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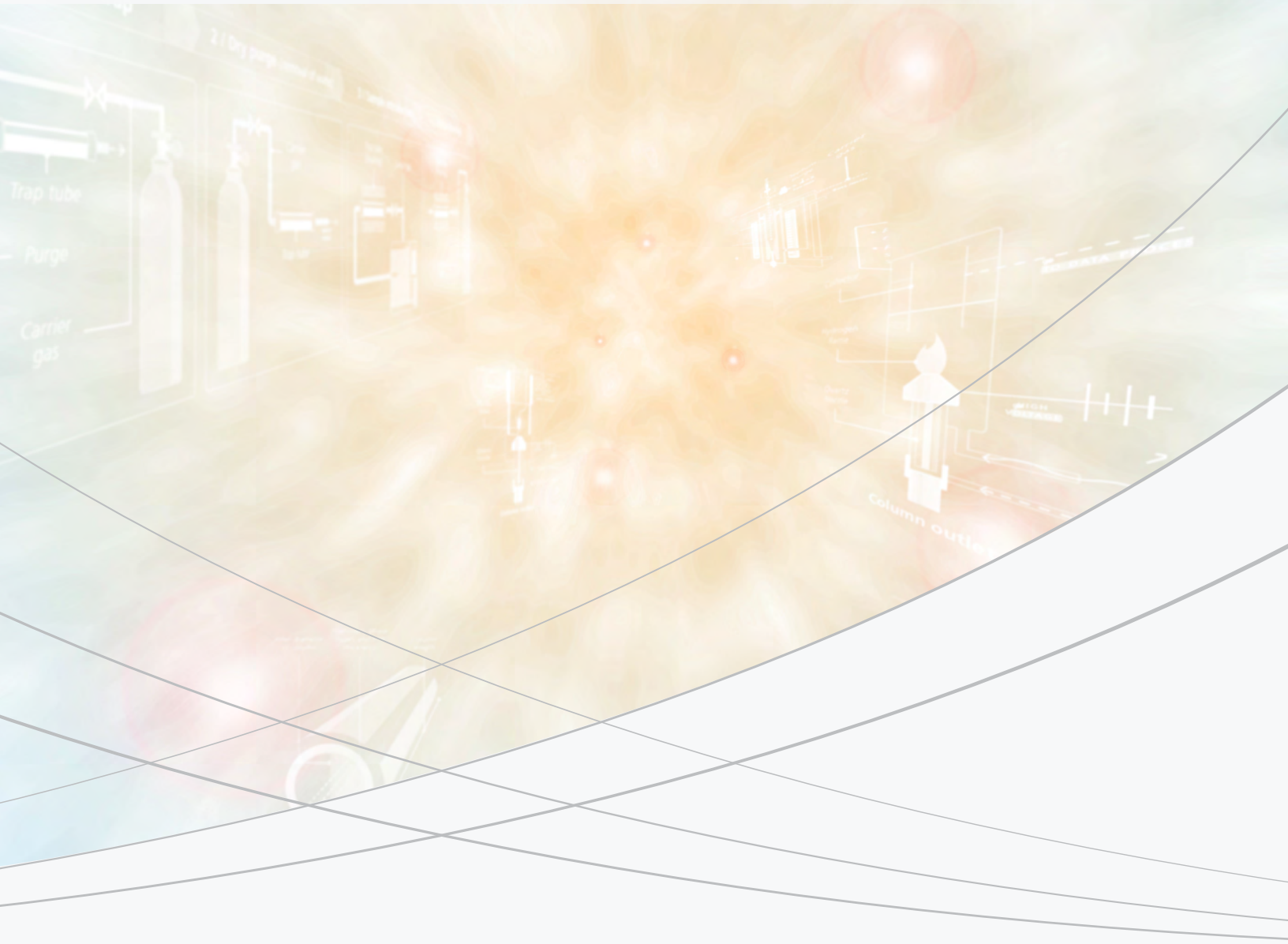
Basics & Fundamentals
Gas Chromatography



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Introduction

Gas chromatography (GC) is an analytical methodology, which was devised by Nobel Laureate, Martin, *et al*. in 1952. More than 60 years after the award, GC systems are widely commercialized and used in various industries, capable of both of quantitation and qualification. GC is applicable for many types of analysis in the markets such as residual solvent analysis in pharmaceuticals, residual pesticides analysis in food safety, trace level analysis for environmental as well as petrochemical and fine chemical industry.

This article describes the basics and fundamentals of GC with tips on the instrumental operations in laboratory use. There may be other more advanced applications which require GC customization (aka Process GC or System GC), which will not be covered in this primer.

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1. GC Structure and Fundamentals

Gas chromatograph is an analytical instrument used to analyze the different components in a sample. An analytical method using a gas chromatograph is called gas chromatography (GC).

Table 1: Terminologies and Definitions of Gas Chromatography

Term	Definition
Chromatography	Method for Separation
Chromatograph	Instrument for Chromatography
Chromatogram	Data of Chromatography

1-1. Basic instrumentation of GC

As shown in Figure 1, the GC consists of a flow control section, a sample injection port, a column, a column oven, and a detector in which is connected to a data processor. Carrier gases such as helium (He), nitrogen (N_2) or hydrogen (H_2) are preferred to be supplied at a constant flow rate to the sample to the injection port. A separation tube called a column is connected between the sample injection port and the detector, all three parts are maintained at an appropriate temperature. The sample injected into the sample injection port instantaneously vaporizes and flows into the column with the carrier gas. In the column, a liquid stationary phase (for example, silicone polymers) is chemically bonded or coated, the vaporized sample is repeatedly dissolved and vaporized in the liquid stationary phase and travels downstream with the carrier gas.

Since the process of dissolving and vaporizing the sample in the stationary phase depends on the physicochemical properties such as the boiling point and the nature of the column, the time of dissolving in the liquid phase and the time of vaporizing will be different for each compound. Therefore, even when mixed components are injected, the time for the components to arrive at the column exit is different, and separation can be detected. The column exit connects to a detector and when substances other than the carrier gas are eluted from the column, the detector converts them into electrical signals which are amplified and sent to a data processor. By analyzing the electric signal of the detector on the data processor, the GC will be able to identify the sample and determine its quantity. Under certain conditions, the time to reach the detector (retention time) is the same after injection of the compound. By injecting the standard sample and the unknown sample, and comparing the retention times, quantitation can be done by comparing the sizes of their peaks.

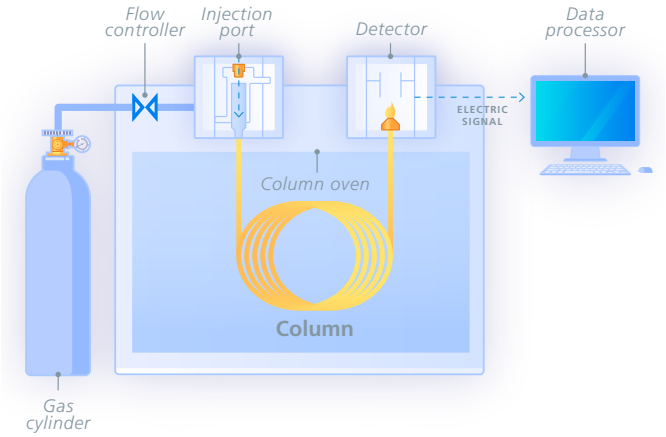

Figure 1: Typical GC Configuration

1-2. Applications of GC

Shimadzu GC Application Data Sheet

Table 2: Some Industrial Applications of Gas Chromatography

https://www.shimadzu.com/an/gc-datasheet.html

Industry	Type of Analysis
Pharmaceutical	Residual solvent analysis
Food and beverages	Component analysis, food safety analysis, halal analysis of alcohol
Environmental	Air, water, soil
Petrochemicals	Simulated distillation, component analysis
Chemicals	Material, polymer, additive, gas purity analysis, gas emission in automotives
Energy and gas	Artificial photosynthesis research

2. Sample Injection Methods

There are many different injection methods: split injection, splitless injection, direct injection, on-column injection.

2-1. Split Injection Method

Split injection is the most popular and versatile method in capillary GC analysis. Split injection can be applied to many types of analysis, and whilst it may be less sensitive, the resolution of the chromatogram is not affected.

Figure 2 shows the flow diagram of split injection. The sample is introduced into the inlet and is vaporized, moving downstream. Large portions of the vaporized sample have to be exhausted before the sample is separated in the column and detected. The split line has a role to protect the column from any sample overload and increase the moving velocity of the component in the injector port.

The split ratio indicates "the ratio between the column flow rate and the split flow rate" and indicates the approximate branching ratio of the injected sample. When the split ratio is high (for example 1:400), the rate of the injected sample being introduced into the

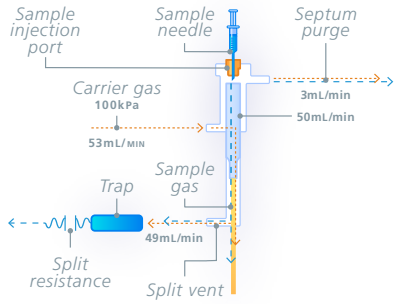

Figure 2: Schematics of a Split Injector Port

column decreases as well as the sensitivity. On the other hand, when the split ratio is low (for example 1:30), the rate of introduction into the column increases as the sensitivity increases. The commonly used split ratio is 1:50 to 1:100 in the column with the inner diameter of 0.25 mm to 0.32 mm but considering the required sensitivity and the load amount on the column, the analysis is performed using the optimum split ratio.

Glass inserts used in the split method are filled with fillers such as glass wool which have been deactivated. Quantity values and reproducibility may differ if wool quality, quantity, and the packing position are different. Since most manufacturers provide the specific requirements for wool and position, it is better to adhere

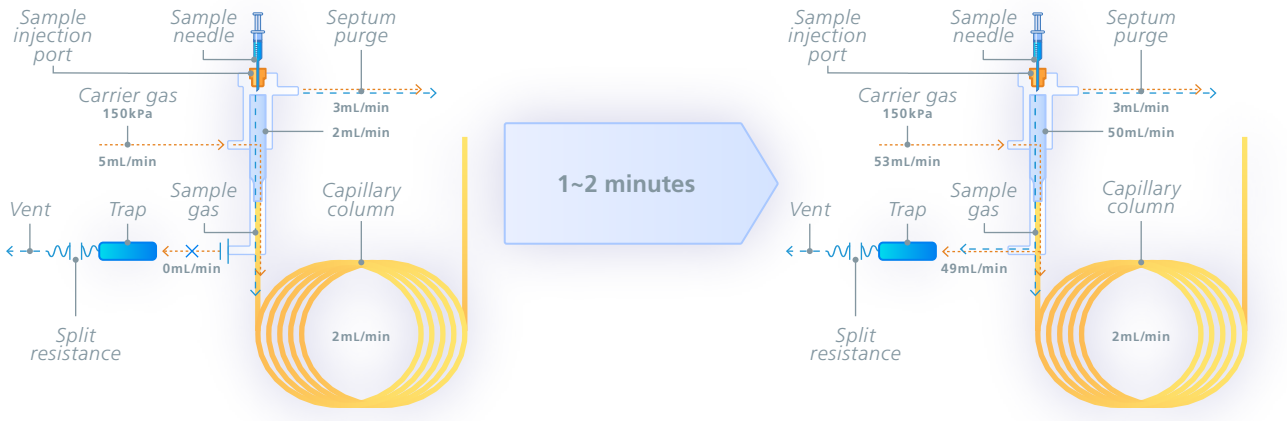

Figure 3: Schematic of a Splitless Injector Port

to this. The split method is a relatively simple sample injection method. Higher quality data can be obtained by understanding and analyzing features well.

2-2. Splitless Injection Method

The inlet for splitless injection can be shared with split injection. The splitless injection method is suitable for higher boiling temperature compounds and it is used to measure lower concentrations in environmental and residual pesticides field. Figure 3 shows the conceptual diagram of the splitless injection method. It is similar to the split injection flow diagram, because when the sampling time is set to zero, the splitless injector essentially operates like a split injector. The sampling time is the amount of time set to direct samples into the column, before the split vent is open.

The operation during sample injection and the flow of carrier gas will be described with reference to Figure 3. Before injecting the sample, keep the column initial temperature at 50 to 60 °C and close the split line as shown on the left side of Figure 3. When the sample is injected into the inlet, it will be vaporized. The vaporized sample gradually moves to the column due to the carrier gas. At this time, the component with relatively high boiling point is concentrated in a narrow band at the tip end portion of the column because the column initial temperature is low. It takes 1 to 2 minutes for most of the vaporized sample to be introduced into the column, and this is the typical set amount of sampling time in a splitless injection. Thereafter, as shown in the right diagram of Figure 3, the split line is opened, the solvent and the sample at the inlet and the column are removed, the temperature is raised, and the concentrated component is separated at the tip end portion of the column to detect.

The splitless method is an analytical method that is suitable for relatively high boiling point compounds with a low analyte concentration. There are some caveats but it is widely used in environmental and residual pesticide analysis as a method that can easily achieve high sensitivity.

2-3. Direct Injection Method

The majority of the sample is introduced into the capillary column. The inner diameter of the capillary column is 0.45 mm or more, which is called wide bore. It is recommended that the column length be 25 m or more. Short columns of 15 m or less are often difficult to use due to low set pressure.

Figure 4 shows an example of a structural view of an injection port dedicated to the wide bore column. In order to suppress sample retention in the glass insert and the wide bore column connection part, a part of the carrier gas is supplied as a purge gas in the column connection part, or a taper is formed in the lower part of the insert so that the wide bore column closely adheres to the bottom part of the insert. When using a septum purge type total volume injection port, it can be used with a carrier gas flow rate near the optimum flow rate (about 5 mL /min of He) for a wide bore column.

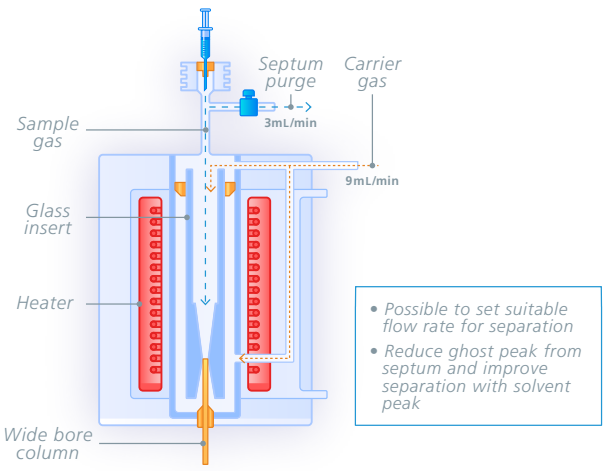

Figure 4: Schematic of Direct Injection Port (with only septum purge flow)

The direct injection method is an excellent analytical technique which enables simple capillary analysis by simple inlet port structure.

2-4. Cold Injection Method

a. Cold on-column caps Injection (Cold OCI)

Inlet temperature is kept below the boiling point of the sample solvent and the tip of the microsyringe is inserted directly into the tip of the capillary column for sample injection. Thereafter, by raising the temperature of the inlet and column, the sample is gradually vaporized directly inside the capillary column. The tip of the capillary column corresponds to the vaporization chamber.

Cold OCI is suitable for analyzing compounds that are unstable to heat (easy to decompose). Due to there being no compositional change of samples, it is an analysis method with high accuracy for measures such as area value reproducibility. Samples with low concentrations (less than about 200 ppm per component) are also suitable.

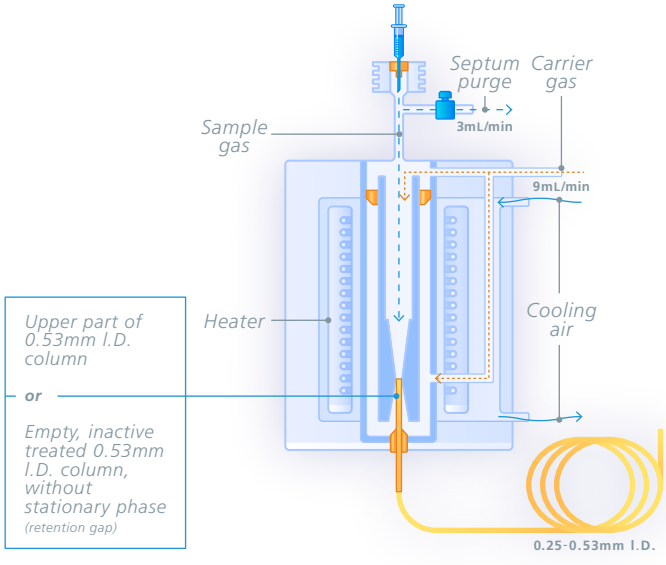

Figure 5: Schematic of a Cold On-Column Injection

However, since the sample is injected directly into the column, the column is liable to be contaminated.

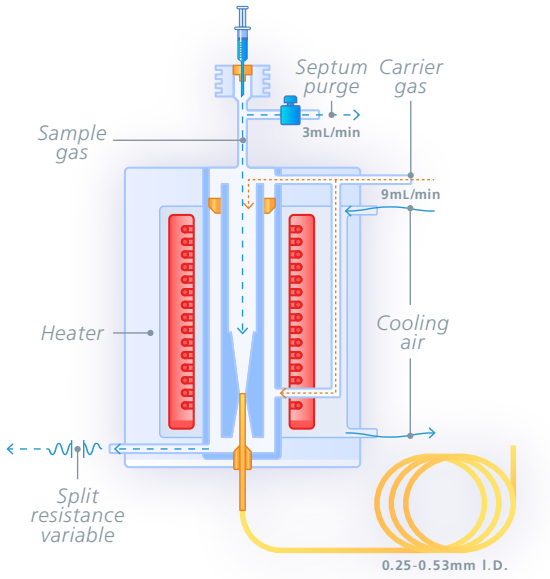

Figure 6: Schematic of a Programmed Temperature Vaporizer

b. PTV Injection System (Programmable Temperature Vaporizer)

At the point of sample injection, the injection port is set to a low temperature, typically below the boiling point of the solvent. Thereafter the PVT is rapidly heated to vaporize the entire sample, along with the solvent. This method is suitable for analyzing compounds that are unstable to heat (easy to decompose) with little change in composition due to the warming of the remaining components at the tip of the syringe needle.

Unlike Cold On-Column Injection analysis, glass inserts can be used in a PTV. Additionally, both split and splitless modes can be applied onto the PTV. As a result, PTV can cope with high and low concentration samples. With the right setting, PTV can prevent nonvolatile contaminants from entering the analytical column altogether.

3. Separation Columns

There are two main types of separation column used in GC available on the market: capillary and packed columns.

A capillary column typically has 0.1-0.53 mm internal diameter and 10-100 m length with very high resolution. The material is fused silica and the inside wall is chemically bonded with the liquid phase. The outside is coated by polyimide resin to increase the intensity. The column with 0.25 mm internal diameter and 30 m length is frequently used.

The GC user chooses the column based on certain attributes such as; the target compounds, the number of components and the instrument configuration. A smaller diameter and longer column is suitable if higher resolution is needed, while a larger diameter column can be used if higher resolution is unnecessary.

Table 3: Polarity and Liquid Phase of Capillary Columns

Polarity	Example of Column	Typical Liquid Phase
Non-polar		Squalane
	SH-Rtx-1	100% Dimethylpolysiloxane
Low-polar	SH-Rtx-5	5% Diphenyl 95% Dimethylpolysiloxane
	SH-Rtx-1301, 624	6% Cyanopropylphenyl 94% Dimethylpolysiloxane
Mid-polar	SH-Rtx-1701	4% Cyanopropylphenyl 86% Dimethylpolysiloxane
	SH-Rtx-17	50% Phenyl 50% Methylpolysiloxane
High-polar	SH-Rtx-200	Trifluoropropylmethylpolysiloxane
	SH-Rtx-Wax	Polyethyleneglycol
	BPX-90	90% Bis-cyanopropyl 10% Cyanopropylphenylpolysiloxane

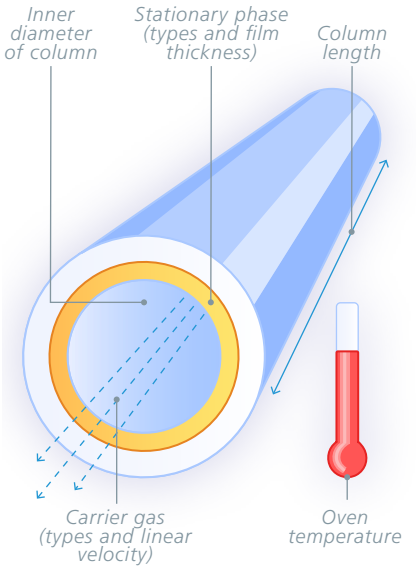

Figure 7: Factors Which Affect Separation in a GC Column

4. Carrier Gas

4-1. Choice of Carrier Gas

The carrier gas should be an inert gas, carrying the sample but not interacting with the target compounds. Such examples are He, N_2, H_2, and Ar, of which, He and N_2 are the most commonly used. In capillary columns, He is preferred due to its ability to maintain the separating resolution at high linear velocity (the speed at which sample travels through the column).

Table 4: Advantages and Disadvantages of Carrier Gases

Carrier Gas	Advantages	Disadvantages
Helium	• Safe • Relatively wide optimum linear velocity range	• Expensive
Nitrogen	• Cheap • Safe	• Optimum linear velocity range is narrow and slow • Long analysis time

Carrier gas always flows into the detector, therefore it is necessary to use one with a high purity (99.995 % or higher). Carrier gases with high purity can suppress baseline noise.

Measures and Proposals to Reduce Helium Gas Consumption are found in the link below: https://www.shimadzu.com/an/gc/eco/gas.html

4-2. Flow Control Modes

When GC was first developed, the primary mechanism to direct the flow within the system was by controlling the pressure. For convenience, the initial pressure (sometimes known as head pressure) is fixed and all the resultant methods created are based on constant pressure mode. There is, however, a major flaw: peak broadening.

There are three different flow control modes that are commonly employed. We can look directly into the associated units to compare the three control modes:

- constant pressure mode: **Pa** (pressure)
- constant flow mode: **cm³/s** (volume per second)
- constant linear velocity mode: **cm/s** (distance per second)

In constant pressure mode, the head pressure is fixed. As the sample travels further and further from the injector port, it experiences less force, and hence it slows down. This results in peak broadening as the retention time increases. Such an impact is more significant in capillary columns, in which the column length is much longer than in packed columns. Over time, GC methods have shifted from this mode in favor of significant improvements.

Next, we look into the very similar constant flow and linear velocity modes. They both overcome the weakness of constant pressure mode, correcting the volume passing a column or distance travelled down to the "per second" accuracy level. However, columns expand at a higher temperature (typical in GC oven programming) and the difference between these two modes become significant.

In an ideal situation, when a tube has a smaller diameter, the same volume occupies a greater length. For the same volume to pass points 1 and 2 in a given time (Figure 8), the speed must be greater at point 2.

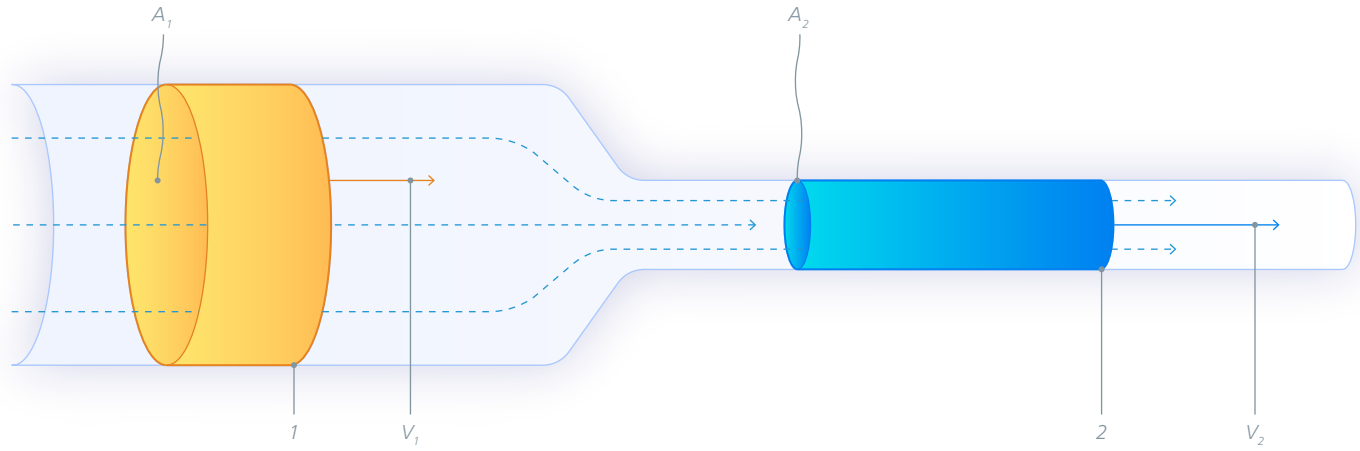

Figure 8: Fluid Flows Through a Narrower Column Faster: The same volume of fluid occupies a different cross-sectional areas (A) at points 1 and 2, where the column size is different. This causes a different flow rate, and thus a different linear velocity (v).

The process is exactly reversible; if the fluid flows in the opposite direction, its speed will decrease when the tube widens. This "speed" is what we know as the linear velocity. During analysis, with the heating program of the GC oven, capillary columns expands gradually. As a result, if the constant flow mode is used, linear velocity will slow down progressively, affecting the separation efficiency.

While not illustrated in any figure of this article, there is another situation where the constant linear velocity mode comes in handy. When the tail pressure is lower (i.e. vacuum), the flow will be faster than it would be with a higher tail pressure (i.e. atmospheric pressure). As a result, the retention time between a GC with an atmospheric detector (e.g. a flame ion detector (FID)) and a GC with a detector in a vacuum state (e.g. a mass spectrometer (MS)) cannot be matched interchangeably. The issue can be resolved, however, if the constant linear velocity mode is employed; in this mode, carrier gas flows through the column regardless of the detector pressure.

In GC instrumentation, it is easier to control the pressure (you only need a regulator to do that). To control the flow, you then need to implement some correction factors. A feedback mechanism is required to adjust the pressure regularly so that the volume passing through a column is maintained. This automatic pressure control mechanism is called "electronic pneumatic control". To control the linear velocity, an additional correction factor for the cross-sectional area is needed. The electronics need a more sophisticated calculation algorithm.

Within one instrument, using constant linear velocity mode can maintain the optimal resolution efficiency of chromatographic separation. Across multiple instruments, constant linear velocity ensures the reproducibility of results. This is true even for reproducibility between vacuum detectors and ambient pressure detectors. In other words, constant linear velocity mode can be extremely beneficial for its method transferability, e.g. between R&D and QC departments of an organization.

5. Detectors

Table 5: Features of GC Detectors. This table serve as a rough indication, it may be different depending on the compound chemical structure and analytical condition

Detector			Example of Detectable Compound	Example of Minimum Detectable Amount*
Universal Detector	Thermal Conductivity Detector	TCD	All compounds except for carrier gas	10 ppm (10 ng)
	Flame Ionization Detector	FID	Organic compounds	0.1 ppm (0.1 ng)
	Barrier Discharge Ionization Detector	BID	All compounds except for He and Ne	0.07 ppm (0.07 ng)
	Mass Spectrometer	MS	Ionized molecule	10 ppm (10 ng) in Scan mode 0.5 ppm (0.5 ng) in SIM mode 10 ppb (10 pg) in MRM mode
Selective High-sensitivity Detector	Electron Capture Detector	ECD	Organic Halogen compounds Organic mercury compounds	0.01 ppb (0.01 pg)
	Flame Photometric Detector	FPD	Sulfur compounds Organic phosphorus compounds Organic tin compounds	10 ppb (10 pg)
	Flame Thermionic Detector	FTD (NPD)	Organic phosphorus compounds Organic nitrogen compounds	0.1 ppb (0.1 pg) 1 ppb (1 pg)
	Sulfur Chemiluminescence Detector	SCD	Sulfur compounds	1 ppb (1 pg)

Table 6. The Compounds in the TCD and the Unique Conductivity Constant

Compounds	Thermal Conductivity Constant (10^{-6} cal/s·cm·°C)
He	408
H_2	547 (Very High)
N_2	73
Ar	52
O_2	76
H_2O	60
Ethane	77
Methanol	52
Acetone	40
Chloroform	24

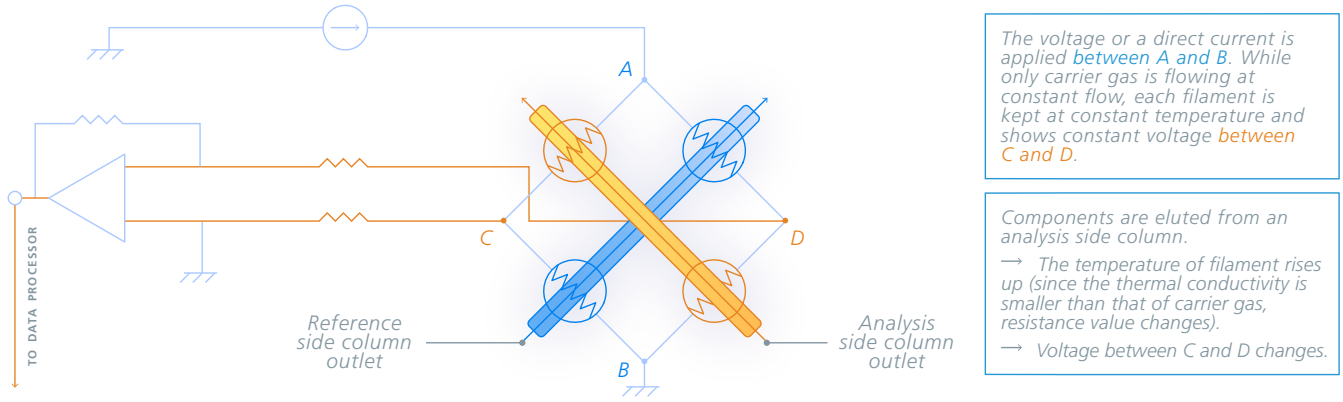

Figure 9: Schematic of a Thermal Conductivity Detector

5-1. Thermal Conductivity Detector (TCD)

TCD detects using difference of thermal conductivity of between sample and carrier gas. Generally, He is used as carrier gas. However, He has a high thermal conductivity constant number. When the target compound is He and H_2, N_2 or Ar is used as carrier gas. All compounds have each unique thermal conductivity constant as shown below.

When thermal conductivity of target compound is larger than the carrier gas, peak is detected as a negative value. TCD can detect most compounds except for carrier gas but its sensitivity is not as high as compared to other detectors, such as the FID or BID. Among the other GC detectors, it has almost the lowest sensitivity. Its main application is analysis of permanent gases it can analyze the compounds that are undetectable using FID, such as water, formaldehyde, and formic acid.

5-2. Flame Ionization Detector (FID)

In FID, the components are burned and ionized; it is used for almost all organic compounds, which have C-H or C-N structure. There are several exceptions such as CO, CO_2 and CS_2 which are non-organic compounds that cannot be detected. Carbonyl group and C=O carbon atom of carboxyl group are also not detectable. FID can

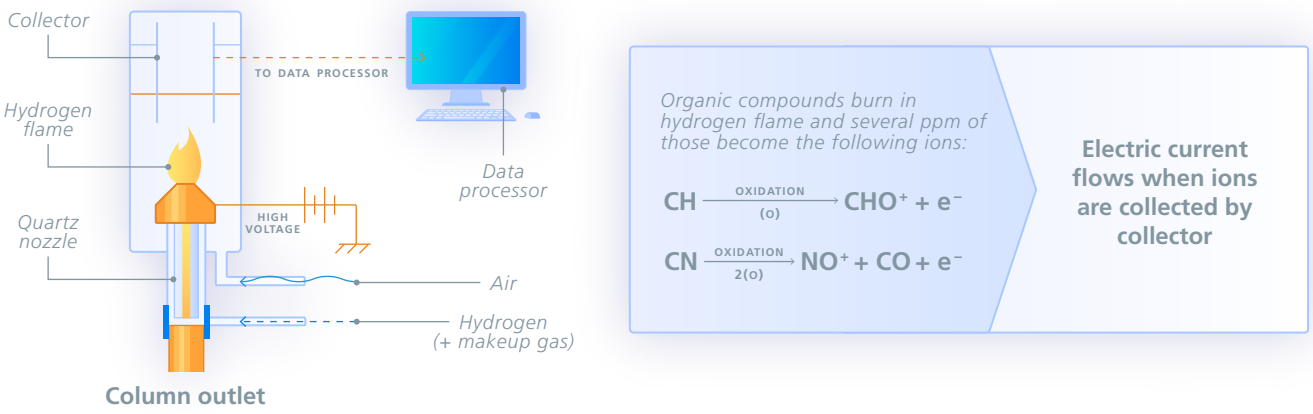

Figure 10: Schematic of Flame Ionization Detector (FID)

detect most hydrocarbons which have a C-H bond in its structure, except for HCHO and HCOOH. CHCOOH, and acetaldehyde (CH_3CHO) which are from the same group as HCHO and HCOOH respectively, and have one more carbon. They are detectable because a carbon of C-H exists in addition to C=O. FID is mainly used to analyze organic compounds and overall, it is a stable and highly sensitive detector, used in various fields.

5-3. Barrier Discharge Ionization Detector (BID)

The BID is a highly sensitive device that creates ionization from a He-based, dielectric barrier discharge plasma. A 17.7 eV plasma is generated by applying a high voltage to a quartz dielectric chamber, in the presence of helium at a relatively low temperature. Compounds that elute from the GC column are ionized by this He plasma energy and then detected by the collection electrode to be processed as peaks. BID is able to detect most of compounds except for He and Ne. The BID is more sensitive than both TCD and FID with the ability to analyze down to sub-ppm levels of water and inorganic gases.

5-4. Mass Spectrometer (MS)

The GC-MS is superior in qualitative and quantitative capability and, with greatly improved performance and operability, it is rapidly spreading in the market. The composition and principle of the GC-MS will be explained in a separate document.

5-5. Electron Capture Detector (ECD)

Radioactive isotopes are equipped (^{63}Ni for Shimadzu product) in ECD. It is highly selective for electrophilic compounds which become negative ions after obtaining electrons such as organic halogens, organic metal compounds, and diketones. The sensitivity depends on the type of halogen, its number and its structure. ECD's main application is environmental analysis, such as residual pesticides, residual PCB, Chlorine VOC in drain water, organic mercury in environmental field, etc.

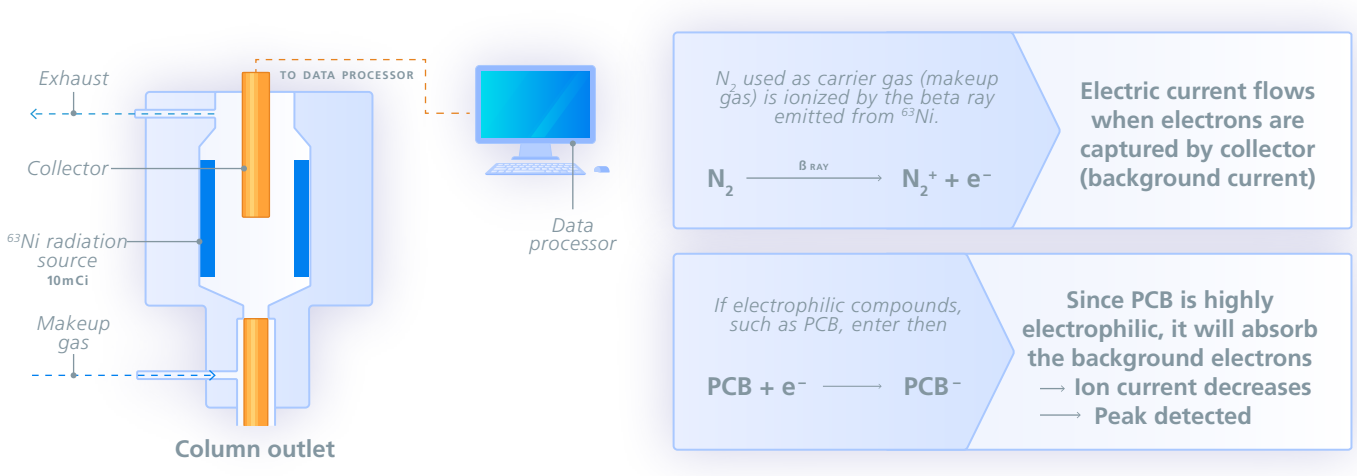

Figure 11: Schematic of an Electron Capture Detector (ECD)

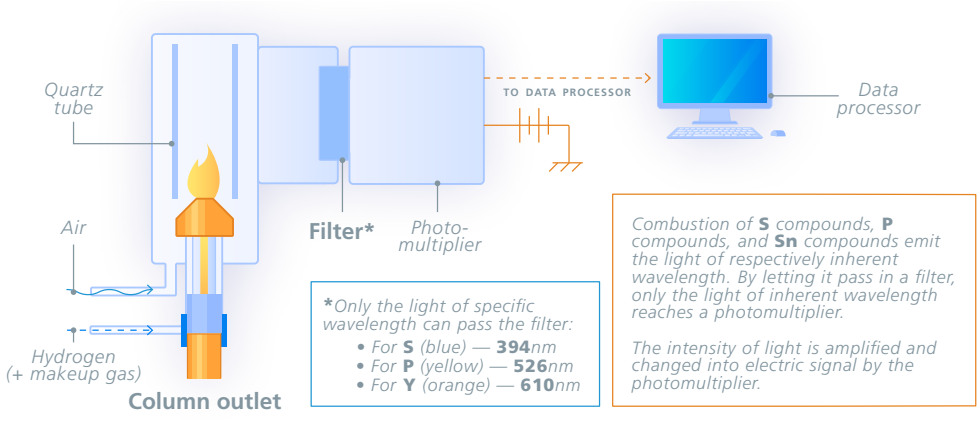

Figure 12: Schematic of a Flame Photometric Detector

5-6. Flame Photometric Detector (FPD)

FPD is a highly selective and sensitive detector, especially for phosphorus (P), sulfur (S), and tin (Sn) compounds. It detects light unique to P, S and Sn in the hydrogen flame through interference filter. FPD is so stable and sensitive that it is easy to use alongside selective detectors. It has been used in food analysis, — to detect phosphorus pesticides, sulfur odors and food flavors — and in environmental analysis to detect organic tin compounds in sea products.

5-7. Flame Thermionic Detector (FTD)

A flame thermionic detector is a highly selective and sensitive detector for organic nitrogen compounds, inorganic and organic phosphorus compounds. It is also known as a nitrogen phosphorus detector (NPD) because it can detect nitrogen and phosphorus compounds. Its principle and basic structure is the same as FPD. Inorganic nitrogen compounds cannot be detected using FTD, so it cannot be applied to ammonium analysis. Its selectivity to phosphorus compounds is lower than FPD. The main applications of FTD include the analysis of drugs, nitrogen pesticides, and phosphorus pesticides, etc.

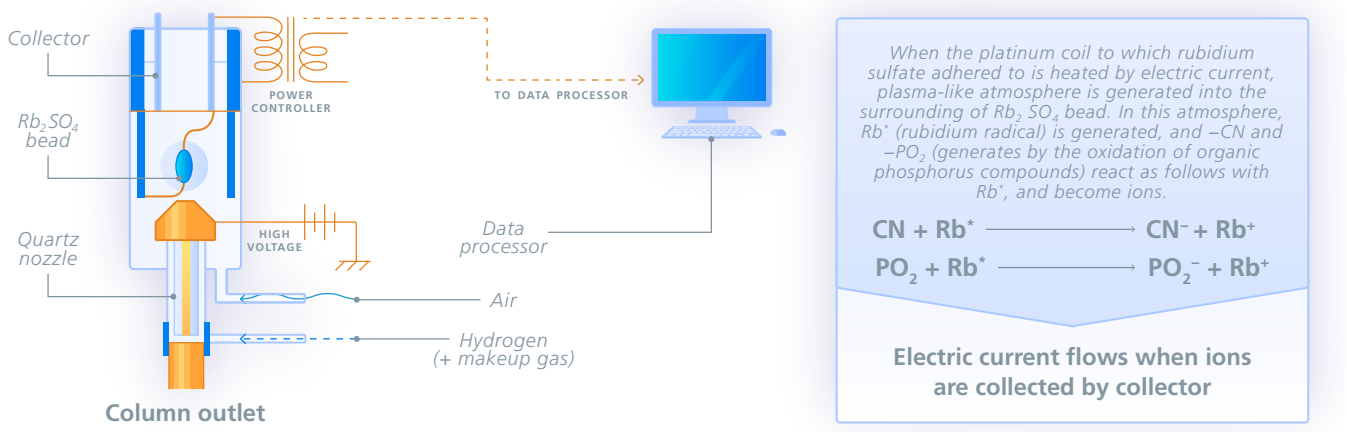

Figure 13: Schematic of a Flame Thermionic Detector

In FTD, organic nitrogen compounds and organic phosphorus compounds are decomposed and ionized in the vicinity of the collector surface of superheated rubidium salt and detected. It is highly sensitive, but this rubidium collector is a consumable. If not restored or replaced, the overall detector's sensitivity tends to drop over time.

5-8. Sulfur Chemiluminescence Detector (SCD)

The sulfur-containing compound (RS) is burned at about 800 °C in a hydrogen-filled atmosphere, to generate sulfur monoxide (SO). SO then reacts with ozone from the ozone generator to obtain an excited state of sulfur dioxide (SO_2),and detects chemiluminescence when it returns to the ground state.

A series of reactions is carried out under reduced pressure of 5 Torr. The emitted light (hν) passes through the optical filter and is detected by the photomultiplier tube (PMT).

$R\text{-}S + O \rightarrow SO + H\text{-}R + H_2O$

$SO + O_3 \rightarrow SO_2^* + O_2$

$SO_2^* \rightarrow SO_2 + h\nu$ (300~400nm)

SCD has high selectivity and sensitivity with a wider dynamic range as compared with FPD for the same sulfur compound. Since the response is hardly influenced by the structure of the compound, it is possible in principle to quantify all the sulfur compounds with one standard substance. There is no unavoidable quenching phenomenon in FPD. SCD is widely used for analysis of petroleum products and foods.

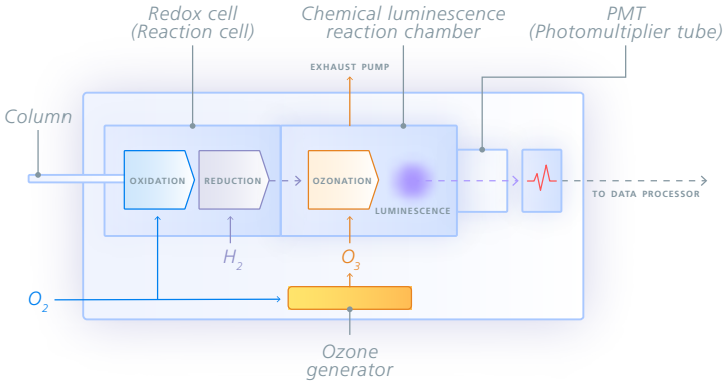

Figure 14: Schematic of a Sulfur Chemiluminescene Detector (SCD)

6. Sample Introduction

The target samples to be analyzed by GC are mostly gas or liquid. In the case of manual injection, the gas sample is sampled by a gas tight syringe and injected into the device. For liquid samples, the sample is injected with a microsyringe exclusively for liquid. Auto injectors capable of automatically injecting samples into liquid samples are widely used.

The injection volume often used for analysis is 0.2 to 1 mL for gas and 1 to 2 µL for liquid. Improvement in sensitivity can be expected if the injection amount is large, but separation often becomes worse due to excessive loading. Hence great care is needed as it will cause problems unless an appropriate amount is injected.

7. Sample Pre-treatment Devices

Various types of sample pre-treatment devices are used with GC and GC/MS. The major sample pretreatment system and methods are described in this chapter. However, you may refer to the complete guide of choosing the right GC pre-treatment device in a separated document.

7-1. Static Headspace (SHS)

The sample is first sealed and heated. The gas in the space (headspace) above the sample matrix, within sealed container is introduced into the GC. Components with relatively high volatility can be detected with high sensitivity. Samples are comprised of liquids and solids, but often are liquid samples. In the case of a liquid sample, when kept at a constant temperature for a certain period of time, the compound concentration of the headspace gas is in an equilibrium state. Therefore, measuring the headspace gas makes it possible to quantify the liquid sample.

After sealing the sample, it should be kept at an appropriate temperature and time, so that gas can be collected and introduced to the GC. A headspace sampler that automatically performs temperature, heat retention time, and gas sampling is also used to automate and improve the accuracy of analysis (Figure 15).

7-2. Dynamic Headspace (DHS)

Purge-and-trap is a device used for high sensitivity measurement of low concentration volatile organic compounds (VOC) in water that operates under the principle of dynamic headspace. Gas is passed through sample water, and VOC is driven out together with gas. This gas is collected and desorbed by a trap tube to be analyzed. There are several kinds of adsorbents for trap pipes, and they are selected by target components.

There are generally four steps in the preprocessing process (Figure 16).

1. Purge trap

An inert gas such as He or N2 is passed through the water sample to purge the volatile VOC, and the evacuated VOC is then trapped in the trap tube (trap).

2. Dry purge (water removal)

At the time of the purge trap, moisture is also trapped in the trap tube, so there is concern about its influence

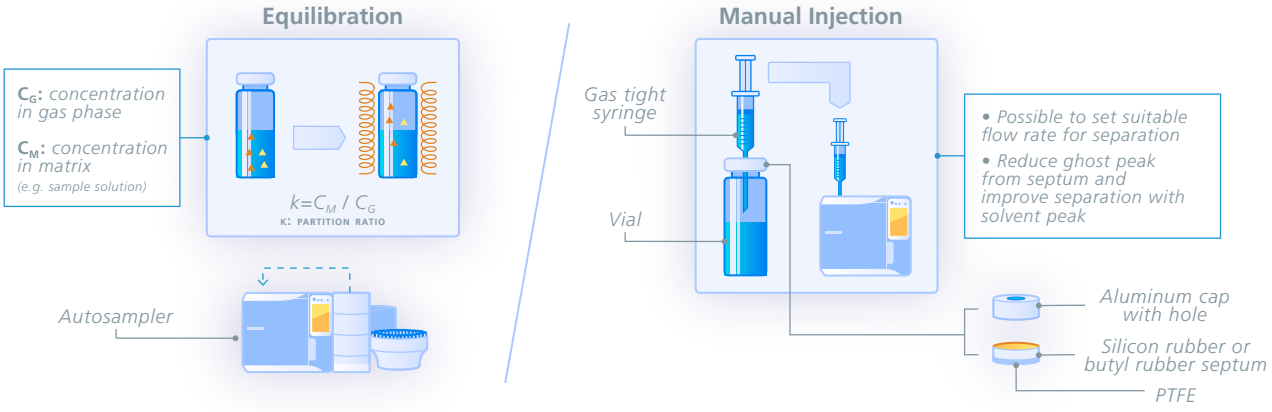

Figure 15: Principle of Static Headspace

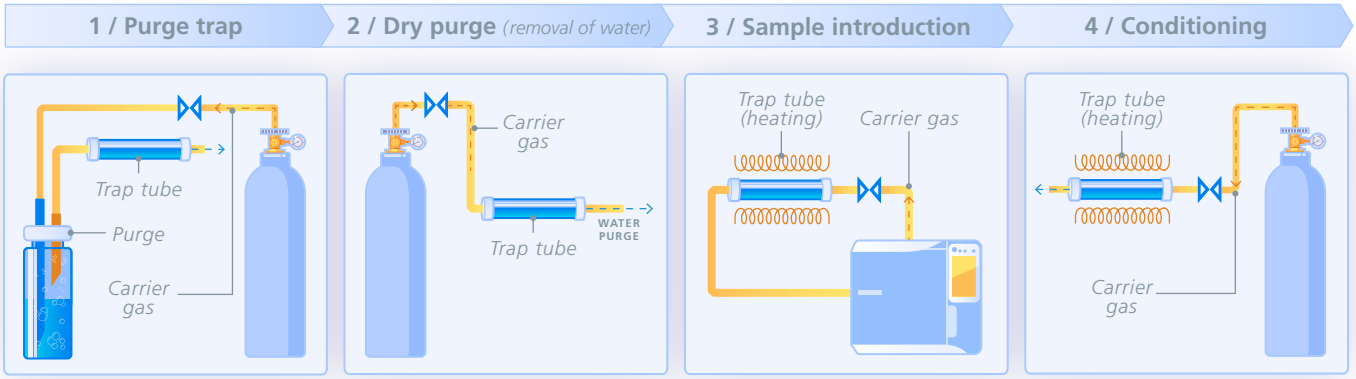

Figure 16: Principle of Dynamic Headspace

on the measurement. Only the inert gas is allowed to flow into the trap pipe after the purge trap to remove moisture as much as possible. It is necessary to perform dry purging to the extent that the component to be measured does not desorb. This method is unsuitable when the holding power of the component to be measured to the adsorbent is low and the difference in holding power with water is small.

3. Sample introduction

Heat the trap tube to desorb the VOC and introduce the sample into GC and GC/MS. When heating and desorbing the trap tube, the direction of the carrier gas is made to flow opposite to that during the time of trapping. This could improve the desorption efficiency.

4. Conditioning

While flowing inert gas, heat and purge any remaining organic matter in the trap tube.

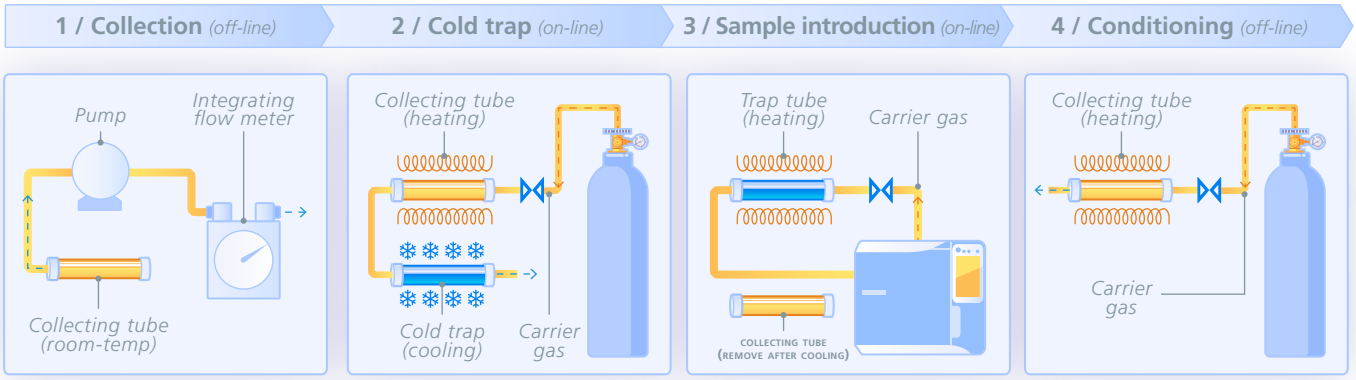

Figure 17: Principle of Thermal Desorption

7-3. Thermal Desorption (TD)

In order to measure low concentration volatile organic compounds (VOC) in the environment and indoor atmospheric air, a certain amount of gas has to be passed through a collection tube filled with an adsorbent, the VOC is to be concentrated and then heated. It is a method of desorbing and analyzing components.

The process consists of four steps of collection, cold trap, sample introduction, and conditioning (Figure 17).

1. Collection

A pump is connected to the collecting pipe, and the sample gas is passed, so that the VOC in the atmosphere is collected. The cumulative flow meter is connected to the rear stage of the pump in order to find the collected volume.

2. Cold trap

The VOC desorbed from the collection tube needs to be re-concentrated before it is introduced the GC and GC-MS via the cold trap. The trap filled with adsorbent is cooled to below room temperature and the collection time is heated with the VOC being desorbed as to be able to recapture it with the trap.

3. Sample introduction

The trap is heated to desorb the VOC and introduce it to GC and GC-MS.

4. Conditioning

By heating the collecting tube, it is possible to reuse it by expelling the remaining organic matter. It is desirable to heat the tube just before collection as much as possible. The collecting tube has to be capped and not exposed to the outside air as to prevent contamination of organic matter.

7-4. Pyrolysis Analysis (Py)

When a polymer sample is heated to about 400 to 900 °C, the decomposed gas is generated. It is a method used to obtain information on macromolecular monomers, dimers, trimers and compounds derived from polymer structures by introducing decomposition gas into GC and GC/MS for analysis. In order to obtain the information on the decomposed gas with good reproducibility, it is required to heat the sample instantaneously at a temperature as accurate as possible. Typical methods for instantaneous heating include filament type, induction (Curie point) heating furnace type and heating furnace type (free fall). In addition, an inert material or surface treatment that does not cause any catalytic reaction is required for the container (cup) with a minimum sample amount (about 100 to 500 µg) (Figure 18).

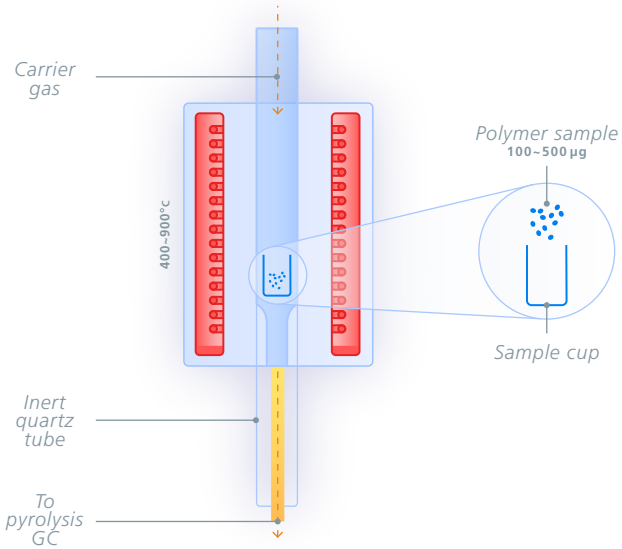

Figure 18: Principle of Pyrolysis Analysis (Py)

8. Summary

GC is useful and universal analytical instrument. By choosing injection method, column, detector and sample pretreatment method, it can be applied for wide variety of application. Understanding the system features and appropriate sample treatment can result to a more efficient process. To analyze actual samples, clean-up process such as dissolution and extraction may be needed. To maintain higher sensitivity, it is important to ensure the instrument condition is optimized.

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