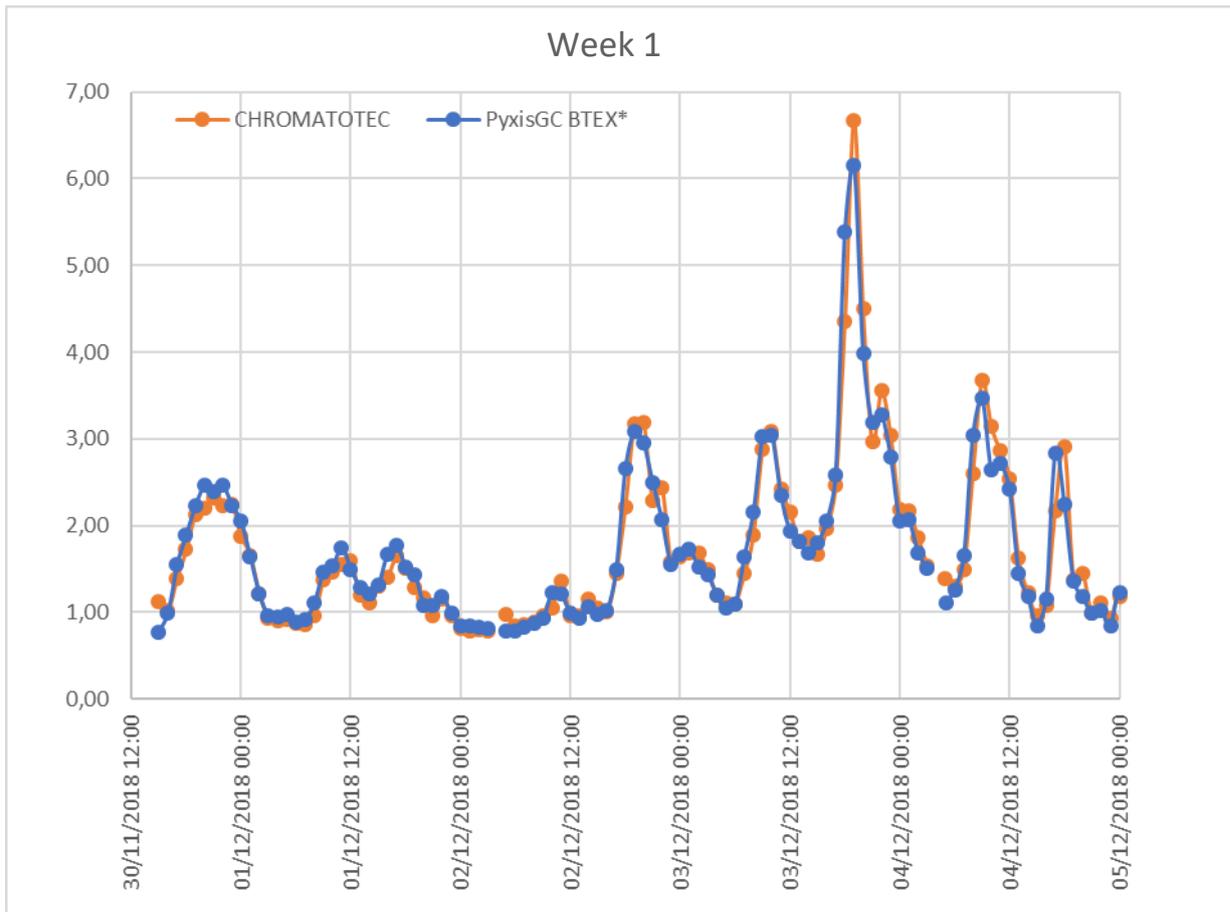
- **R² (linear regression)**: index that measures the linear relationship between two quantitative characters, plotting the punctual measurements of the two instruments.

- **EQUIVALENCE** = $1 - \frac{|y-x|}{x}$

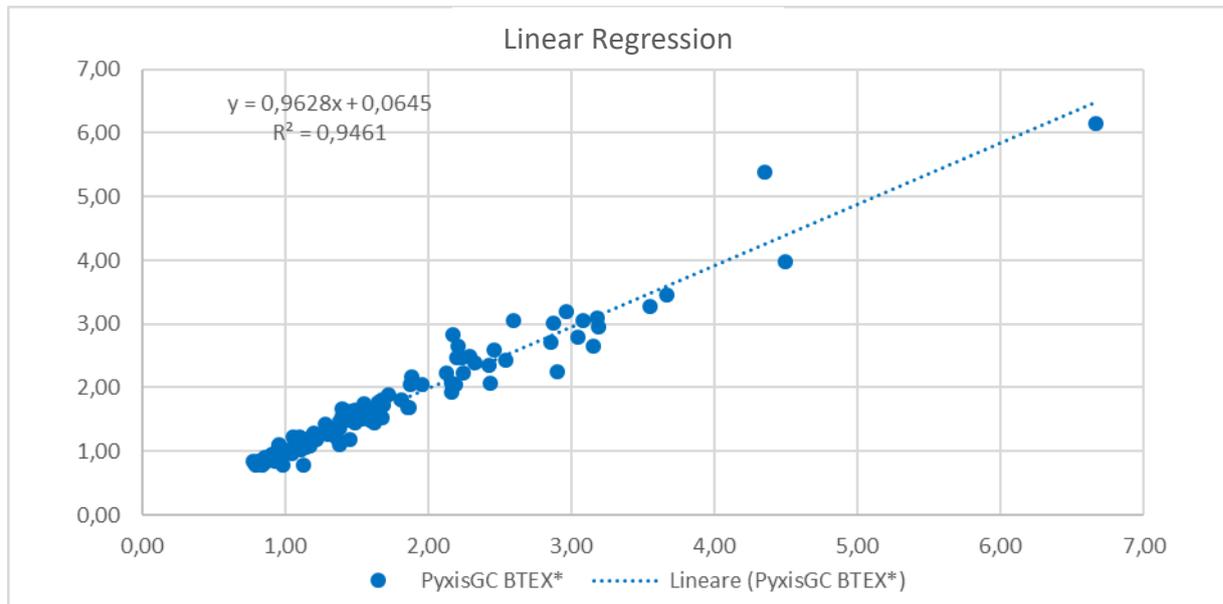
where x= Arpa (Chromatotec) measurement; y= PyxisGC BTEX measurement.

This value was obtained to make the overlapping of the measurements carried out on both instruments easy to interpret.

WEEK 1



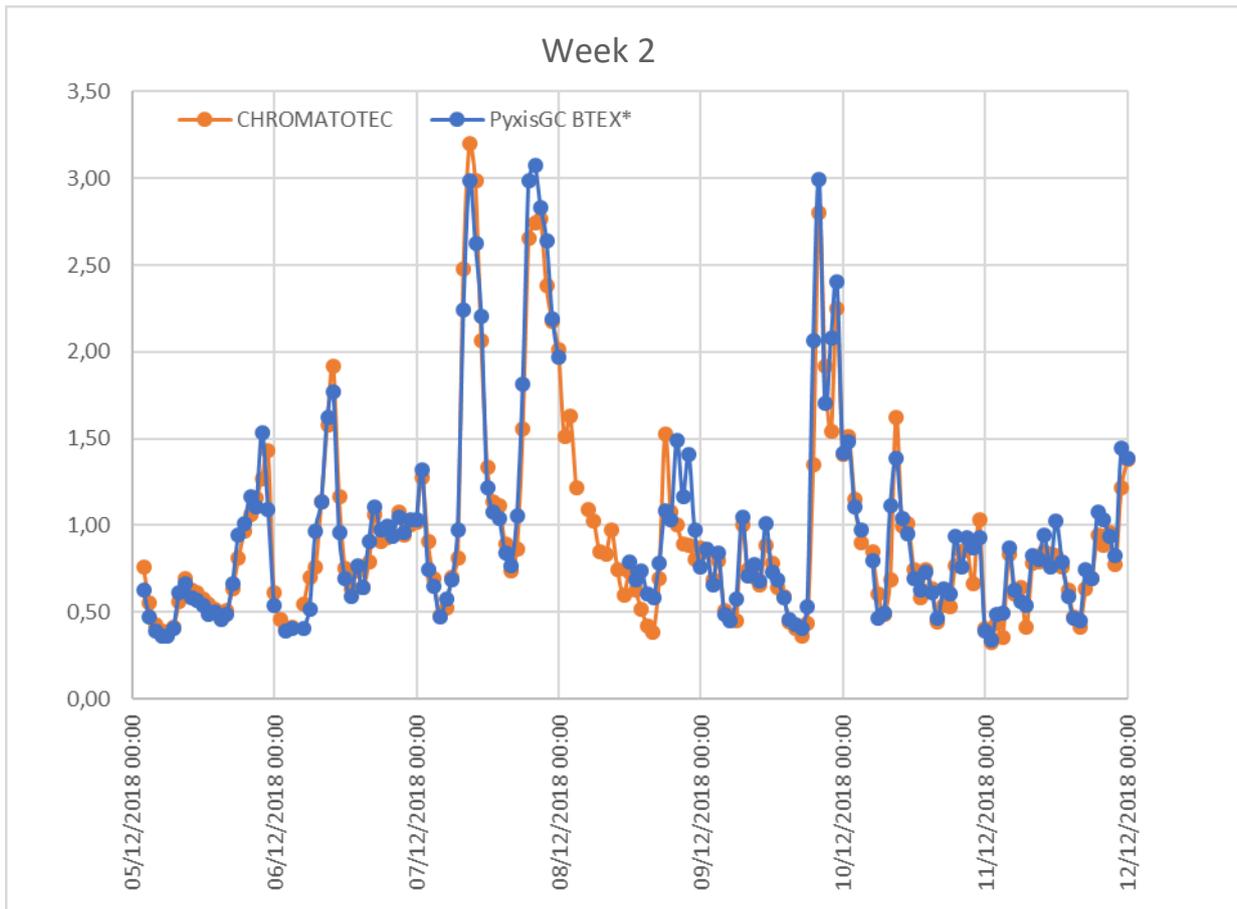
Graph 3 - Hourly average Benzene trend (concentrations expressed in ug/m³) Week 1



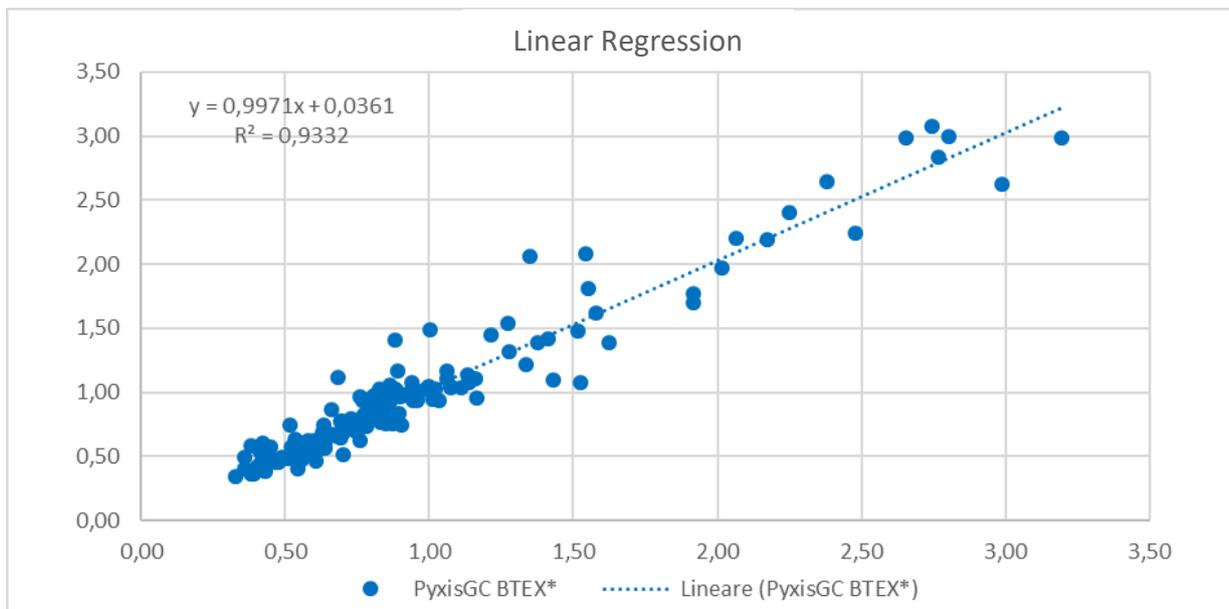
Graph 4 - Calculation of linear regression between instrumental measurements (Benzene concentrations expressed in ug/m³) Week 1

EQUIVALENCE: 92% ---- R²: 0.9461

WEEK 2



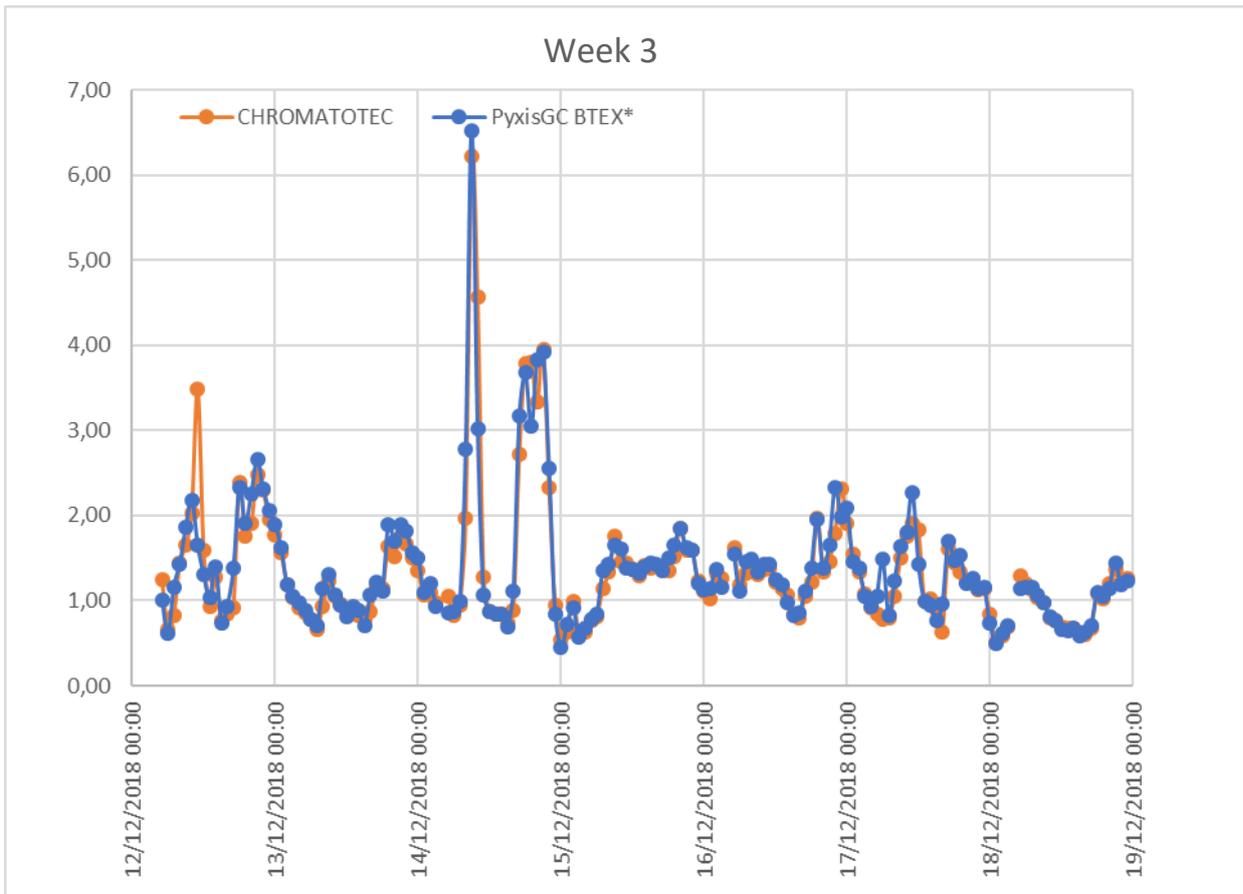
Graph 5 - Hourly average Benzene trend (concentrations expressed in ug / m³) Week 2



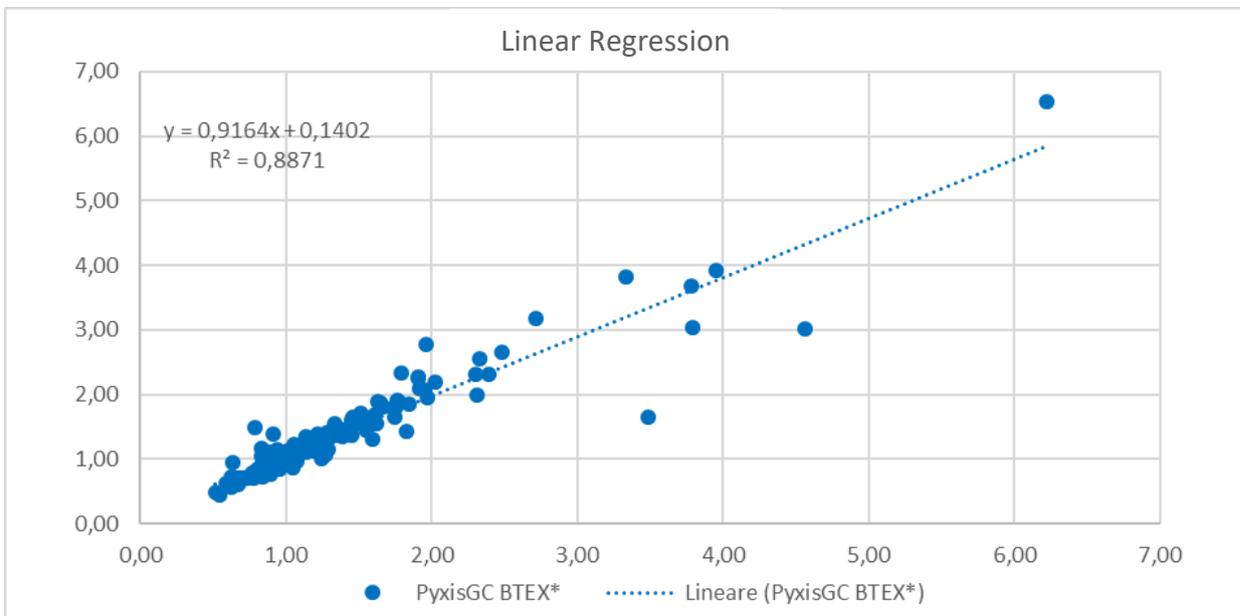
Graph 6 - Calculation of linear regression between instrumental measurements (Benzene concentrations expressed in ug/m³) Week 2

EQUIVALENCE: 89% ---- R²: 0.9332

WEEK 3



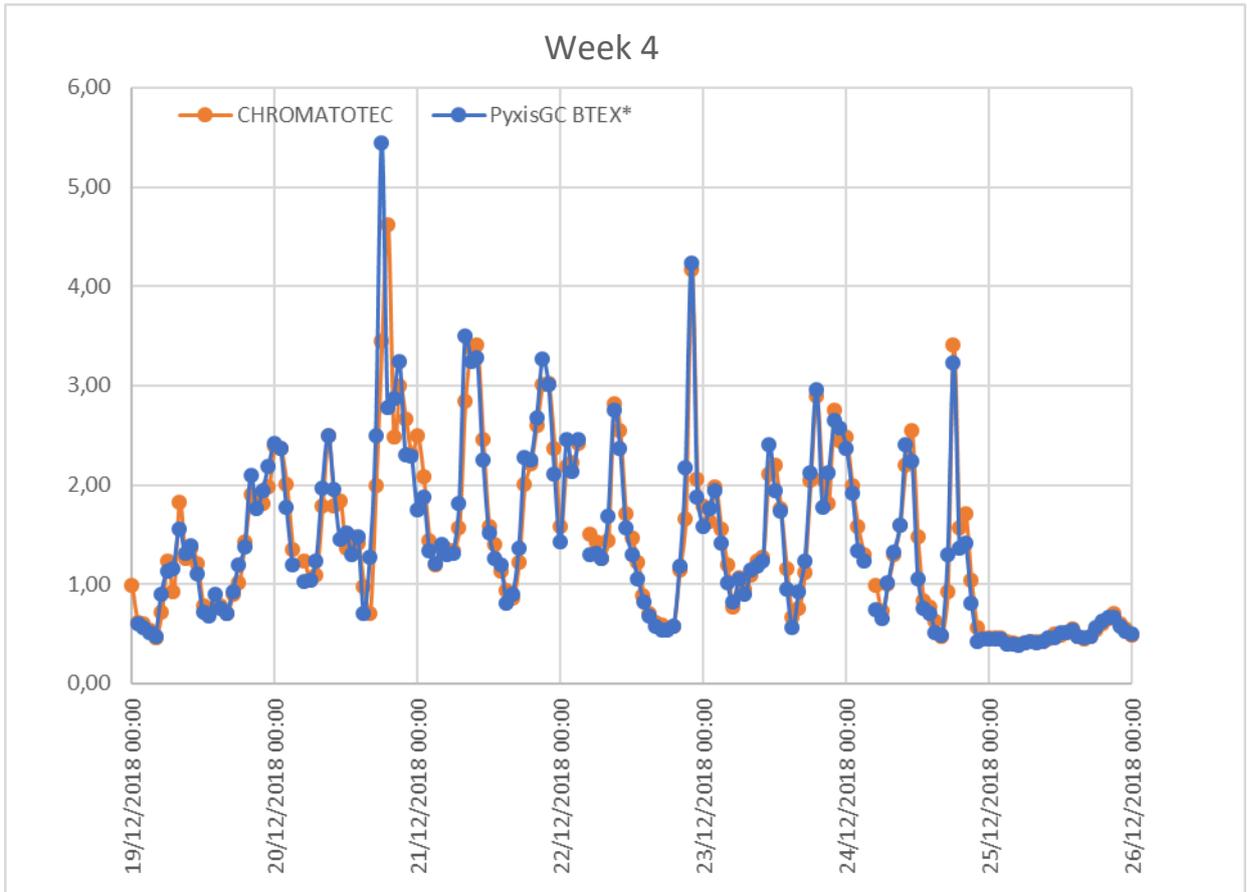
Graph 7 - Hourly average Benzene trend (concentrations expressed in ug/m³) Week 3



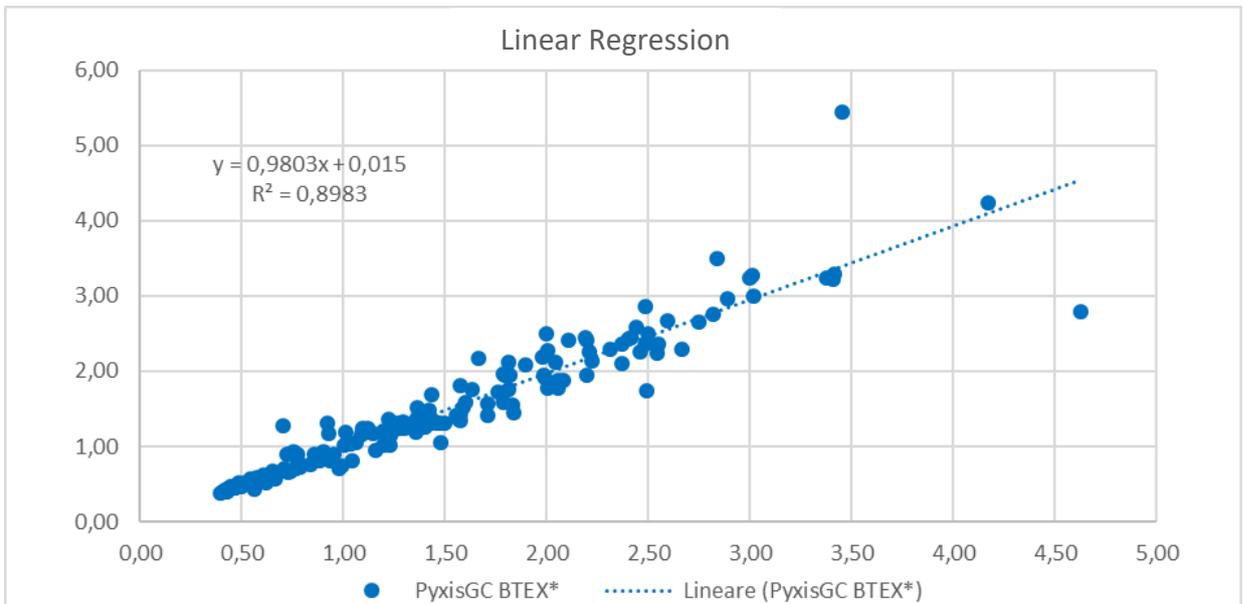
Graph 8 - Calculation of linear regression between instrumental measurements (Benzene concentrations expressed in ug/m³) Week 3

EQUIVALENCE: 91% ---- R²: 0.8871

WEEK 4



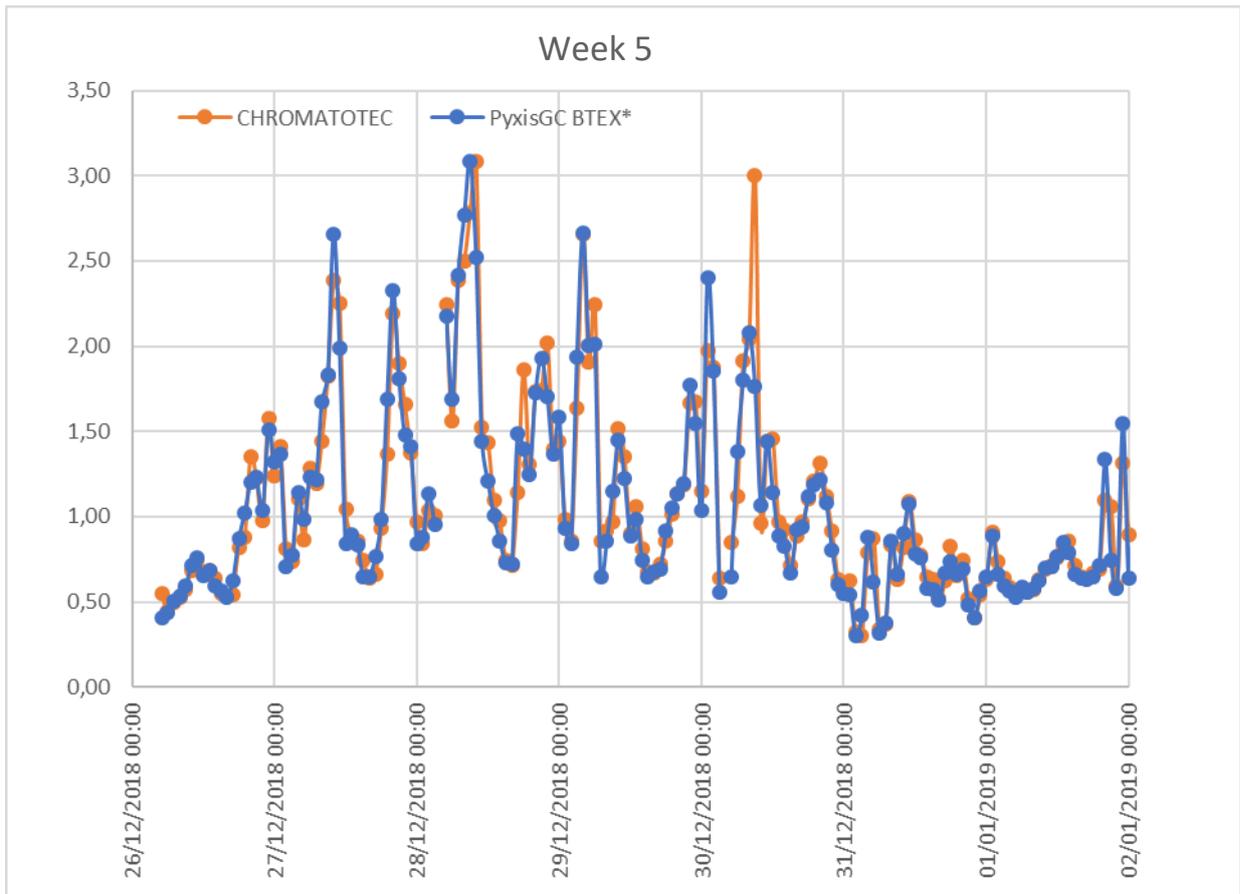
Graph 9 - Hourly average Benzene trend (concentrations expressed in ug/m³) Week 4



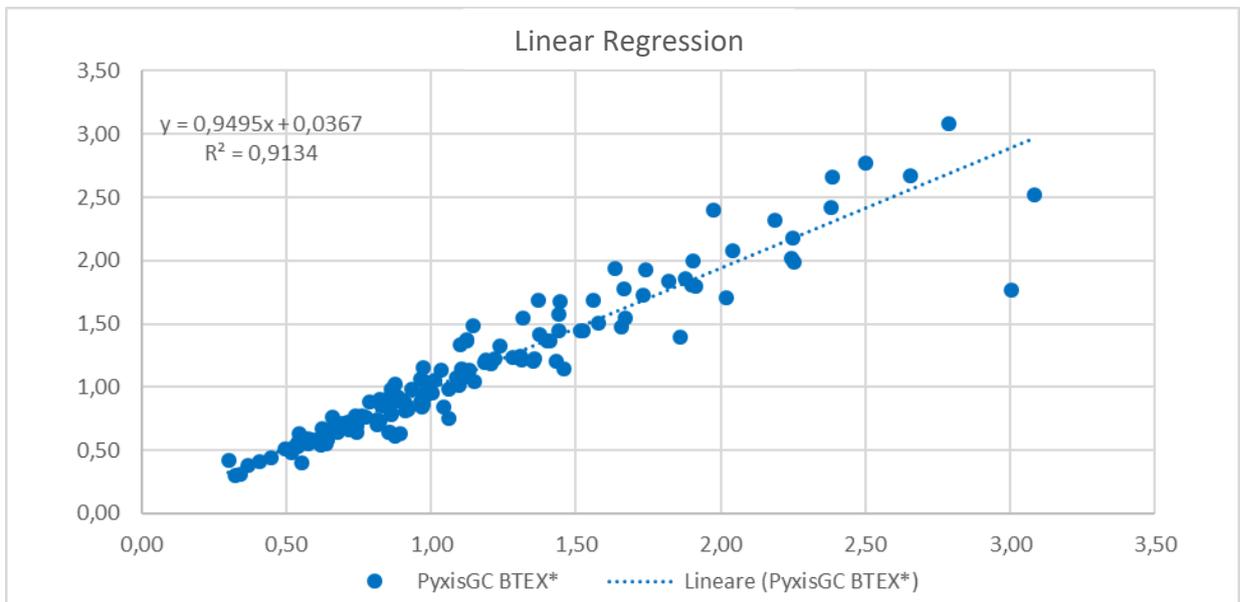
Graph 10 - Calculation of linear regression between instrumental measurements (Benzene concentrations expressed in ug/m³) Week 4

EQUIVALENCE: 91% ---- R²: 0.8983

WEEK 5



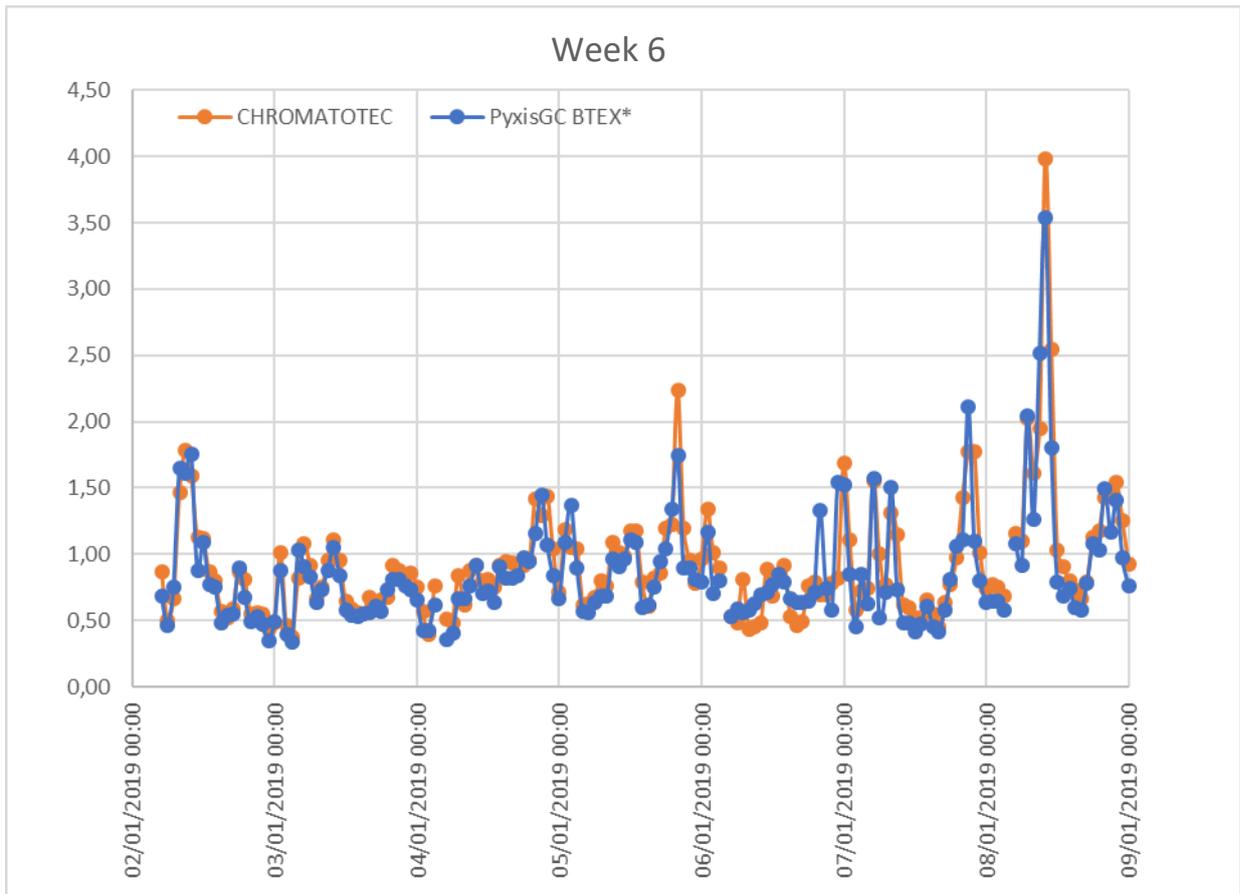
Graph 12 - Hourly average Benzene trend (concentrations expressed in ug/m³) Week 5



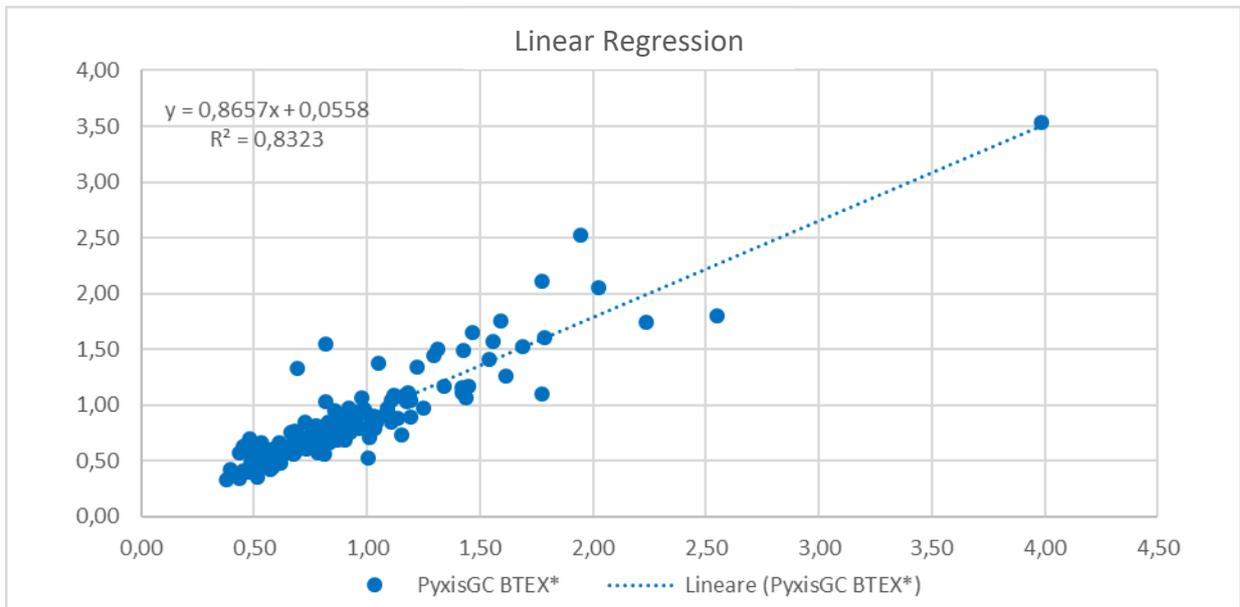
Graph 11 - Calculation of linear regression between instrumental measurements (Benzene concentrations expressed in ug/m³) Week 5

EQUIVALENCE: 92% ---- R²: 0.9134

WEEK 6



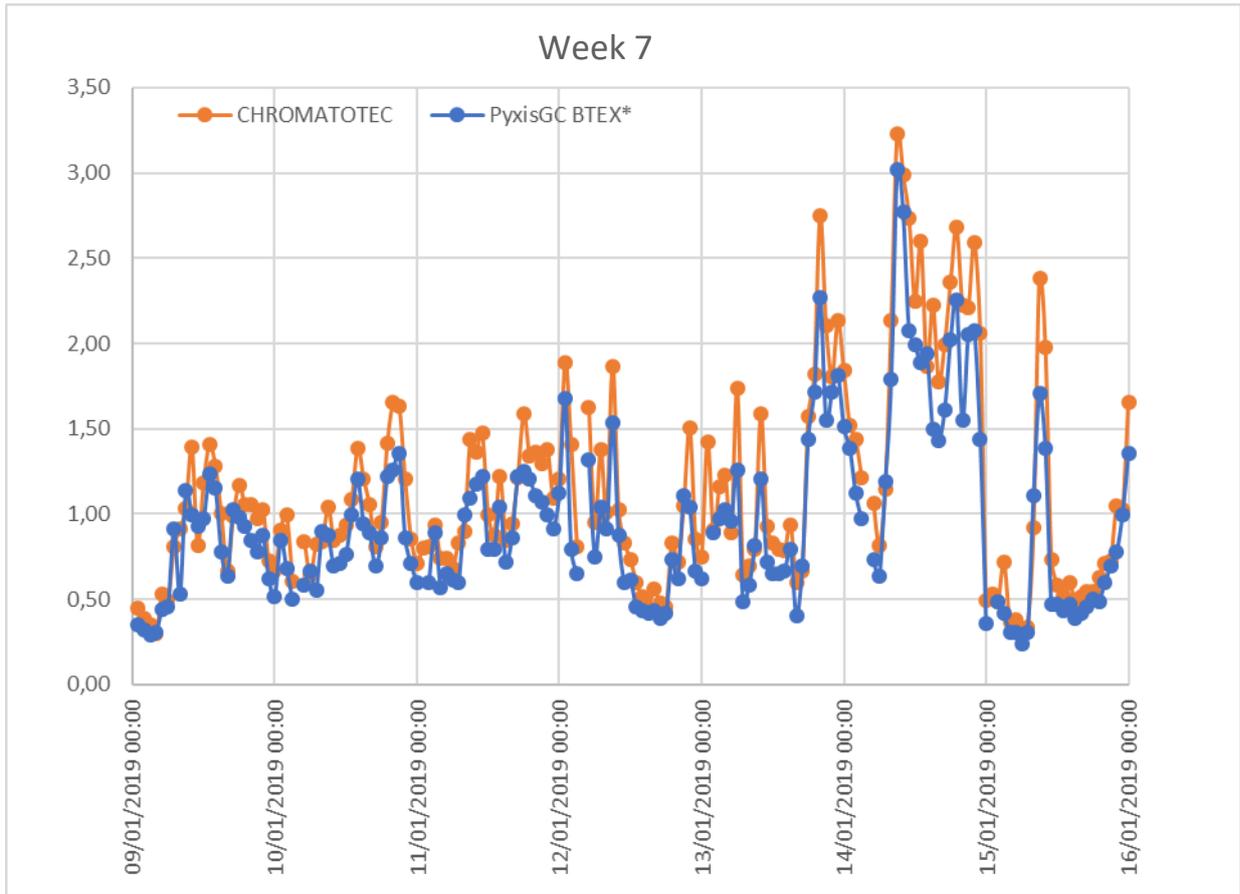
Graph 13 - Hourly average Benzene trend (concentrations expressed in ug/m³) Week 6



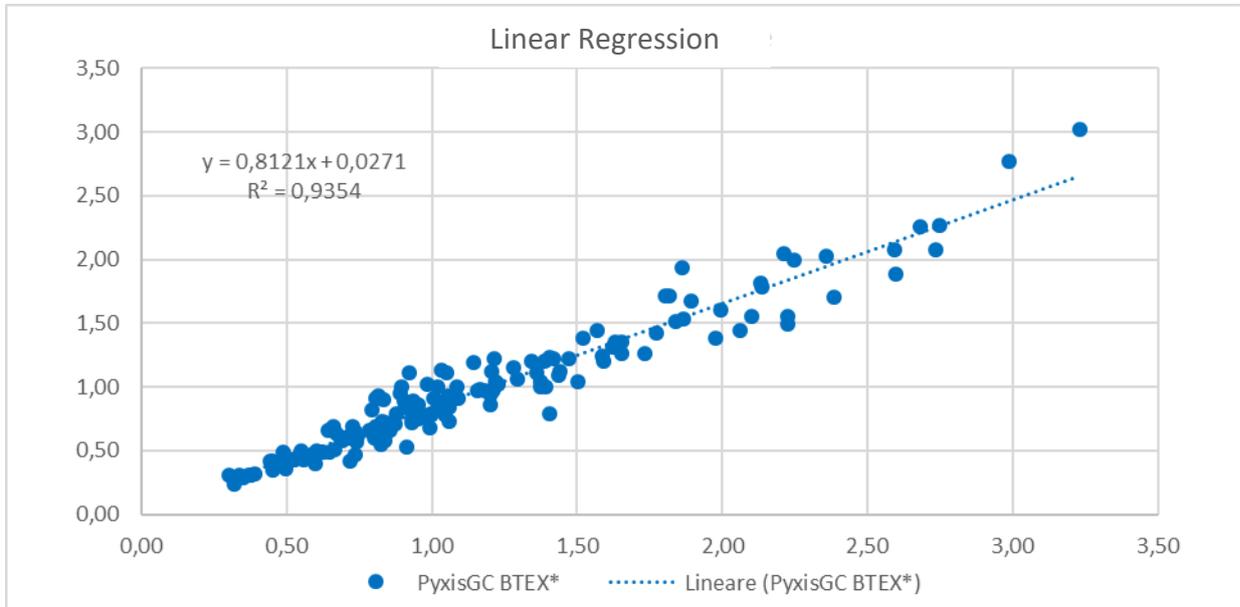
Graph 14 - Calculation of linear regression between instrumental measurements (Benzene concentrations expressed in ug/m³) Week 6

EQUIVALENCE: 85% ---- R²: 0.8323

WEEK 7



Graph 15 - Hourly average Benzene trend (concentrations expressed in ug/m³) Week 7



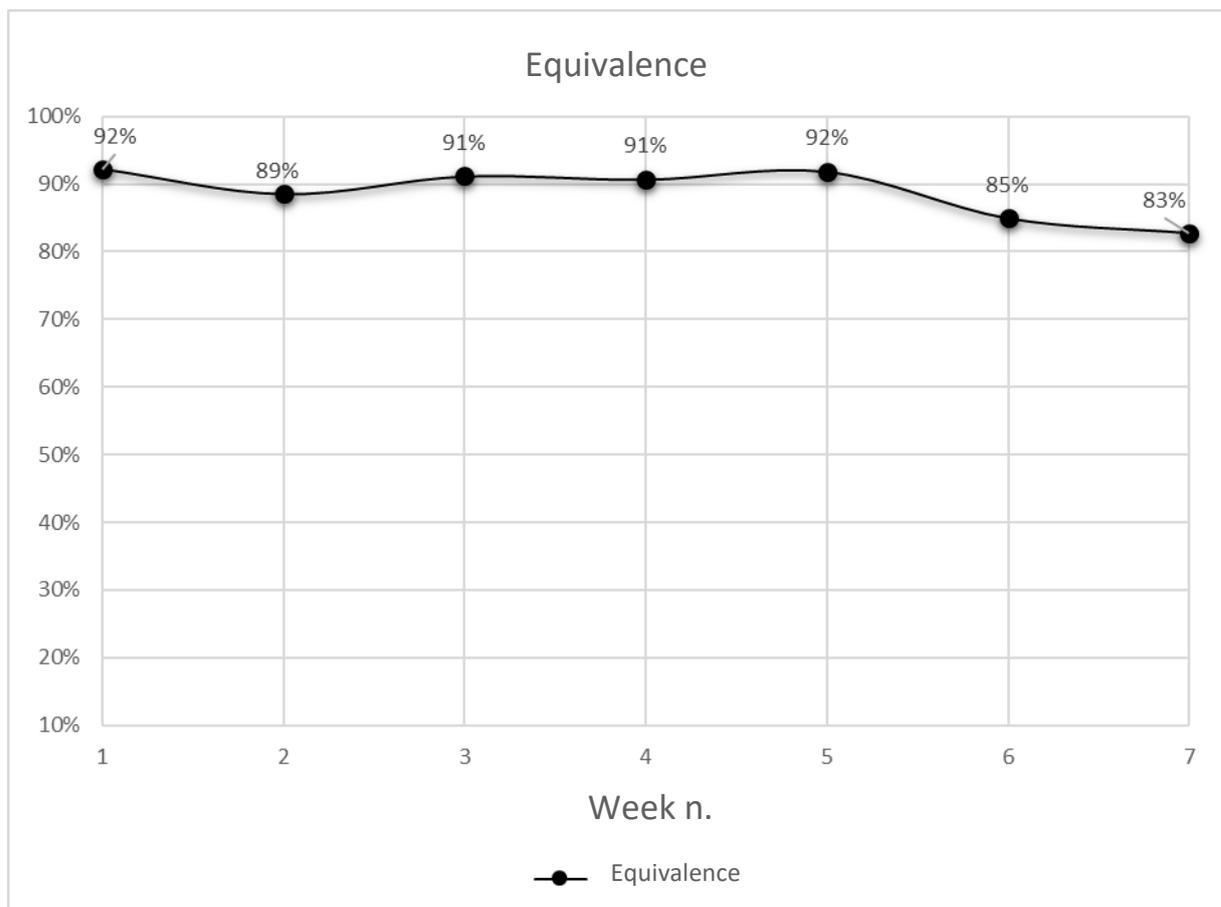
Graph 16 - Calculation of linear regression between instrumental measurements (Benzene concentrations expressed in ug/m³) Week 7

EQUIVALENCE: 83% ---- R²: 0.9354

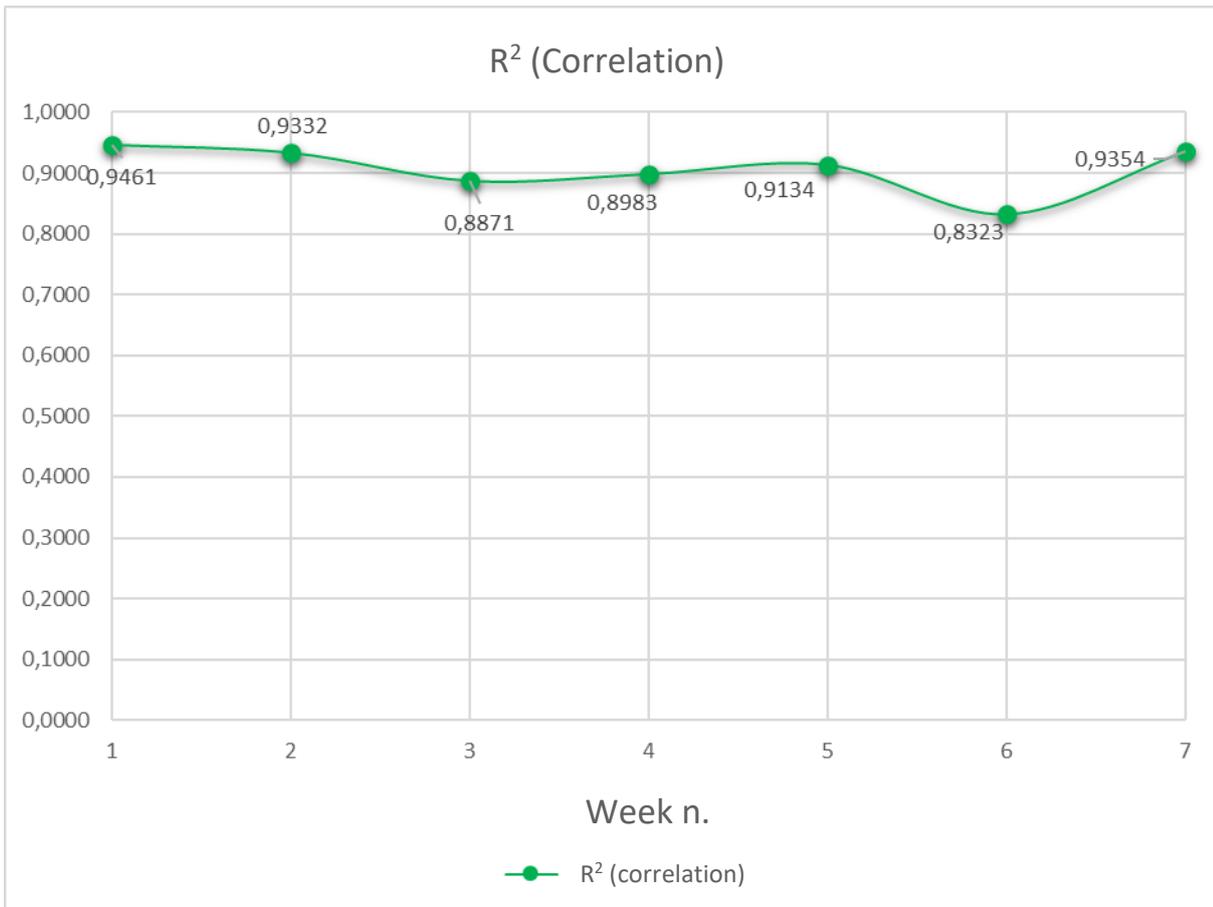
The table and graph below show the equivalence and correlation (R^2) value calculated as a percentage between the measurements obtained with both instruments.

WEEK	% EQUIVALENCE	R^2	R^2 %
1	92%	0,9461	95%
2	89%	0,9332	93%
3	91%	0,8871	89%
4	91%	0,8983	90%
5	92%	0,9134	91%
6	85%	0,8323	83%
7	83%	0,9354	94%
AVERAGE	89%	0,9065	91%

Table 2 - Data relating to Equivalence and Correlation of the measurements carried out



Graph 17 – Equivalence trend of the measurements made



Graph 18 - Correlation trend of the measurement made

Therefore, based on the average correlation data of 0.9065 (91%) it can be stated that the reported results prove the reliability of the PyxisGC BTEX and the accuracy of the obtained measurements.

Furthermore, the data shows that the PyxisGC BTEX perfectly follows the measurements taken with the Puglia ARPA control unit, although it has never been calibrated during the entire test period.



3. CONCLUSIONS

In the light of the results obtained, the following observations emerged:

LINEAR REGRESSION: As can be seen from the graphs of the individual weeks and the graph of the total linear regression of the entire measurement campaign (Graph 18), the data demonstrates perfect functionality of the PyxisGC BTEX and the technical-analytical validity of the individual components: sampling, pre-concentration, injection, separation, and detection system.

EQUIVALENCE: The calculated Equivalence data shows how, up to the fifth week, the values are constant and linear around 91% (Graph 17). There is deviation from linearity in the two weeks following testing.

This trend can derive from 2 main factors:

- Instrument pre-calibration: both instruments were calibrated in different places, times, conditions and using different cylinders; this causes a different starting condition.
- PID detector drift: the lack of an auto-check and auto-calibration system in the PyxisGC BTEX during the entire 7 week period, did not allow the correction of the natural drift of the detector.

Drift correction of the PID detector:

After assessing the data relating to equivalence, in order to optimise the long-term performance of the PyxisGC BTEX, a self-calibration system was set up on a scheduled basis to eliminate the deviation of the equivalence data.

This system includes a canister containing a known toluene concentration; the gas is diluted in a controlled manner with filtered ambient air to reach the low concentrations required by the instrument to perform the calibration.



Figure 5 - Inside PyxisGC BTEX

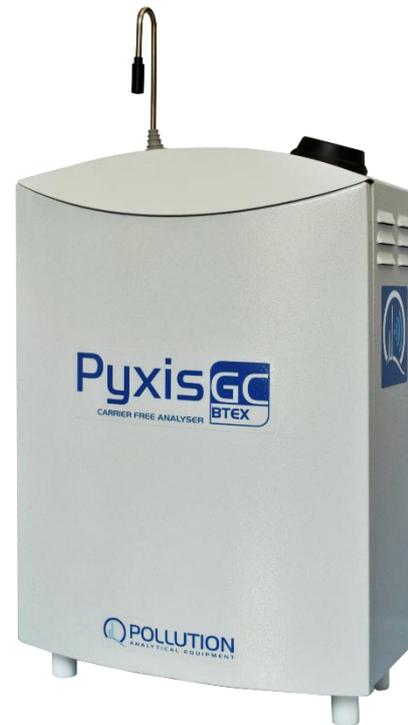


Figure 4 - Outside PyxisGC BTEX

3.1 Advantages of the PyxisGC BTEX

Simple installations suitable for all places

The lack of gas-carrier cylinders for the operation of the instrument allows the installation of the control unit in all possible locations (electric poles, traffic lights, buildings, schools, industrial plants, tunnels, etc.).

Moreover, the miniaturisation and the extremely reduced weight of the entire system guarantee great practicality of use for professionals and a very low impact from a landscape/environmental point of view.

Possibility to create an online smart and remote network

The software installed in the PyxisGC BTEX systems allows to monitor and manage the data acquired by the *POLLUTION GUARDIAN* cloud platform, which can be accessed through a web browser and dedicated APP. Thanks to the communication system integrated in the PyxisGC BTEX, it is possible to create a network of control units, with point-to-point distances up to 400 meters.

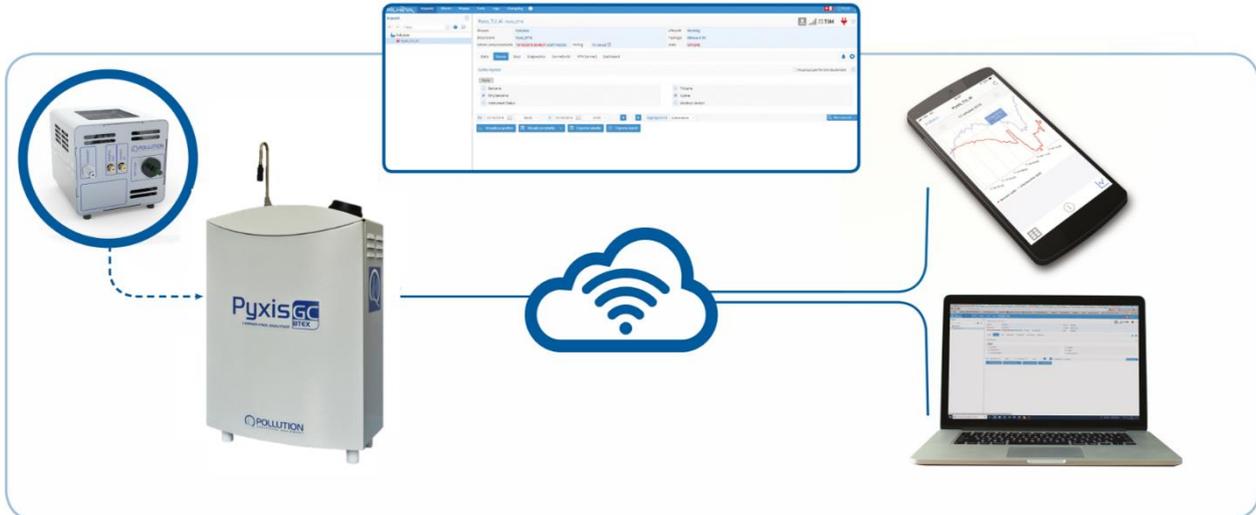


Figure 6 - Cloud System for PyxisGC BTEX

3.2 Applicability of the PyxisGC BTEX

The PyxisGC BTEX features that emerged in the report have numerous benefits and advantages in the possible application fields listed below:

- Fence Line Monitoring
- SMART Factory IoT
- SMART CITY
- INDOOR MONITORING
- EMERGENCY INSTALLATIONS (fires, accidental gas leaks, reclamation of contaminated sites, landfills, etc.)