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Transportable, Portable and Micro Gas Chromatographs

Witkiewicz Z[1] and Wardencki W[2*]

[1]Institute of Chemistry, Military University of Technology, Warsaw, Poland

[2]Department of Analytical Chemistry, Chemical Faculty, Gdańsk University of Technology, Gdańsk, Poland

[*]**Corresponding author:** Wardencki W, Department of Analytical Chemistry, Chemical Faculty, Gdańsk University of Technology, Gdańsk, Poland, E-Mail: waldemar.wardencki@pg.edu.pl

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Abstract

Significant benefits can be obtained by miniaturization of conventional benchtop gas chromatographs. The attractive features arising from miniaturization include low-power requirements, low heat capacity of separation columns, low consumption of mobile phase, enhanced performance, higher portability, and easily coupling to detectors and secondary chromatographic systems. In separation sciences, including gas phase separations, the downsizing of the main components of laboratory instruments is increasingly investigated to obtain high performance separation systems. This review covers the important advances in downsizing of laboratory gas chromatographic systems. The paper briefly describes a key components of micro Gas Chromatograph (μGC) systems: injectors/preconcentrators, columns and detectors, and characterizes the whole modern portable and transportable gas chromatographs with emphasis on their connection with mass and ion mobility spectrometers (MS and IMS). The idea of microfabrication of chromatographic columns, based on Microelectromechanical Systems (MEMS) technology, and a brief history of the miniaturized GC systems are presented. The examples of applications of mobile gas chromatographs together with their analytical characteristics are given.

Keywords:* *Micro gas chromatographs; Mobile chromatographs;

Introduction

There has been a distinct tendency in modern analytical chemistry to detect, identify and determine the analysts in sample collection sites. It is especially important in the analysis of different hazardous pollutants of the environment, mainly in the monitoring of air quality [1]. It is obvious that the analysis in such cases should be fast and reliable. All these requirements may be fulfilled using instruments that can work in the field conditions. Miniaturized analyzers should work in different places and in different conditions, that is. They should be transportable or portable. Gas chromatographs that have been still improved since the advent of gas chromatography in 1950 can fulfill these requirements. Among them are micro chromatographs.

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Using chromatographs outside the laboratory eliminates the step of analysis connected with sampling, transport, and storage and sample preparation. All these activities are subjected to risks of contamination, degradation, and decomposition of sample components. Furthermore, some components of the samples may be lost during storage or transport. Comparing to the analysis done in the laboratory, performing analyses on-site distinctively shorten the time from sample introducing into the device and obtaining the final results of the analysis. It is especially important in the determination of toxic substances that can appear in the environment, for example, due to a failure in industrial plants.

Mobile gas chromatographs usually are not as universal as laboratory gas chromatographs and are frequently destined for analysis of a specific group of chemical compounds, the example is given, hydrocarbons present in different mixtures or chemical weapons. But mobile chromatographs do not possess drawbacks of laboratory instruments. Conventional gas chromatographs are bulky, mainly due to the incorporation of a relatively big heating oven chambers and the necessity of using carrier gas cylinders or gas generators. They are more power consuming and have slow response leading to a rather long analysis time. The number of analyses that can be realized during one day of a work is frequently limited, especially if analyses are performed in a programmable temperature of a column. Large instrument size and high capacity air-bath oven limit applications of high ramps of column programming temperatures. Long time of cooling, after reaching the final column temperatures in programmable approaches (about 250-350°C), also do not allow to the short time of analysis.

General characteristics of mobile chromatographs

Two general categories exist for mobile chromatographs. The largest mobile instruments weight several dozen kilograms and are moved to the field use in the vehicles. Such instruments may be part of the equipment of mobile laboratories and may be supplied with gases and electricity of a vehicle. Instruments that can be carried by one person that is not exceeding 25 kg are known as a handheld or person-portable GC instruments.

Person-portable chromatographs are generally smaller and lighter than conventional transportable chromatographs and use less energy. They are usually powered by the batteries and equipped in small gas tanks. Among person-portable instruments, there are devices with weight about one kilogram but there are also devices built on chips, obtained using Microelectromechanical Systems (MEMS). The MEMS technology allows fabricating instruments and their elements in the micrometer scale.

Today mobile chromatograms are produced using the modern technological advancements and allow to obtain results fully comparable with laboratory instruments. Some constructions applied in mobile chromatographs are not still used in laboratory instruments. Their functionality and quality of analyses are fully comparable with conventional instruments. Some mobile chromatographs are working as monitors and may work a long time without supervision and send results for long distances, for example from planets to the Earth.

The ovens of mobile chromatographs have compact construction having the possibility of the column heating up to 400°C using different ramps of temperature programming and the possibility of column cooling from 250°C to 50°C in a time shorter than 5 min. The construction with two ovens in which different temperatures are applied is also available. The eluate from the first column may be introduced to the column inserted in the second oven, in which separation of unresolved components from the first column can be continued. It is a typical system for fast and simultaneously for two-dimensional chromatography.

Conventional heating in air-bath ovens is not convenient for transportable chromatographs and especially for person-portable instruments. Using air-bath ovens needs high energy; heating is conducted in relatively bulky ovens causing that relatively long time of heating and column cooling is needed. Therefore, in mobile chromatographs, the direct heating of columns or using heaters adjusted to the shape of a column or resistive heating of metallic housing of column is used. Such heating approach is characterized by low power requirements and additionally, provides fast changes of column temperatures, whereas the oven size is only a little bigger than a column. Further improvements in this technology may lead to apply a resistive column heating in laboratory instruments.

Liquid and gas samples can be introduced into the column using different types of injectors; even 5 injectors may be mounted in one chromatograph. In some instruments, integrated systems for analysts Preconcentration or sample preparation in another way may be installed. For example, two parallel sorption traps are used from which the analysts are released by thermal desorption. Purge and trap and static headspace analysis systems are also used. The injectors may be connected with e.g. ten-port valve enabling simultaneous separation of samples in two columns, or column switching and changing of gas flow direction.

The quartz or metallic capillary columns with an internal diameter of 0.53 mm, with typical length or steel or Teflon, packed columns with a small diameter and 2 m long or longer may be used in mobile chromatographs. They are columns both for gas-liquid chromatography and gas-solid chromatography. For example, molecular sieve 13X, polymer Hayes Sep D, silica gel or Carbopack B are used as packing material for adsorptive columns. Two columns in oven, parallel or in series may be used at the same time for analysis of the same sample.

All types of detectors used in laboratory instruments are used in mobile chromatographs. The transportable chromatograph is known in which 6 different detectors were installed. The detectors can work as single devices, registering simultaneously chromatograms of the same sample separated in two columns or can work connected in series, for example as in TCD-methanizer-FID system. Methanizer transforms carbon oxides that are not detected using TCD, into methane that is easily detected (for example 5 ppm) by FID. Air for FID was supplied from quietly working compressor.

The computer programs enable registering signals from each detector, their evaluation and calibration of devices using different standard substances.

Micro gas chromatographs

The construction of chromatographs in microscale became possible due to constant improvements in the technology of micro-electro-mechanical systems. Applying such microstructures creates the possibility of designing systems described as "lab-on-chip". It allows downsizing of all key elements of gas chromatography: that is injectors, detectors, and especially columns, in sizes of single or hundreds micrometers. Micro chromatographs provide not only the desirable feature of smaller size and higher portability but also lower power consumption and minimal production and maintenance costs.

"Lab-on-chip" systems are divided on the base of the structure into two categories as a module or fully integrated constructions. Module microstructures are mainly used for research purposes, in which frequent modifications or changes in experimental conditions are needed. The integrated microsystems are more complicated but are very usable.

The basic initial substrate used in microfabrication of MEMS systems is silicon or silicon connected with borosilicate glass (Pyrex), characterized by high chemical inertness and high inertness for temperature changes. Other materials such as porous silica gel, metals, ceramics or nanotubes are rarely applied for fabrication of the microsystems.

The technology of microfabrication of gas chromatographic columns is schematically presented in **FIG. 1**.

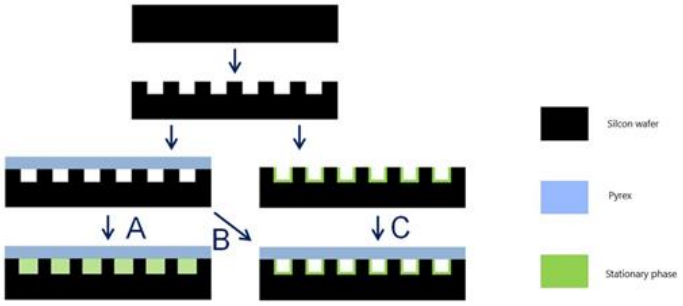

FIG. 1. **The general idea of Silicon-Pyrex microcolumns fabrication for miniaturized gas chromatographs using MEMS technology**.

First, in a blank silicon wafer, after photolithographic treatment, microchannels are etched, with chosen shape and dimension. The channels usually have a rectangular shape. Their dimensions (typically 50-150 μm) depend on etching procedure and process parameters. Then, the wafer is either bonded to a Pyrex lid and diced (left side of **FIG. 1**), or either covered with thin layer of stationary phase, for example, carbon nanotubes (right side of **FIG. 1**). In the first case, a solid material (for example

silica gel, graphitized carbon alumina, molecular sieve) is packed or monolith structure is grown inside the column (process A) or the inner walls of the column are coated with a gel material stationary phase, for example, PDMS (process B). In the second case, the wafer is bonded to a Pyrex lid and diced (process C).

There are several techniques developed for bonding and sealing the MEMS micro GC columns: among them are anodic bonding, eutectic bonding, and fusion bonding. Anodic bonding is a well-established, and probably the one with the lowest capital cost bonding technique for properly sealing of microchannels columns and currently accounts for the majority of the silicon microfabricated GC columns.

Anodic bonding relies on heating silicon-glass substrate to high temperature (300-500°C), and next, silicon wafer placed on the table is positively polarized, whereas Pyrex lid inserted on silicon is negatively polarized with relatively high voltage (500-2000 V). In the result, both surfaces are connected by strong chemical bonding. This step is usually conducted in the final step of fabrication of microstructures after micromechanical procedures.

In some miniaturized gas chromatographs interchangeable cassettes containing automatic injector with sampling loop, columns with resistive heating and micro thermal conductivity detector are applied. The device can work without supervision with the possibility of transferring remotely the results of the analysis. Automatic calibration is also possible.

The chip-based technology becomes also successfully applied for construction of multidimensional micro gas chromatographs. For example, a novel smart multi-channel two-dimensional (2-D) Micro-Gas Chromatography (μGC), with better performance in comparison to the conventional 2-D μGC, was designed and introduced (**FIG. 2**) [2].

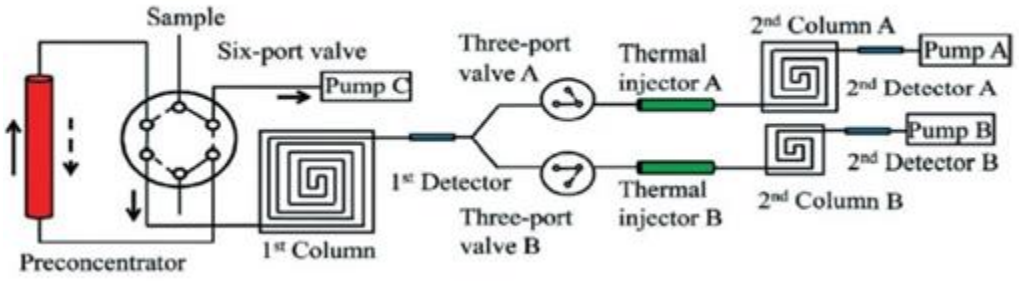

FIG. 2. **The schematic diagram of chip-based multidimensional gas chromatograph: each column has separate injector and on-column optical fiber detector [2].**

In this smart μGC design, a non-destructive on-column gas detector and a flow routing system are installed between the first-dimensional separation column and multiple second-dimensional separation columns. The effluent from the first-dimensional column is monitored in real-time and decision is then made to route the effluent to one of the second-dimensional columns

for further separation. The system was employed it in two important applications that highlight its uniqueness and advantages, that is an analysis of 31 workplace hazardous volatile organic compounds, and rapid detection and identification of target gas analysts from interference background.

In downsizing of gas chromatographs very useful are also the advancements observed in the fabrication of polymeric composite materials on the base of metalloorganic structures (Metal Organic Framework-MOF) [3,4].

The downsizing of key elements of gas chromatographs instruments is still increasingly investigated to obtain high-performance separation systems. Numerous reviews presenting the state of art of miniaturization have been published in last three decades [5-11]. Below a short history of miniaturization of GC instrumentation is presented.

Important advances in miniaturization of gas chromatographs are given in **TABLE 1**.

TABLE 1**. Important advances in miniaturization of gas chromatographs.**

1979	Introducing the first lab-on-a-chip GC system consisting of a microinjector, a circular-spiral channel column and TCD integrated on a planar silicon wafer [12]
1997	First microfabrication of capillary channels for 2D separations [13]
1998	Fabrication of a functional micro-GC system [14]
1999	Fabrication of multi-capillary column [15]
2002	Producing first micro-FID utilizing a glass-silicon-glass stack configuration [16]
2003	Development of miniaturized GC-MS system for in-situ analysis [17]
2006	Introducing a new three-dimensional micro-concentrator [18]
2007	Fabrication of microcolumns for fast chromatography [19]
2008	Developing portable GC with toroidal ion trap MS detection [20]
2009	Designing the so-called semi-packed micro-separation columns [21]
	First free comprehensive two-dimensional GC system (GC × GC) consisting of a pair of microfabricated columns and microfabricated detector [22]
	Introducing MEMS-based multi-capillary gas chromatographic columns [23]
	Developing a portable GC system with chemiresistor array detector [24]
2010	First commercial micro-GC system (model C2V-200) [25]
	Utilization of a silica-sputtering technique to create a porous silica layer in a MEMS-based GC column [26]
2011	Launching of μChemLab™ consisting a truly monolithically integrated μGC system [27]
2014	Microfabrication of high-resolution GC column with porous silicon acting as support [28]
2015	Development of a disposable gas chromatographic column [29]
	Applying gold eutectic-fusion bonding for microfabrication of all silicon GC column [30]
	The invention of micro helium discharge photoionization detector (μDPID) [31]
2016	Development of a novel portable miniaturized GC/μPID for near real-time low-level detection of BTEX [32]
2017	Integration of ionic liquids for high-performance separation of chemical mixtures [33]

Hybrid mobile gas chromatographs

Similarly to laboratory devices, the mobile instruments consisting of gas chromatographs and other analyzers are constructed. Usually, Mass Spectrometer (MS) is connected with GC [34] but more frequently than in the case of laboratory instruments, GC is connected with Ion Mobility Spectrometers (IMS) [35]. Coupling gas chromatograph with IMS is easier than with conventional MS [36,37]. Ion mobility spectrometer works at the gas flow rate similar to the flow in a chromatographic column and itself IMS is a small and light device.

In last years miniaturized quadrupole mass spectrometers and with ion traps became available. They are so small, that do not greatly influence GC-MS size and weight. The ion traps with toroidal or cylindrical structures enable to register ions in the range from about 50 to about 450 m/z. The instruments with such spectrometers may be easily mobile [19,38]. The using of chemical pumps, instead of typical mechanical ones, for obtaining vacuum additionally favors miniaturization of spectrometers.

Gas chromatographs are connected with ion mobility spectrometers with drift chamber (Drift tube-IMS-DT) or with differential spectrometers (Differential mobility spectrometry-DMS) [37]. The second connection is newer than the first one. Gas chromatographs connected with ion mobility spectrometers are usually equipped with multicapillary columns. The important advantage of GC-IMS systems is a possibility to use ambient air as a carrier gas. It is especially important in IMS, in which there is no necessity to deliver other carrier gas.

Analytical performance of most transportable gas chromatographs is similar or even equal to the performance of conventional chromatography. In the case of person-portable instruments, these performance is a little smaller. Some companies offer to deliver of chromatographs in technical configurations accommodated to particular demands and for analysis of selected analysts. The areas of their applications are very broad.

Application of mobile gas chromatographs

Mobile gas chromatographs are used for analyses in the field for rapid screening of chemicals, including environmental volatile and semi-volatile compounds, explosives, chemical warfare agents, hazardous substances, and for use in food safety and industrial applications. Miniaturized chromatographs are usually adapted to analyze particular types of chemical compounds, for example, volatile organic compounds, explosives materials or toxic chemical weapons. Mobile gas chromatographs are applied in automatic movable or stationary stations, frequently in life-threatening conditions. Among analyzed mixtures, there are different polycyclic polychlorinated biphenyls, explosives [39], chemical weapons like G gases and sulphuric mustard gas [40], and like V type gases [41], volatile chloroorganic compounds [42], greenhouse gases, mine gases, and gaseous sulfur compounds [43].

The developments in the electro Micromechanical Systems (MEMS) technology, especially in the fabrication of monolithically integrated modules still broaden the applications of micro gas chromatographs with low detection limit. For example, recently proposed a unique GC-on-chip module, comprising a monolithically integrated semi-packed micro separation column and highly sensitive micro helium discharge photoionization detector (μDPID), provides rapid separation (<2.5 min) and detection of gas mixture consisting nine compounds ranging from 98°C to 174°C in boiling points, with minimum detection limit of 10 pg. (**FIG. 3**) [31].

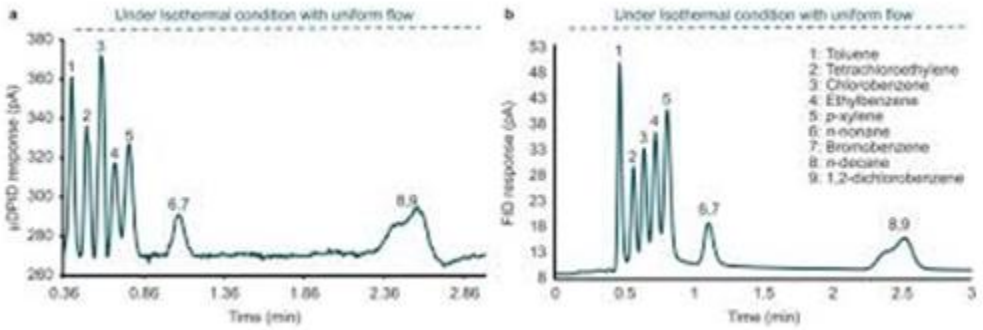

FIG. 3. **Comparison of the response of the μDPID and the FID to 10 μl headspace volume of C7-C10 mixture under isothermal conditions at 40 and a flow rate of 1.33 ml min^{-1} [31].**

The overall performance of mobile gas chromatographs can be improved by coupling them with other analyzers like mass spectrometers. The first mobile gas chromatography coupled to mass spectrometers were applied in cosmic research [44]. Since 2009 year in International Space Station the instrument known as air quality monitor (AQM) is used, in which gas chromatograph is connected with differential mass spectrometer [45]. Using portable, fast GC-MS system, 25 volatile organic compounds after extraction from the water were separated in 65 seconds. GC-DMS instrument was used for the detection of naturally occurring pollutants in wines [46].

Mobile GC-MS system was used, for example, to analyze semi-volatile organic compounds, like terpenes, chloroorganic pesticides, phenolic compounds, polyaromatic aromatic hydrocarbons, and phthalate esters. Instrumentation of this system allowed analysis of all these compounds in air, water, and soil [39].

Gas samples can be automatically introduced directly into mobile chromatographs. Mobile chromatographs may be equipped in additional devices allowing preparation of different samples with different matrices for analysis, like in case of laboratory chromatographs. For example, analysts from the air can be preconcentrated on adsorbents and next released by thermal desorption. Analyzes from liquid and solid samples may be thermally desorbed [39] or using purge and trap technique or headspace can be analyzed. Solid-phase Microextraction may be also used [47].

Conclusion

Due to intensive academic and industrial research in last three decades on the downsizing of the main components of laboratory instruments the miniaturized gas chromatographic systems can provide fast and reliable qualitative and quantitative analysis of different chemical vapors in ultra-small instruments. Such systems provide the desirable feature of smaller size and higher mobility, lower power consumption and dramatically expand the fields of use of chromatographic chemical analysis to seconds rather than minutes to hours.

Although progress has been made in the development of micro-GC instruments through the use of macro fabricated columns and various microscale detectors further investigations are needed to meet the needs of analytical scientists. Especially, a research on the integrating the basic components of μGC systems, including sampling/injectors, separation columns and detector on a single monolithic module should be continued. Monolithic integration of all microelements of GC systems can lead to a reduction in the chip-to-chip interface, thereby reducing changes of cold spots offering improved performance as well as reducing the manufacturing costs of this technology.

Improvements are also needed in the fabrication of microcolumns. In spite of significant progress made in microcolumn separation efficiency there some problems with obtaining a uniform thin coating that will not delaminate from the structural walls of the microcolumns.

There is also high demand for more autonomous portable GC instruments, i.e. devices that do not require daily maintenance and can be placed in a remote location for long-time service. This can be realized, for example, by the remote battery charging with the radiofrequency transmission.

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