Instrumentation of Gas Chromatography

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Instrumentation for gas chromatography (GC) comprises well-defined components, each of which contributes to the overall chromatographic performance. Most of these components have reached a mature level of technical development after nearly 50 years of development. Nonetheless, advances are occurring in an evolutionary, not revolutionary, manner with sample handling and in refinements of detectors. One area of dramatic advance in GC instrumentation is the development of small, sophisticated, portable gas chromatographs. Each component of a gas chromatograph (columns excepted) is discussed below where principles and recent developments are emphasized.

1 INTRODUCTION

The key parts of a gas chromatograph include: a source of gas as the mobile phase, an inlet to deliver sample to a column, the column where separations occur, an oven as a thermostat for the column, a detector to register the presence of a chemical in the column effluent, and a data system to record and display the chromatogram. In addition, some facility is needed so that temperatures of various components can be accurately known and controlled. These parts of a gas chromatograph have been unchanged in function or purpose for over the last 40 years although technology has been ever improving in design, materials, and methodology. In particular, analog electronics for control of temperature zones and data acquisition were replaced with digital electronics and interfaced with computers in the 1970s and 1980s. The arrangement of these components is shown in a block diagram in Figure 1 and this arrangement is common to virtually all gas chromatographs regardless of age, model or manufacturer. A modern gas chromatograph is shown in Figure 2. In the discussion below, the general function of each component is provided with comments on the status of the technology. Most descriptions of GC will include a cursory description of instrumentation; few will provide a detailed treatment of the instrumentation or technical details. Some of the best discussions of hardware can be found in publications released by instrument manufacturers.[1,2] Unfortunately, these may not be found routinely in libraries but the reward for efforts to obtain them is found in the useful details for optimizing an analysis or practical help for maintaining the instrument.

The column may arguably be considered the key component of a gas chromatograph and accordingly has been treated separately under another heading. However, the total variance of a separation (σ_T) will conform to principles of error propagation and be a sum of variances from the injector (σ_I), column (σ_c), detector (σ_d), and data system (σ_{ds}), i.e. $\sigma_T = \sqrt{\sum(\sigma_i + \sigma_c + \sigma_d + \sigma_{ds})}$. Thus, each of these components contributes to the overall efficiency of a GC separation and merits individual attention.

2 CARRIER GAS

The carrier gas or mobile phase in GC is an essential, but limiting, facet in separations. Carrier gas is the means to move constituents of a sample through the column and yet the choice of possible gases is restricted. Moreover, the carrier gas has properties that sometimes can complicate an analysis. Unlike liquid chromatography (where a wide selection of mobile phase compositions may be possible),

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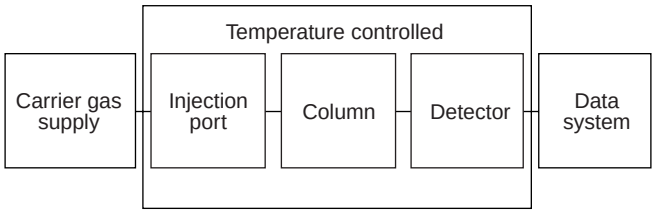

Figure 1 Block diagram of gas chromatograph.

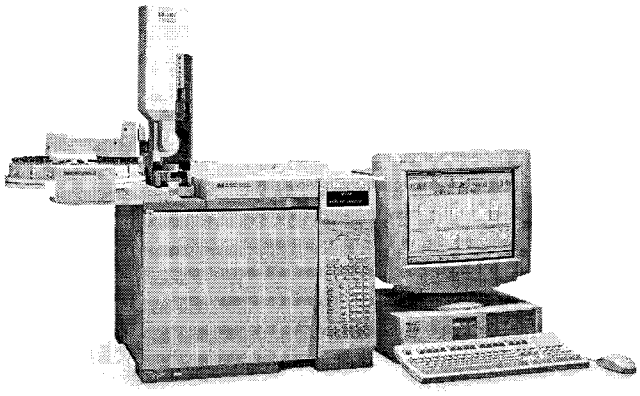

Figure 2 Photograph of modern gas chromatograph.

very little can be gained in separations through altering the mobile phase composition to influence the partition coefficient (k) or separation factor (α) in GC.

2.1 Selection of Gases

The choice of a practical carrier gas is simple: nitrogen or helium. Air may be used as a carrier gas under certain conditions with portable or on-site chromatographs but this is uncommon with laboratory-scale instruments. The choice of nitrogen or helium is made, in part, on the principles of separation and, in part, on economics: ~$20 for a nitrogen cylinder versus ~$50 for a helium cylinder. However, the selection is more complex than the prices of gas cylinders alone. Column efficiency in GC contains a term for contributions to longitudinal broadening in the carrier gas and this is given by the D_g term in the van Deemter equation.[3] This term is proportional to the square root of molar mass for the carrier gas, and nitrogen or argon would be preferred over helium based on D_g only. This effect can be seen in Figure 3, where nitrogen provides better performance than helium and has the lower contribution to plate height. However, the shape of the curve for height equivalent to a theoretical plate (HETP) versus flow rate (as linear velocity) for helium shows a reasonably good efficiency at high flow rates.[1,4] (HETP is equal to L/N, where L is the column length and N is the number of theoretical

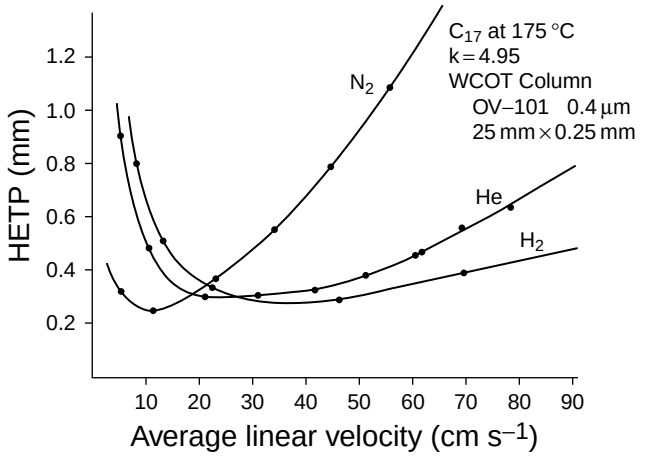

Figure 3 Plot of separation efficiency versus column flow rate.

plates in a column.) In contrast, the van Deemter curve for nitrogen is comparatively narrow. Consequently, a GC separation using nitrogen at 10 cm s^{-1} can be accomplished with comparable separating efficiency using helium at 50–60 cm s^{-1}. The practical consequence of this is that costs for using helium, on a per sample basis, might be lower than those for nitrogen when the speed of analysis is factored into the calculations.

2.2 Control of Flow

One difficulty in GC is the compressibility of the carrier gas and subsequent influence on separating performance. This was recognized in the first paper on GC where correction factors for gas flow rates were described.[5] The implications for isothermal methods are significant but will be critical with temperature programmed GC when column temperatures may span 200 °C or more.[6,7] When temperature is increased for a column with constant pressure on the inlet, the average flow rate in the column will decrease owing to increased viscosity of the gas mobile phase in a proportional but nonlinear manner. Under such conditions, flow rates may slow at high temperature and both separation speed and efficiency may suffer. Flow may be kept constant through mass flow meters that have inlet and outlet orifices, adjustable based upon pressure differences.[2] Constant flow can be delivered across a range of pressure drops that may arise due to changes in temperature but cannot compensate for changes in barometric pressure. An advance in instrumentation during the past decade has been the commercialization of flow programming so that flows may be made highly reproducible.

2.3 Gas Sources and Purity

A common gas source for nitrogen or helium is the pressurized cylinder or bottled gas supply, readily supplied

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as a steel tank with a two-stage pressure regulator. This is still a common gas source though gas generators for nitrogen (air and hydrogen too) can be commercially competitive with bottled gas and have advantages in safety.

Regardless of the gas source, special attention must be given to the purity of tubing used to connect the source and the gas chromatograph and to impurities in the gas supply. Most columns do not tolerate moisture and oxygen well when operated at temperatures over 100 °C. Best results for column longevity and chromatographic reproducibility occur when the carrier gas is cleaned over molecular sieve beds (to reduce moisture). In addition, specialized traps can be purchased to reduce or remove hydrocarbons and oxygen in the carrier gas.

3 SAMPLE INLETS

The chromatographic process begins when sample is introduced into the column, ideally without disrupting flows in the column. The chromatographic results will be reproducible inasmuch as this is accomplished with a minimum of change in pressure or flow of the carrier gas or mobile phase. Also, the injection step establishes the initial (and best possible) peak width for the GC measurement. Thus, delivery of sample into the column should be controlled, reproducible, and rapid.

3.1 Syringes and Switching Valves

A common method for placing samples on a GC column is to use the microliter syringe with a needle to penetrate a plastic membrane. In this method a gas-tight seal is maintained and sample is deposited into a heated zone. If liquid or solid, sample is volatilized and swept to the column and this can be accomplished by manual injections in ~1 s. Syringe injection is a convenient and generally effective method though the thermoplastic septum develops leaks after repeated injections. Fatigue of the plastic septum limits the number of injections to ~30 before the septum must be replaced. A second difficulty arises with impurities from off-gassing or decomposition of the septum and these are seen as so-called ghost peaks or peaks in control blanks.[8]

Advances with capillary columns introduced unprecedented precision and accuracy to GC measurements and limitations with syringes became apparent.[9] Discrimination toward high boiling point components was seen with syringe injections and techniques to remedy the failings have been developed.[9] Sometimes thermal volatilization may lead to decomposition of samples so efforts to remove the discrimination and decomposition motivated the use of so-called on-column injections where sample is deposited directly from the syringe into the column. Another complication with syringe injections is the introduction of particulate and reactive materials into columns. Protection is afforded by precolumns. Further information on syringe injections and the range of options for injection methods can be found in excellent reference sources.[10,11]

Gas samples can be injected into the column using gas-tight syringes or using rotary gas switching valves that offer enormous flexibility for GC instruments.[12] Precision gas switching valves allow a gas sample to be measured with a precise volume and introduced into carrier gas flow without interrupting column flow. Sample is loaded into a loop and then, with a change in the valve position, is swept into the column under flow of the gas source. Heated switching valves such as those made by VICI, Inc.[13] are also useful in the analysis of sorbent traps. When traps are heated and switched in-series with the analytical column, constituents will be thermally desorbed for GC separations. Switching valves can be automated via electronic actuators and can be incorporated into purge-and-trap methods that are useful for characterizing aqueous samples for volatile organic constituents.

3.2 Pyrolysis

Another inlet option which is now routine in certain specific applications of material sciences is that of sample pyrolysis where solid samples are rapidly heated to a point of thermal decomposition in a reproducible manner. At temperatures in excess of 600 °C, substances such as natural or synthetic polymers thermally decompose to small molecular weight, stable substances that provide a chromatographic profile which is unique to certain materials. Such an injector enlarges the application of GC to solid samples that would not normally be considered suitable for GC characterization, and pyrolysis methods have become standardized for some applications such as assaying plastics. A journal now exists for analytical applications of pyrolysis, the *Journal of Analytical and Applied Pyrolysis*. Attachments to inlets are commercially available and serve to extend GC in forensic and industrial applications, as shown in Table 1.

3.3 Other Topics

In purge-and-trap methods, a small amount of water is treated vigorously with an inert gas flow sufficient to sweep volatile organic compounds (VOCs) into the gas phase from the water phase. The gas flow, including the VOCs, is passed through an adsorbent trap where VOCs are retained and concentrated while water vapor and

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Table 1 Examples of applications of pyrolysis GC

Example	Ref.
Lignin by pyrolysis methylation	14
Synthetic polymers	
Fast GC	15
Bibliography	16
Rosin glycerin esters in paper	17
Chlorinated polyethylene structure	18
Coating materials: bibliography	19
Proteinaceous binders in paints	20

Table 2 Examples of applications of SPME methods with GC analyses

Example	Ref.
Dimethyl sulfide in beer	23
Diacetyl in wine	24
Organochlorine compounds in water	25
Wine headspace compounds	26
Ecstasy and amphetamine in confiscated samples	27
Parathion in biological samples	28
Trimethylamine in urine	29
Volatile compounds in sunflower oil	30

gas are unretained and released to vent. After a short time of purging, the trap is placed in series with the gas chromatograph via a gas switching valve and VOCs are transferred to the analytical column. This is done through a rapid increase in temperature of the sorbent trap and the vapors are carried to the analytical column under carrier gas flow. Purge-and-trap methods are the basis for an expansive use of GC for monitoring the VOCs at ppb levels in aqueous samples as specified[21,22] by the United States Environmental Protection Agency (USEPA). Such instrumentation and methods are available today in commercial instrumentation and can be fully automated. An advantage in this method of sample preparation is that nonvolatile constituents, which could potentially foul the column, remain in the sample container and do not enter the column.

Another adaptation of GC for trace organic analysis was the development of solid-phase microextraction (SPME) with a syringe design. In this, a stationary phase is bonded on a silica fiber that can be introduced to a gas or aqueous sample. Organic chemicals partition into the coated fiber during a time of contact and the fiber is withdrawn into a syringe needle. In an injection port the fiber is pushed from the protection of the needle and sample is thermally desorbed into the GC column. This method has advantages of convenience and simplicity and the growth of use of SPME injections has been dramatic during the 1990s. A list of some representative applications is given in Table 2.

4 OVENS

4.1 Conventional Designs

Liquids or solids must be converted to vapor state and maintained as a vapor throughout the GC separation. Therefore, most gas chromatographs are equipped with ovens to keep the column at temperatures from 40 to 350 °C. Exceptions are those chromatographs that are used in separating simple gases such as light hydrocarbons or permanent gases. Early gas chromatographs were equipped with isothermal ovens. Today, temperature-programmed ovens allow separations of chemicals spanning a range of vapor pressures in a single analysis.

Conventional ovens, unchanged in decades, consist of a resistive wire coil that radiates into the inner volume of the oven. Heat from the resistive wire source is spread, ideally in an even manner, throughout the oven volume using a fan attached to an electric motor. A thermistor or thermocouple inside the oven is part of regulating the oven temperature via the amount of heat released by the heating element. This is controlled by the power delivered to the element and a feedback circuit to control and program the oven temperature. Efforts to create isothermal conditions, i.e. no thermal gradients inside the oven volume, are essential for reproducible chromatography and are criteria in evaluating good oven designs. Gradients in excess of a few degrees between various regions of an oven are practical in the best of oven designs and can be more than a few degrees in poorly designed ovens. One of the only systematic evaluations of GC ovens was given by Welsh[31] and his discussion provides measures for characterizing GC ovens.

4.2 Other Designs for Control of Column Temperature

Several alternatives to conventional ovens have been devised and may be especially helpful for short columns or instances where little space is available for a bulky, heated air oven. Two approaches have been used and include small thermal ovens[32] and innovative column heating arrangements.[33] Column heating based on resistive heating is compact, uses minimal power, and can decrease analysis times.[34] These methods are based upon application of heat directly to the column or a base upon which the column is crafted or attached. The approach is unlikely to become a laboratory standard but is being explored for use in miniature or portable gas chromatographs.[35]

5 DETECTORS AND DATA SYSTEMS

The subject of detectors in GC is a pivotal theme since the separation processes will have been wasted if the analyte

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cannot be detected. Excellent primers on GC detectors are available[36,37] and any general text on instrumental analysis will have introductory material on the common detectors. A biennial review contains an extensive section on developments of GC detectors and can serve as a guide to primary literature.[38]

5.1 Detectors

Effluent from the column enters a detector where the composition of the carrier gas stream is characterized through one of several possible chemical or physical properties of molecules. The mainstays in GC have been the flame ionization detector (FID), the thermal conductivity detector (TCD) and the electron capture detector (ECD). Other commercially available detectors include the photoionization detector (PID), the nitrogen–phosphorus detector and the atomic emission detector, though these have been less prevalent historically than the FID, TCD, and ECD. Other detectors have been introduced through the years but have never become widely used in GC methods. The FID relies upon the formation of gaseous ions from organic molecules combusted in a hydrogen–air flame; the TCD is based upon changes in the heat absorbing properties of the gas effluent when the carrier gas is altered with analyte; the ECD response is governed by the ability of some molecules to attract and remove thermalized electrons. Despite long-standing conventions for the design and operation of these detectors, advances still occur.

Examples of evolutionary changes include the small FID designs[39] and designs where gas mixing is arranged to provide optimum response.[40–42] A recurring theme in advances in ECD has been a nonradioactive alternative to the normal source, 10 mCi of ^{63}Ni. Despite promising discoveries, the radioactive source is still the favored choice. The applications of ECD illustrate the advantages of selective detectors where analyte can be found in the presence of potentially interfering matrix.

Examples where the ECD was chosen to detect a specific chemical family over interfering backgrounds include: halocarbons in air for oceanographic tracer studies;[43] chlorobutanol in mouse tissues and fluids;[44] organochlorine compounds in milk products;[45] pesticides and other organochlorides in water;[46] organochlorine pesticides in edible oils and fats.[47]

In the last two decades, inexpensive mass spectrometers or mass-selective detectors (MSDs) have dramatically transformed the practice of GC. Once the purview of laboratories able to sustain the high cost of mass spectrometers and the high level of maintenance, instrument manufacturers made mass spectrometers both robust and inexpensive. This development, when combined with the appreciation that analytical confidence is highest with a mass spectrometer as the detector, has resulted in a near general availability of gas chromatography/mass spectrometry (GC/MS) instrumentation. In a GC/MS analysis, a mass spectrum can be obtained continuously at fixed intervals of ~0.1 s throughout the analysis. Consequently, a mass spectrum can be obtained for each chromatographic peak and the shoulders and baselines in the chromatogram. No other detector can provide the richness of information available in such results. Detection limits can be enhanced through the use of single ion monitoring where the mass spectrometer is used for detecting the intensity of one or a few ions. This can provide the specificity of a mass spectral pattern for response without losses in detection limit associated with scanning over unused m/z space. One revolution in the past decade has been the application of powerful desktop computers to control instrumentation and especially to control data acquisition and handling.

5.2 Data Systems

At a fundamental level, acquisition of chromatographic results has been little changed since the early days of GC, though the digital revolution has meant that strip chart recorders, once the mainstay of collecting chromatograms, cannot be found today and only electronic recording-integrators or microcomputers are used. Signal from the detector amplifier is digitized and stored to disk allowing enormous convenience in retrieving and replaying results. This means that peak retention times, peak areas, etc. are automatically reported and have been since the mid-1970s. In addition, software allows the results to be displayed in an automated manner, i.e. reports can be generated according to standard reporting formats. All this can be economically integrated into the total instrument control and management through computers and is an option on all chromatographs and a standard feature on most instruments.

6 MINIATURIZED, HIGH-SPEED, AND PORTABLE GAS CHROMATOGRAPHS

One area of GC that has shown vibrancy with advances during the 1990s is that of small, fast, and portable GC instruments. Though process gas chromatographs were amongst the first sophisticated analyzers placed into industrial on-site measurements, the subject has taken new significance following the burgeoning environmental movement. Making measurements where a sample is located rather than relocating samples to a centralized laboratory underlies this trend. Recently, a new journal

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has appeared to support these efforts, *Field Analytical Chemistry and Technology*, which includes portable GC advances.

In high-speed GC, retention times can be pushed under a few minutes or seconds with short, narrow bore columns or high flow rates. Part of the challenge in fast GC is the compressibility of the carrier gas and the necessary speed (low time constants) for subcomponents such as injectors and detectors for high-speed separations.

6.1 Instrument Designs

An example of the size possible for small gas chromatographs is an ultimate miniature gas chromatograph created using silicon micromachining and integrated circuit processing techniques.[48] This GC analyzer contains a 0.9 m long × 300 μm wide × 10 μm high rectangular column coated with a 0.2-μm thick liquid phase. The injector is a 10-μm-long sampling loop with the same cross-section as the column. Dual detectors based upon a coated chemiresistor and on thermal conductivity are used. The complete system is packaged in less than 23 cm^2 and is 2.5 mm high. Although limited in scope to the detection of ammonia and nitrogen dioxide, this miniature chromatograph offers exciting possibilities for future field instruments.

One trade-off for high-speed GC is the loss of capacity due to the smaller diameter and shorter columns. Application of packed capillary columns in high-speed GC has been shown to improve capacity and selectivity[49] while obtaining high-speed separation for light hydrocarbons.[50] An alternative is the multicapillary column which improved capacity while maintaining the efficiency obtained with small internal diameter columns.[51]

Injection techniques for high-speed GC must provide narrow bandwidths due to fast analysis time requirements without compromises in resolution.[52] One means to accomplish this is through cryogenic inlets which provide narrow bandwidths[53] and in some instances injection times can be shorter than 10 ms.[54]

6.2 Detectors

Mass spectrometers might be considered ideal detectors and adaptations to fieldable gas chromatographs have been made. One design is the size of a standard size suitcase, weighs ~31 kg and uses ~600 W under peak loads.[55] Naturally, field portability is a goal and man-portability has been demonstrated.[56] A cart-portable unit has been described and was capable of screening 1000 samples an hour at a speed of 20–32 km an hour.[57] Portable GC/MS instruments were also used for industrial hygiene applications.[58] As the need for field measurements increases and mass spectrometry (MS) becomes miniaturized, the number of field portable GC/MS systems can be expected to increase dramatically.

Another sophisticated detector for GC is the ion mobility spectrometer and recently it too has been coupled to gas chromatographs for field analysis.[59,60] The utility of this hand-held gas chromatograph/ion mobility spectrometer analyzer was demonstrated by rapid screening (~20 s) with chemical warfare agents such as dimethyl methylphosphonate.[61] One of the most impressive demonstrations was that from the US Army where a portable pyrolysis-gas chromatograph/ion mobility spectrometer was developed for screening airborne pathogens. This unit is shown in Figure 4.

This part of GC instrumentation may be expected to undergo continued development as interest in speed of analysis pushes dimensions and hardware. Therefore, it represents one of the few areas of growth in GC

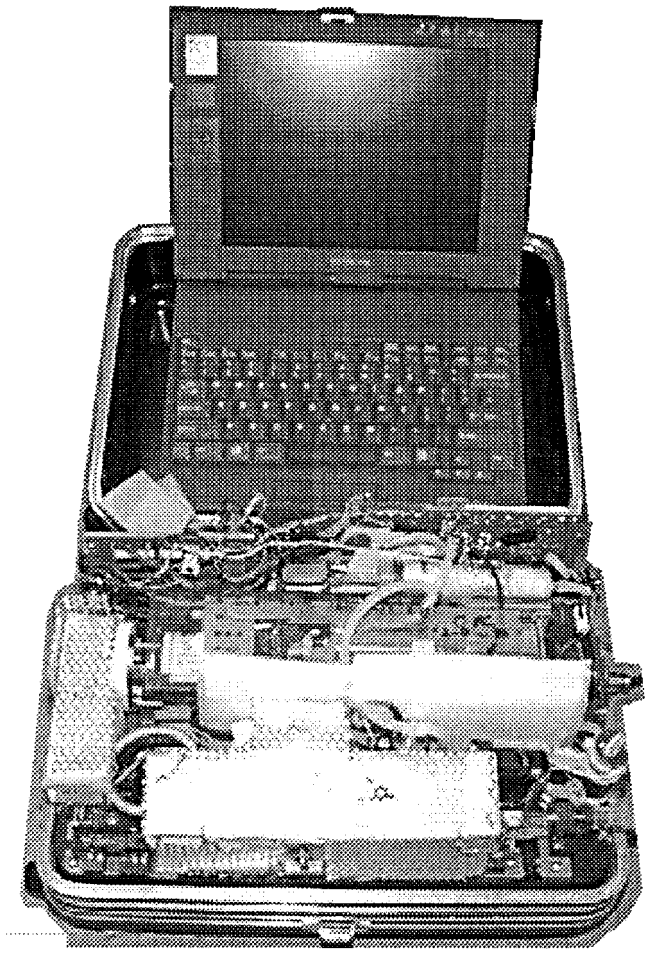

Figure 4 Gas chromatograph/ion mobility spectrometer for bacterial analysis.

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instrumentation that can be recognized in the absence of a revolutionary change in GC methods.

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LIST OF SYMBOLS

α	Separation factor
D_g	Diffusivity of solute in carrier gas
k	Partition coefficient

ABBREVIATIONS AND ACRONYMS

ECD	Electron Capture Detector
FID	Flame Ionization Detector
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
HETP	Height Equivalent to a Theoretical Plate
MS	Mass Spectrometry
MSD	Mass-selective Detector
PID	Photoionization Detector
SPME	Solid-phase Microextraction
TCD	Thermal Conductivity Detector
USEPA	United States Environmental Protection Agency
VOC	Volatile Organic Compound

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