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Reliability of Field (Onsite) Chemical Measurements: T4i FemtoMachine® Calibrator for QA/QC of Chemical Detectors and Analytical Instruments

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T4i engineering

RELIABILITY OF FIELD (ONSITE) CHEMICAL MEASUREMENTS: T4I FEMTOMACHINE® FOR QA/QC OF CHEMICAL DETECTORS AND ANALYTICAL INSTRUMENTS

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Abstract

This study presents the testing, features, and applications of the T4i FemtoMachine®, a hand-portable field calibrator designed for chemical detectors. The calibrator generates stable vapor concentrations of a wide range of compounds relevant to environmental monitoring and human exposure, including volatile organic compounds (VOC), hazardous air pollutants (HAP) and toxic chemicals. Compounds evaluated in this study include acetone, benzene, toluene, xylene (BTX), methyl salicylate (MSA), dimethyl methyl phosphonate (DMMP), diethyl malonate (DEM), and diethyl phthalate (DEP). Operating on principles of permeation, heating, successive dilution with purified air, and vapor splitting, the calibrator delivers consistent concentrations from low ppb to low ppm ranges. Unlike conventional calibration systems, the T4i FemtoMachine® eliminates the need for gas cylinders by generating its own zero air. Long-term testing over 2,880 continuous operational hours (24/7) has demonstrated high stability and repeatability in vapor generation. Engineered with specialized materials, its inner surfaces minimize chemical interactions with the tested compounds. The calibrator's oven (patent pending) ensures precise temperature control (±0.2°C) and flow rate regulation (±1%). Applications include both indoor and outdoor calibration of atmospheric pressure detectors, such as photoionization detectors, ion mobility spectrometers, gas chromatographs, and gas chromatograph-photoionization detectors, as well as vacuum-based chemical analyzers, including mass spectrometers.

Key-words: *T4i FemtoMachine®, field calibrator, hand-portable calibrator, zero air, vapour generator, permeation tube or vial, chemical detectors, chemical analyzers, Volatile Organic Compounds (VOC), Hazardous Air Pollutants (HAP)*

Introduction

Accurate detection, identification, and monitoring of chemical compounds require frequent qualitative and quantitative calibration of detectors, analyzers, and chemical sensors to ensure reliable measurements. This need is particularly pronounced for field instruments, which operate outside laboratory settings to provide on-site vapor measurements of volatile organic compounds (VOC), hazardous air pollutants (HAP) and toxic chemicals. Routine environmental analyses and emergency response scenarios involving the accidental or deliberate dispersion of chemicals demand highly precise field calibrations to support critical decision-making, such as evacuation procedures and the deployment of protective equipment. Despite the growing reliance on field instruments, there remains a gap in the availability of portable calibrators designed specifically for on-site calibration of detectors and analyzers, including hand-portable photoionization-based detectors, field ion mobility spectrometers, field mass spectrometers, and field gas chromatography-photoionization or gas chromatography-mass spectrometry analyzers. Existing laboratory calibrators are predominantly bench-top devices, requiring detectors to be brought to the calibrator for calibration. These systems are not suitable for field applications due to their weight, bulkiness, dependence on mains power, and, in many cases, the necessity of gas cylinders for operation. To address this limitation, a novel hand-portable calibrator, the T4i FemtoMachine®, has been developed. Weighing less than 2.5 kg and approximately the size of an A4 sheet of paper, this device is rugged (IP66-rated), follows MIL-810H standard, and can be used both inside and outside laboratory settings. The calibrator operates on the principles of permeation tubes and vials, where vapors of a specific chemical compound are generated in an oven via diffusion through a polymer tube or glass vial containing the liquid or solid substance. These vapors are subsequently diluted with clean gas or air and, if necessary, further split to achieve precise calibration concentrations. The T4i FemtoMachine® incorporates an innovative, patent-pending oven that integrates a miniaturized heating system with a tube-like vapor mixer, enhancing efficiency while maintaining chemical inertness. This design ensures high-performance vapor generation within a concentration range from low parts per billion (ppb) to high parts per million (ppm). Compared to conventional bench-top calibrators, the T4i FemtoMachine® offers several competitive advantages, including miniaturization, elimination of pressurized gas cylinders, uniform heating of the vapor generation system, easy preparation of various concentration levels, and continuous operational monitoring.

Theoretical part

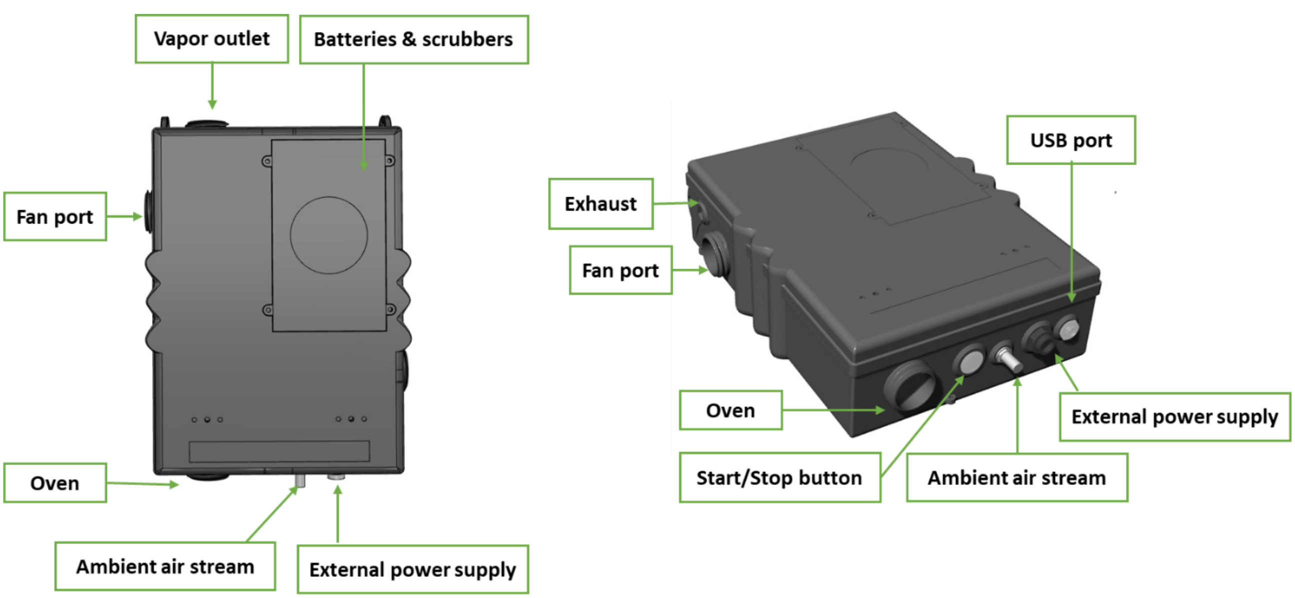
Vapors are molecules of liquids or solids that exist in the gas phase. While gases are defined as substances that remain in a single phase at 20°C, vapors coexist with their liquid or solid phase at the same temperature. The key characteristic of vapors is vapor pressure, which is highly dependent on temperature. The boiling point of a liquid is the temperature at which its vapor pressure equals atmospheric pressure at sea level. Vapor pressure is commonly measured in millimeters of mercury (mmHg or Torr) or kilopascals (kPa).

Volatile Organic Compounds (VOC) refer to liquid or solid organic substances with high vapor pressure and low water solubility, leading to their emission in the gas phase. VOC sources include solvents, gasoline and other fuels, wood preservatives, paints, cleaning agents, disinfectants, pesticides, construction materials, adhesives, office supplies, industrial processes, waste collection and treatment, and natural events such as forest fires. VOCs are typically defined as organic compounds with a boiling point of ≤250°C at standard atmospheric pressure (101.3 kPa). Based on their boiling points, VOCs are categorized as: very volatile organic compounds (VVOCs) with boiling points ranging from 0°C to 50-100°C, volatile organic compounds (VOCs) with boiling points between 50-100°C to 240-260°C, semi-volatile organic compounds (SVOCs) with boiling points from 240-260°C to 380-400°C.

VOC concentrations in indoor and outdoor air are typically expressed in parts per million (ppm), parts per billion (ppb), parts per trillion (ppt), or mass per cubic meter (mg/m^3, $\mu g/m^3$). The quantification of VOC levels can be performed through laboratory analysis of collected samples or on-site measurements using field detectors and analyzers. The latter is particularly critical in emergency scenarios where chemicals have been released into the environment, whether accidentally or deliberately. For accurate VOC measurements in indoor and outdoor environments, analytical instruments must undergo qualitative and quantitative calibration to ensure reliability. Devices capable of generating stable, continuous vapor concentrations at different levels play a vital role in instrument calibration and contribute significantly to Quality Assurance (QA) and Quality Control (QC) processes in chemical detection and environmental monitoring.

Experimental part

1. T4i FemtoMachine® annotated diagram



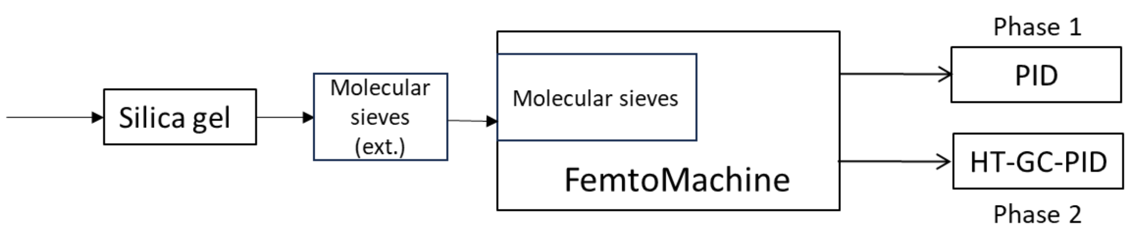
2. Materials and methods

Materials: Permeation tubes of Acetone, Benzene, Toluene, and Xylene (BTX), Methyl Salicylate (MSA), Dimethyl Methyl Phosphonate (DMMP), Diethyl Malonate (DEM), and Diethyl Phthalate (DEP) were used.

Method: Through the software of the instrument a method is developed in which the oven temperature and the concentration level and the output flow are set. The method developed can be stored and run each time calibration is needed. Various other parameters can be set in the method such as the time of running the method and the time of running purified air for cleaning the device. Other options of the software include bake-out of the device for cleaning sticky compounds, autopilot for running routine, everyday method, message box for receiving operation messages. Temperatures for the permeation tubes used in this study were as following: Acetone, Benzene, Toluene, Xylene, MSA at 60°C, DMPP at 90°C, DEM at 80°C, and DEP at 30°C. The calibrator output with the stable concentration of the chemical was first measured by connection to a MiniPID2-HS sensor at 10.6 eV operating at ambient temperature (phase 1). Then (phase 2) the calibrator was connected to a fast mini-GC unit operating at high temperature (HT-GC). The output of the unit was connected to the PID of phase 1. The signals of the PID sensor (phase 1) and the GC-PID (phase 2) in mV were recorded versus time of measurements.

3. Experimental set up

The experimental set up, placed in a fume hood, is described in the following block diagram:



To ensure the avoidance of cross-contamination of the vapor generator and, consequently, the reliability of the sensor's recording, a series of air purification scrubbers was used to supply clean air to the FemtoMachine’s inlet. A pair of external silica gel scrubbers was connected to an also external molecular sieves scrubber that directly supplied with clean air the vapor generator’s inlet. In the exhaust of the generator the high temperature GC is connected.

4. Procedures

The following procedures were applied for every chemical:

Phase 1

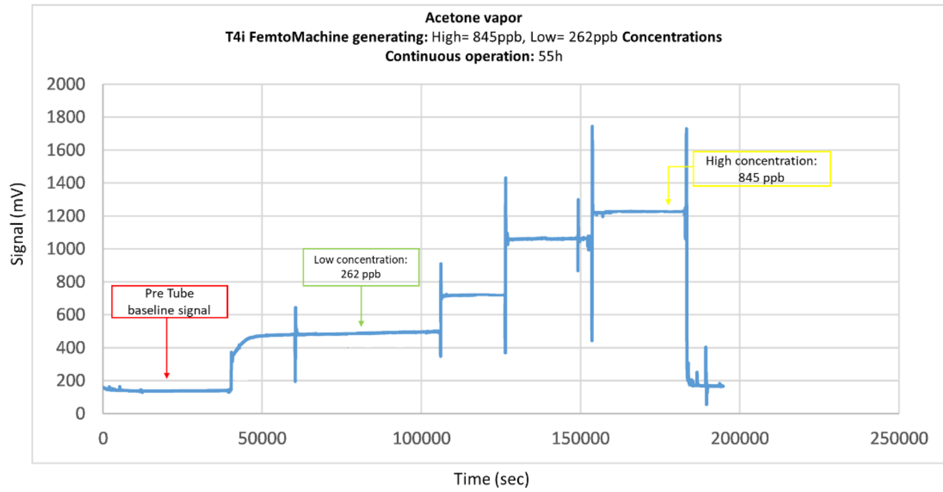
1. Use of a permeation tube in the vapor generator for the production of chemical vapors at different concentration levels
2. Connection of the vapor generator with the PID and signal recording for defining time required for stabilization of the produced concentration
3. Change of the concentration levels of the chemical and signal recording for defining time required for variation of the concentration
4. Repeat steps 1-3 for all chemicals

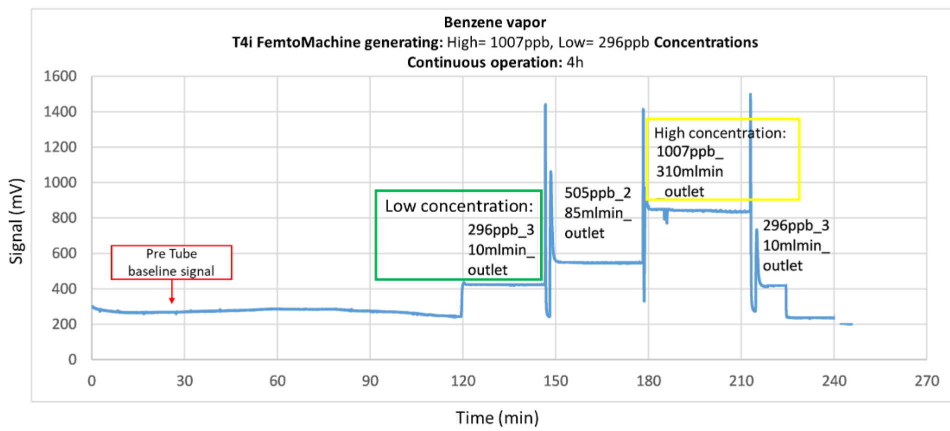
Phase 2

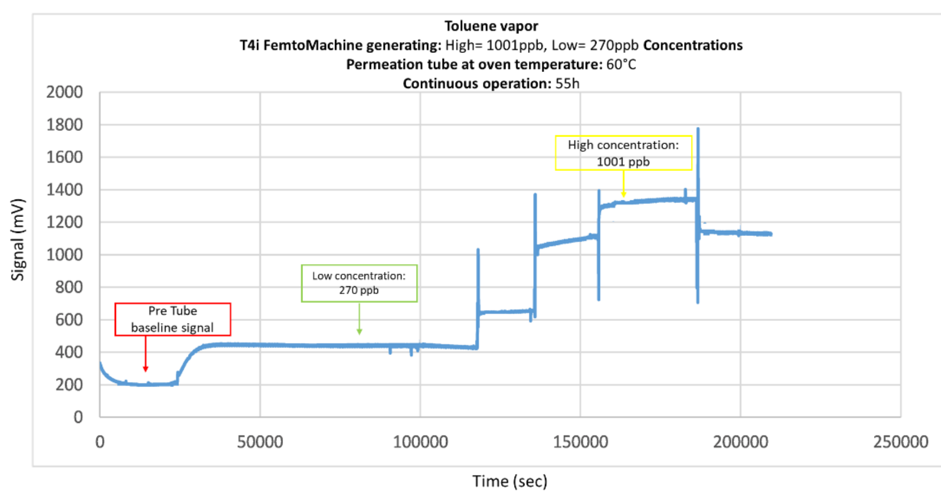
1. Use of a permeation tube in the vapor generator for the production of chemical vapors at the maximum possible concentration
2. Using data acquired from phase 1, connect the HT-GC/PID to the vapor generator, which produces a known chemical concentration
3. After several samplings, define retention times

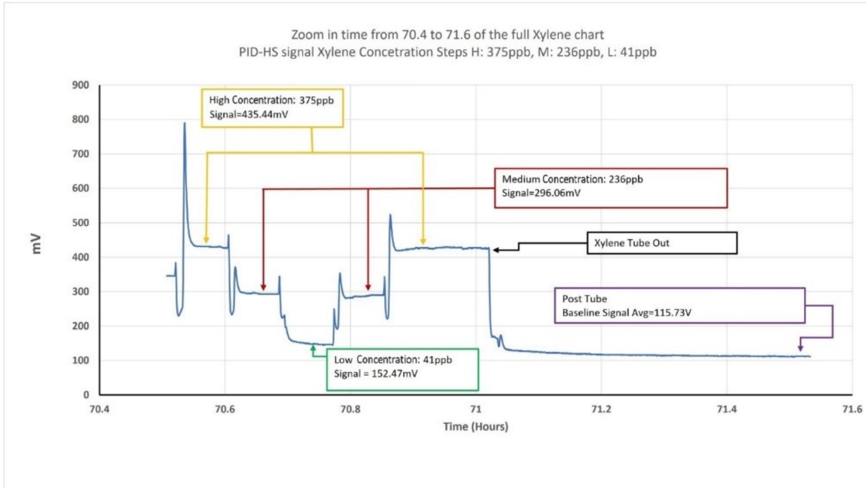
Results and discussion

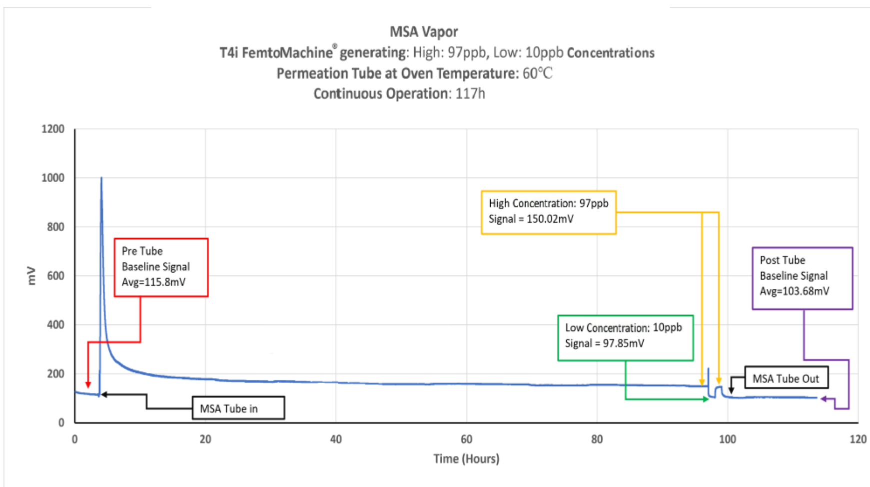
Phase 1

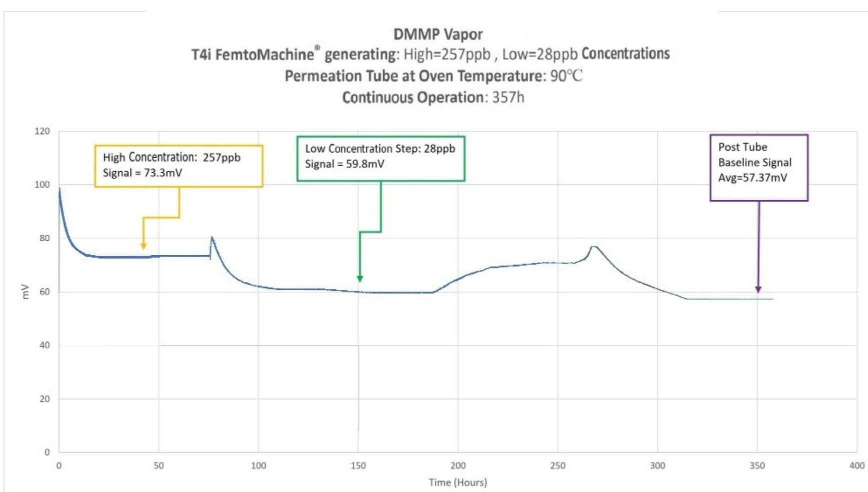


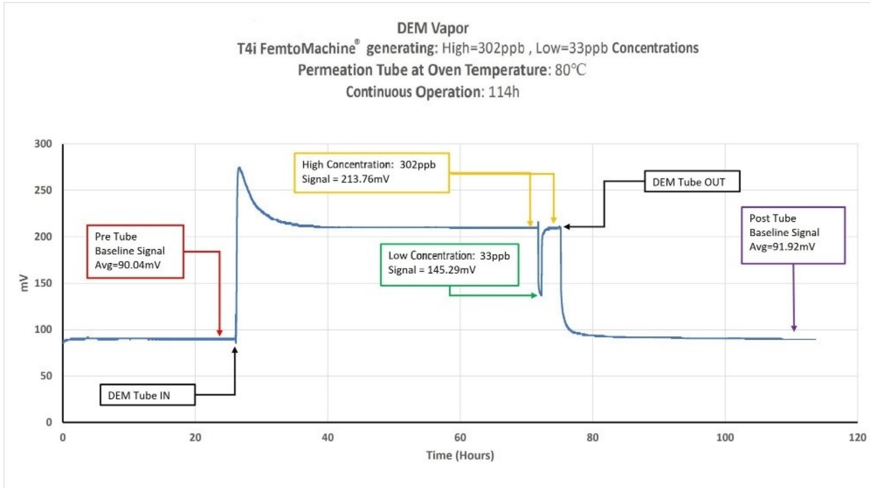





Zoom in time from 70.4 to 71.6 of the full Xylene chart
PID-HS signal Xylene Concetration Steps H: 375ppb, M: 236ppb, L: 41ppb
High Concentration: 375ppb
Signal=435.44mV
Medium Concentration: 236ppb
Signal=296.06mV
Xylene Tube Out
Post Tube
Baseline Signal Avg=115.73V
Low Concentration: 41ppb
Signal = 152.47mV
mV
Time (Hours)


MSA Vapor
T4i FemtoMachine® generating: High: 97ppb, Low: 10ppb Concentrations
Permeation Tube at Oven Temperature: 60℃
Continuous Operation: 117h
High Concentration: 97ppb
Signal = 150.02mV
Pre Tube
Baseline Signal
Avg=115.8mV
Post Tube
Baseline Signal
Avg=103.68mV
Low Concentration: 10ppb
Signal = 97.85mV
MSA Tube Out
MSA Tube in
mV
Time (Hours)


DMMP Vapor
T4i FemtoMachine® generating: High=257ppb , Low=28ppb Concentrations
Permeation Tube at Oven Temperature: 90℃
Continuous Operation: 357h
High Concentration: 257ppb
Signal = 73.3mV
Low Concentration Step: 28ppb
Signal = 59.8mV
Post Tube
Baseline Signal
Avg=57.37mV
mV
Time (Hours)



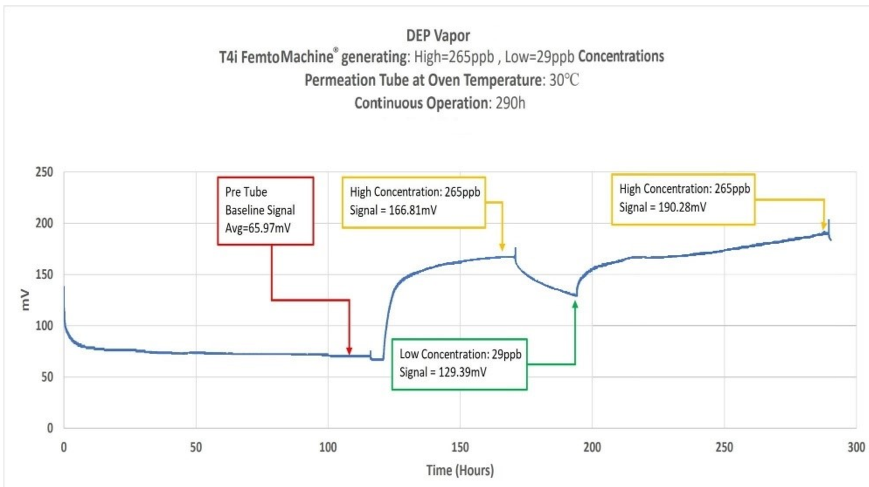
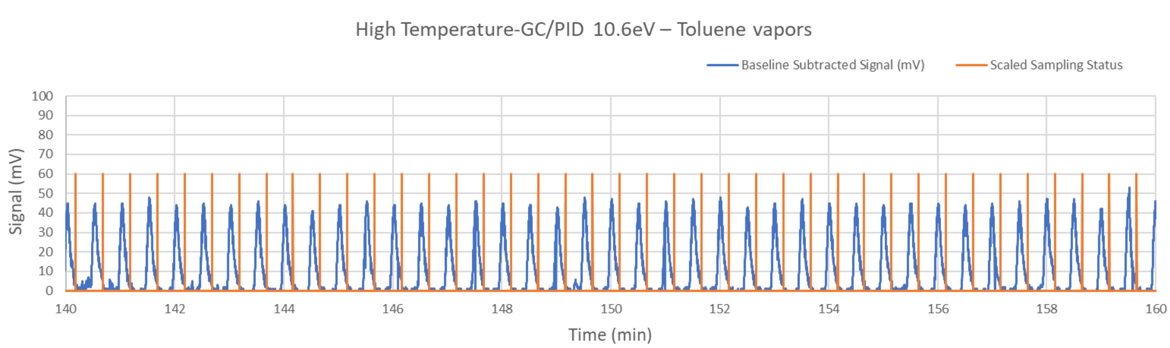


Figure 1: PID signals versus time for different concentrations of vapors of Acetone, BTX, MSA, DMMP, DEM, DEP generated by T4i FemtoMachine®.

Phase 2



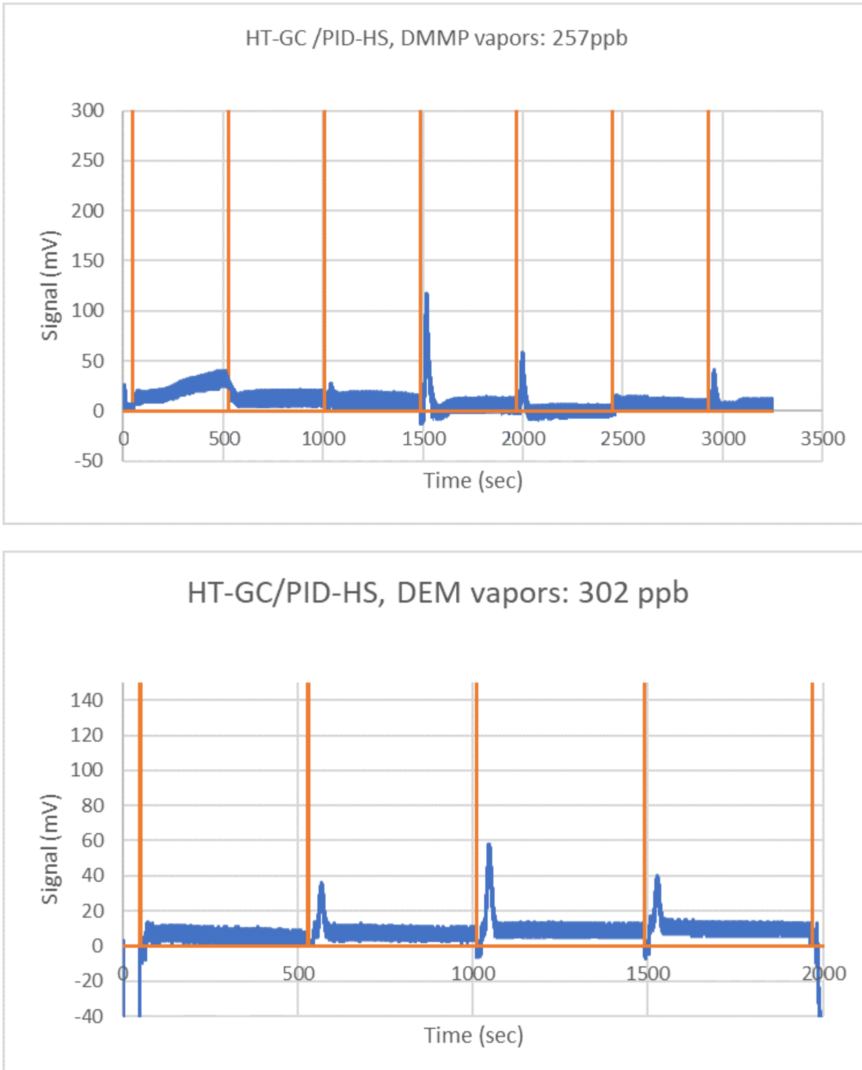


Figure 2: HT-GC/PID signals versus time for a single concentration of Toluene, DMMP and DEM vapors.

Conclusions

T4i FemtoMachine® has demonstrated its capability to generate stable vapor concentrations across a broad range of chemical compounds while operating continuously. Designed as a lightweight, hand-portable, and battery-operated device, it enables calibration of chemical detectors both inside and outside laboratory settings, ensuring the reliability of field measurements. Another application is for testing chemical sensors, chemical detectors/analyzers set ups and configurations in long term operation by providing stable vapor concentrations. Long-term performance evaluation over 2,880 hours of continuous operation has confirmed its stability and minimal maintenance requirements, with only periodic replacement of molecular sieve scrubbers needed. Furthermore, the T4i FemtoMachine® is CE-marked and complies with multiple industry standards, reinforcing its suitability for rigorous analytical applications.

Acknowledgements

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2. T4i FemtoMachine®, QA/QC of chemical sensors/detectors/analyzers in the field, https://www.t4ieng.com/t4i-femtomachine/
3. EU-funded EDF2021 research project TeChBioT. Surveillance and Reconnaissance Techniques for Chemical and Biological Threats. Grant Agreement No. 101103176, https://techbiot.eu/ (accessed Dec 1, 2022).