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Change of MS Method and Comparison of SIFT-MS Method

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Abstract

Purpose: This study examines the history of the evolution of MS analysis and intends to consider the future direction of technological development through the difference from the latest technology, SIFT-MS. **Research design, data and methodology:** A method of analysis will be described in detail at the below by SIFT-MS (Selected Ion Flow Mass Spectrometry), which is a technology developed by a company called SIFT Technologies. **Results:** The initial concept of mass spectrometry was begun in the late 1890s, and it continues to evolve even after the 21st century through the ripening stage of the 20th century. The development process of mass spectrometry by year has been described in detail in the Main text. **Conclusions:** Mass spectrometry, qualitative and quantitative analysis of substances plays a very important role in the research and medical fields. The development of these analytical methods is expected to continue in the future, and faster and more accurate qualitative analysis and mass spectrometry will be developed than the level currently reached. In addition, it is expected that hardware and software will be configured so that non-analysis experts can handle it easily, and it will be used as a technology that is more closely related to our lives.

Keywords : Mass Spectrometry, GC-MS, History, Qualitative analysis, Quantitative analysis

JEL Classification Codes : Q50, Q51, Q52, Q55, Q59

1. Introduction

Mass spectrometry is an analytical technique that uses a mass-to-charge (m/z) ratio to identify chemicals in sample. This method identifies a compound by determining its molecular weight and analyzing its isotopic abundance. A mass spectrometer converts a sample into gaseous ions and then identifies the ions by their mass-to-charge ratio and relative abundance. Today, mass spectrometry is recognized as a well-known detection

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method with various advantages such as selectivity, sensitivity, and multi-sample analysis. Mass spectrometry can be combined with various chromatographic techniques such as liquid chromatography, thin layer chromatography, gas chromatography or inductively coupled plasma.

Mass spectrometers are widely used in many research fields and industries, including the pharmaceutical and food industries, health clinics, clinical research laboratories, forensics and environmental testing laboratories. Mass spectrometry converts individual molecules into ions and then analyzes the relative abundance of the ions produced. In the ion chamber of mass spectrometry, each individual molecule is ionized to form a molecular ion, which has one less electron than the parent molecule. Molecular ions or 'radical cations' are fragmented into ions, which are then further subjected to fragmentation process. Mass spectrometry produces many ions from one complex sample. Ions are accelerated in an electromagnetic field and separated according to the ratio of mass to charge (m/z). The detector in mass spectrometry records the ions in proportion to their relative abundances and produces a mass spectrum about that molecule. Due to the above principle and usability, its accuracy has been recognized, and it is now widely adopted and is being utilized as an analytical method for process test methods.

In this study, we will examine the history of MS analysis and consider the future direction of technological development through differences from the latest technology, SIFT-MS.

2. Research Method

The history of mass spectrometry was presented by collecting and organizing historical information by year of reliable sites (https://www.slideshare.net/, https://en.wikipedia.org/wiki/). In addition, the accuracy of historical information was verified by presenting and comparing the research data of each research paper and related papers. SIFT-MS (Selected Ion Flow Mass Spectrometry) is a method that can make qualitative and quantitative analysis are possible until real-time PPT level for organic carbon, inorganic carbon, and volatile organic compounds through special chemical ionization method (CI) using three reagent ions (H_3O^+, NO^+, O_2^+).

Currently, this technology is a technology developed by a company called SIFT Technologies, and the method will be discussed in detail below and the difference from the existing method will be explored.

3. Research Results & Discussion

3.1. The Core Technology of MS Analysis-Ionization

The ionization process is a core technology essentially required in mass spectrometry, and various techniques are used depending on the method. One of the most important factors in the ionization process is the transfer condition of internal energy. Some of the ionization processes are energetic and cause strong fragmentation. Also, some ionization processes gently produce molecular ions.

3.2. History of MS Analysis

EI (Electron Ionization) and CI (Chemical Ionization) methods are suitable for compounds that are stable against heat and have strong evaporation as ionization proceeds in a gaseous state. The EI method, devised by Dempster, was improved by Bleakney and Nier. EI is used for analysis in the organic chemistry sector. This ionization method targets gaseous molecules and causes strong fragmentation. For this reason, molecular ions do not always occur. The heated filament emits electrons, which are accelerated toward the cathode and collide with the gaseous analyte injected into the ion source. In the case of CI, it is a technology that generates ions by using slightly excess energy compared to EI. CI makes less fragmentation, so molecular ions can be easily observed, and ions are generated by colliding with basic ions present in the ionization source. The generated ions are obtained by ionizing a gas or liquid sample, and when an analyte is injected, and they transfer the hydrogen ions to generate MH^+ ions.

After vaporizing the sample, it was made into ions and then accelerated to separate the ions according to the mass-to-charge ratio. Since then, a mass spectrometer (mass spectrometer) that obtains a mass spectrum through the previous way is currently positioned as an analysis equipment that plays an important role in investigating chemical composition. With mass spectrometry,

information on elemental composition and molecular structure, qualitative/quantitative analysis of complex mixtures, information on the solid surface, and information on the ratio of isotopes in the sample can be obtained.

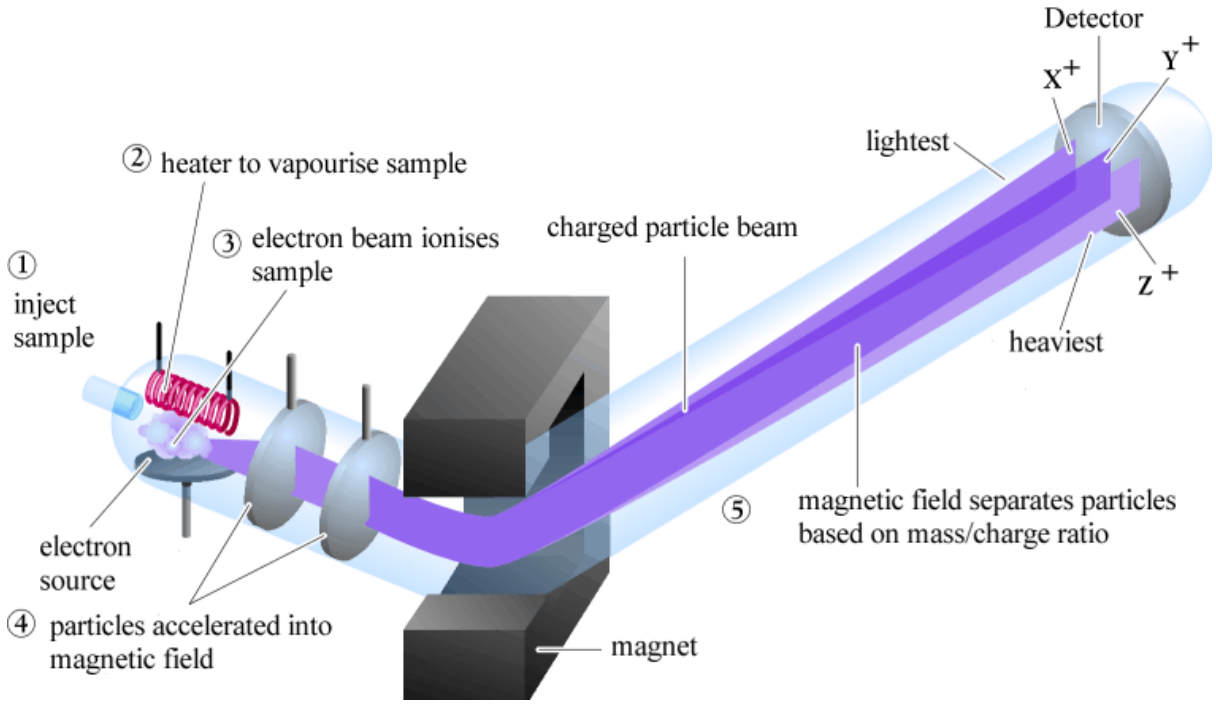

Figure 1: Mass Spectrometer Structure

The initial concept of mass spectrometry began in the late 1890s, and it continues to evolve even after the 21st century through the ripening stage of the 20th century (Buie, 2011). The history of mass spectrometry began in 1886 by Eugen Goldstein. He discovered a line in which charged gas molecules move from the anode to the cathode under low pressure conditions. And this was called a bipolar wire. Wilhelm Wien discovered in 1899 that these poles were bent by a strong electric or magnetic field. Then he devised a device and attempted to separate molecules according to the charge-to-mass ratio. Joseph John Thompson set up Vienna's device at a lower pressure, and he discovered the electron in 1897. Then he proposed a model of the atom, and thanks to his work the mass spectrometer could be invented. The prevailing view is that mass spectrometers were born from Joseph John Thompson. At that time, it was called a parabola spectrograph, not a mass spectrometer.

It is presumed that it was called parabola spectrograph because of the shape of the bipolar wire curved in a parabola shape. In 1946, the first time-of-flight (TOF) mass spectrometer was developed by W. Stephens. In 1948, Vickers released MS-2 which used the first electron ionization mass spectrometer and the first ion cyclotron mass spectrometer known as Omegatron was developed at the University of Minnesota. This device combines two sample inlets and a switching valve to enable sample changeover faster. In 1956, Roland Gohlke and Fred McLafferty developed the GC-Mass spectrometer by combining gas chromatography and mass spectrometer (Buie, 2011). Mode 12-101, developed by Bendix Aviation Corporation by combining GC and TOF mass, made it possible to measure mixtures immediately without requiring pre-treatment and separation time. In 1964, The first quadrupole mass spectrometer (GC-MS) for residual gas analysis was commercialized by EAI's W. M. Brubaker, P. Michael Uthe and Robert Finnigan.

In 1967, PerkinElmer introduced the first Magnetic Double-Focusing GC-MS, Model 270. In 1968, the electrospray ionization (ESI) technique at atmospheric pressure was studied by Malcolm Dole (Buie, 2011).

Although this research has not seen the light of more than 20 years, after that the development of LC-MS will

make a great contribution to the utilization of mass spectrometers in biochemistry. In the 1970s, Fourier-transform, Secondary Ionization (ionization), Plasma desorption (desorption), laser desorption, thermal desorption, spark source (light source), glow discharge (discharge), etc. Several types of modified mass spectrometers had been tried. In 1976, Hewlett-Packard (HP) introduced the world's first Integrated, digital Benchtop GC-MS system 5992. In addition, this equipment uses a hyperbolic chromium-molybdenum (Cr-Mo) alloy quadrupole filter for the first time. In 1983, the first commercially available ion-trap system was introduced at the Finnigan MAT. The radio frequency scanned by the ion trap technique increased the mass to charge ratio. Today, the ion trap system is usually used for GC and LC-MS detectors, and consists of a single mass spectrometer device. In 1983, PerkinElmer SCIEX introduced the ELAN250, the first inductively coupled mass spectrometer (ICP-MS) consisting of platinum cones and an inert sample introduction system. In 1987, Finnigan released the MAT90 series Mass Spectrometer (Buie, 2011).

MAT90 Series is the first mass spectrometer that all be controlled by computer. In 1988, John Bennett Fenn presents two papers on electrospray ionization (ESI) technology. The development of this technology provides an opportunity for the mass spectrometer to be dramatically widely applied to the analysis of biopolymers and synthetic polymers including proteins and DNA. Until this method was developed, the use of mass spectrometry in polymer research was very limited due to limitations in thermal and chemical decomposition during mass spectrometry. In 1988, HP introduces the world's first mass spectrometer 5971 MSD equipped with a hyperbolic glass quadrupole.

In 1990, PerkinElmer introduced the ELAN5000, the first ICP-MS equipped with a turbomolecular-pump. In 1992, mass analysis technology made it possible to analyse low-level peptides. In 1992, Shimadzu launched the Kompact MALDI series, which can analyse peptides, proteins, sugars, fats, and nucleotides in pharmaceutical and metabolite research fields. In 1996, mass spectrometer was fused with HPLC (High Performance Liquid Chromatography) equipment and mass spectrometry research on viruses began. In 1997, Shimadzu released the LCMS-QP8000 model equipped with ESI and Atmospheric Pressure Chemical Ionization interfaces. The 2002 Nobel Prize in Chemistry is awarded to three scientists who have contributed to the technology capable of determining the structure and discrimination of macromolecules in biochemistry (Buie, 2011). Two of these will be awarded to scientists who contributed to the development of the mass spectrometer. One is Koichi Tannaka from Shimadzu, who developed the MALDI mass spectrometer, and the other is John B. Fenn, who developed electrospray ionization (ESI).

In 2002 Shimazu Corporation releases laser ionization quadrupole ion trap time of flight mass spectrometer AX IMA-QIT. In 2005, Direct Analysis in Real time (DART) ion source technology was patented (Buie, 2011). DART technology is a technology that can perform ambient ionization immediately in the atmospheric state without sample pre-treatment. This technology was developed by Laramee and Cody, and is commercialized by JEOL and IonSense. In 2006 Water corporation introduces SYNAPT High-Definition Mass Spectrometer (HDMS). HDMS is characterized by being able to measure the size, shape, and charge of ions in addition to mass measurement. In the same year, Agilent released the innovation GeneSpring MS software. With this software, it will be possible to invent and extract relevant biomarkers from mass spectrometry data. In 2008, Agilent introduced mass spectrometer technology that can detect femtogram mass for the first time.

In 2009 Thermo Fisher Scientific introduces the LTQ Velos product. In order to pursue the fastest and most sensitive ion trap mass spectrometer, this product has increased scan speed and resolution at the same time. In 2010, Bruker acquired the in vitro diagnostic CE Mark for the first MADI-TOF-based microbial identification workflow solution, IVD MALDI Biotyper. This system pioneered the use of mass spectrometers in clinical diagnostics. Waters Corp. launches the Xevo MS Platform (Xevo TQ-S, Xevo G2 QTof). This product has strengths in difficult quantitative analysis based on the step-by-step change in sensitivity (Buie, 2011). Mass spectrometer continues to evolve due to recent advances in biotechnology. A mass spectrometer product with tandem

expansions and multiple connection technology grafted onto HPLC equipment is expected to be released, and the size of the product will continue to become smaller like a portable device. And mass spectrometer is an indispensable instrument for modern chemical analysis, and it is expected to continue to solidify its position as the ultimate chromatography detector.

3.3. SIFT-MS Method

SIFT-MS is a method that can make qualitative and quantitative analysis are possible until real-time PPT level for organic carbon, inorganic carbon, and volatile organic compounds through special chemical ionization method (CI) using three reagent ions (H_3O^+, NO^+, O_2^+), and the analysis proceeds through five major steps. Step 1 is a step of forming reagent ions by irradiating microwaves, and step 2 is a step of selectively moving a desired reagent ion from among the reagent ions generated in the first quadrupole mass tube. In step 3, the sample and reagent ions are flowed through a special flow tube of SIFT-MS to form a reaction, and in step 4, through the second quadrupole mass tube, mass filtration is performed from low mass to high mass, and only the specified mass value is scanned. Step 5 is the detection process, and the selected reaction product ions are detected with a stable and reproducible sensitivity using a particle amplifier. After the above 5 steps, the analysis result can be checked through the SIFT-MS display.

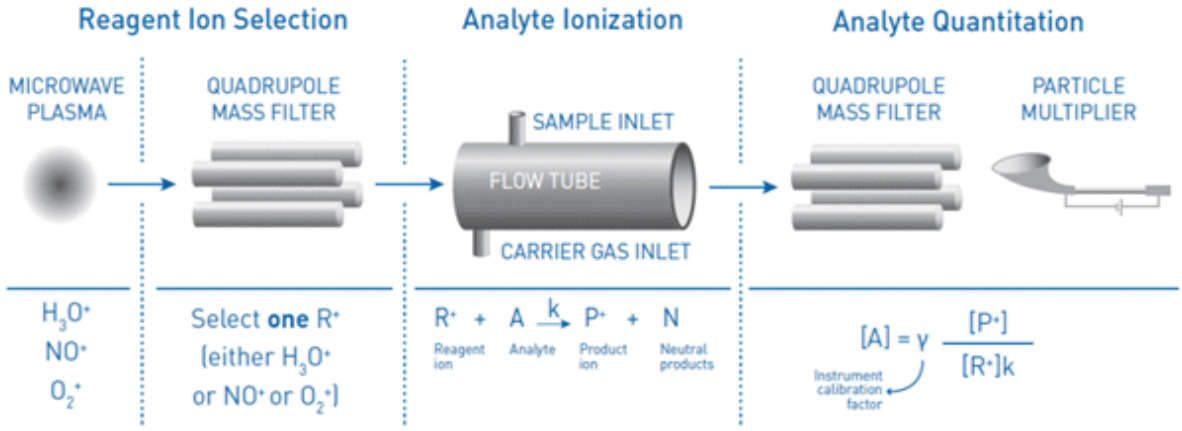

Figure 2: SIFT-MS Analysis Steps

SIFT-MS has two main features. First, the gentle chemical ionization method has the following advantages as it can show reproducible analysis results by accurately controlling the reagent ion energy because the energy required for ionization and sample fragmentation are less than that of the existing EI method GCMS.

• During measurement, there is very little crushing in the ion generation process, so the quantitative error is very good.

• Analysis time compared to GC-MS is 1/10 of that of GC-MS as it does not require a chromatograph.

• Accurate control of ionization energy enables absolute quantification.

As a second feature, compared to CI-type mass spectrometers (PTR-MS, APCI-MS) that use a single reagent ion (H_3O^+), SIFT-MS allows multiple reagent ions (H_3O^+, NO^+, O_2^+) to act independently at the same time. It shows the various reaction mechanism and has the following advantages.

• Clear separation of isobars and isomers is possible.

• The fastest qualitative and quantitative analysis is possible due to the speed of replacing multiple reagent ions within 1/100 of a second.

• With a wide range of motion, it is possible to accurately analyse automobiles and breathing samples with large deviations in mass values due to various driving processes.

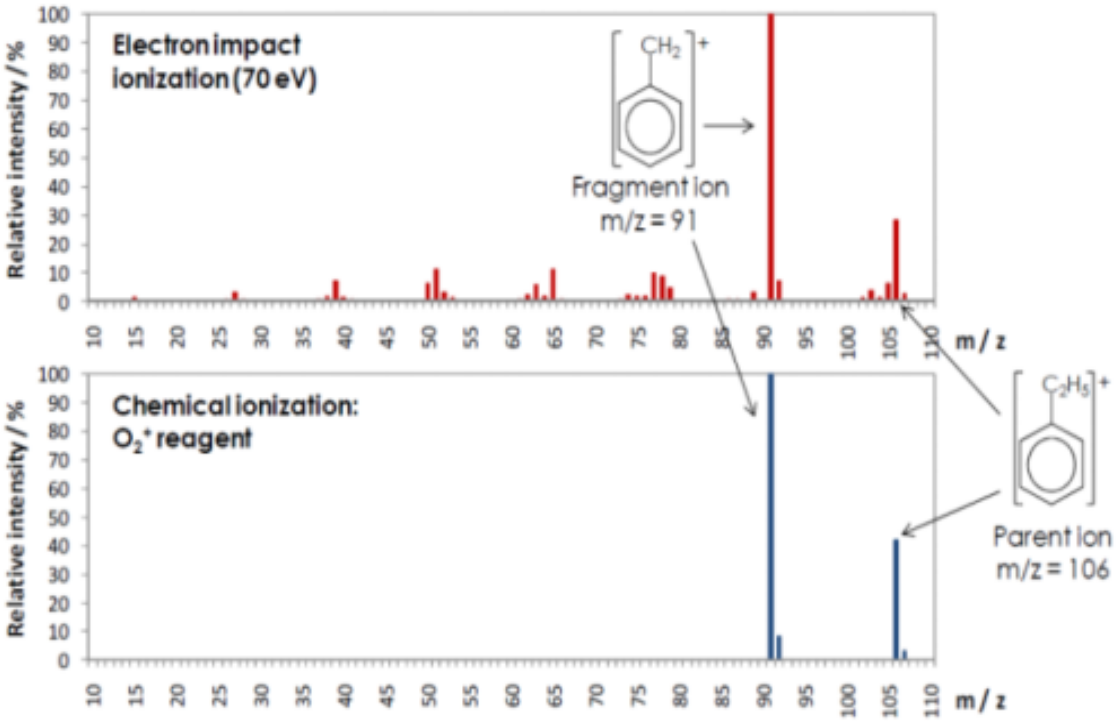

Figure 3: Comparison of Conventional MS Analysis and SIFT-MS Analysis

SIFT-MS can analyse both analyzable substances in GC and HPLC without separate processing, and can reduce cost and time. If SIFT-MS with the above-mentioned advantages is used, it is possible to analyse various substances quickly and accurately, and it is possible to reduce the analysis cost.

3.4. The Comparison Test Result Review of Experiment SIFT MS VS GC MSD

A "side-by-side comparison of spike canisters" shows that target levels of VOCs range from low to moderate ppbv (parts per billion by volume). The canisters were tested using SIFT-MS and FC/MS, and the comparative study results are shown in Table 1. Among VOCs, compounds present in the instrumental method but not in the mixture are shown in italics. In a mixture containing a total of 17 VOCs, the two techniques showed similar results. Concentration differences greater than 30% were only observed for styrene (higher in SIFT-MS than GC/MS) and acetone and carbon disulphide (lower concentrations detected by GC/MS in all mixtures). It is not clear why acetone was found at lower concentrations in SIFT-MS measurements. This is because the association of NO^+ with acetone was used to determine the concentration of $CH_3COCH_3.NO^+$ at m/z 88, the amplitude of the associated ion peak. Experiments with two acetone permeable tubes (Vici Metronics) showed the Syft Vocie200's accuracy (Langford, Graves, & McEwan, 2014).

Table 1: Side-by-side Comparison of GC/MS and SIFT-MS of Four Different Mixtures Containing 17 VOCs with Listed Concentrations Shown as ppbv

	Mix 1		Mix 2		Mix 3		Mix 4	
Analyte	GC/MS	SIFT-MS	GC/MS	SIFT-MS	GC/MS	SIFT-MS	GC/MS	SIFT-MS
1,1,1-trichloroethane	40	34	1.2	0.8	19.4	17.7	4.4	3.5

1,2-dibromoethane[a]	*<0.5*	*0.2*	*<0.5*	*<0.11*	*<0.5*	*0.2*	*<0.5*	*<0.11*
1,3-butadiene[a]	*<2*	*0.2*	*<2*	*<0.16*	*<2*	*<0.16*	*<2*	*<0.16*
C2-alkylbenzenes[b]	8.9	12.0	<0.5	0.7	5.1	6.9	0.8	1.1
C3-alkylbenzenes[c]	13	19	4.7	7.6	0.9	2.2	6.4	9.5
acetone	930	463	250	115	32	20	44	17.1
acetonitrile[d]		343		76		15.2		13.8
acrylonitrile	30	41	1.1	1.2	30	28	6.4	6.9
benzene	114	147	30	35	4.4	6.6	3.9	4.2
butanone	142	107	33	22	6	3.5	3.3	2.8
carbon disulfide	220	96	10.1	4.0	127	61	46	17
carbon tetrachloride[a]	*<0.5*	*1.0*	*<0.5*	*<0.77*	*<0.5*	*<0.77*	*<0.5*	*<0.77*
chlorobenzene	48	62	17.6	16.3	4.8	4.2	18.3	15.7
chloroethene[a]	*<0.7*	*<0.82*	*<0.7*	*<0.82*	*<0.7*	*<0.82*	*<0.7*	*<0.82*
dichlorobenzene[a]	*<0.5*	*<0.53*	*<0.5*	*<0.53*	*<0.5*	*<0.53*	*<0.5*	*<0.53*
isooctane	62	69	18.2	18.4	2	2.1	1.5	2.5
methyl bromide[a]	*<0.5*	*<0.56*	*<0.5*	*<0.56*	*<0.5*	*<0.56*	*<0.5*	*<0.56*
naphthalene	<0.5	<1.2	<0.5	<1.2	<0.5	<1.2	0.5	<1.2
styrene	1.7	3.5	<0.5	<0.42	0.8	2.2	<0.5	<0.42
tetrachloroethene	20	20	0.8	<1.3	11.5	11.2	2.1	1.3
tetrahydrofuran	192	191	67	44	10.3	8.4	8	5.2
toluene	39	41	1.2	1.6	21	21	7.1	4.2
trichloroethene	24	22	5.6	4.6	1.1	1.0	0.7	<0.73

Note: [a]These analytes were not included in the mix but were simply in the methods for analysis of both instruments. They represent signal backgrounds.
[b]C_2-alkylbenzenes is the total of ethylbenzene and the three xylene isomers for the SIFT-MS study. In this experiment, only ethylbenzene was added to the VOCs in the mixtures.
[c]C_3-alkylbenzenes is the total of all isomers for SIFT-MS. In this test, only 1,3,5-trimethylbenzene was added to the VOCs in the mixtures.
[d]Acetonitrile was not measured by the GC/MS instrument.
(as cited in Langford et al., 2014)

3.5. Experiment Side by Side Comparison of Four Samples Test Result Review

Side by side comparison samples test results are shown in Table 2. The first set of samples was taken from the contaminated soil and the crawl space around the underground fuel storage tank at the site where the soil was contaminated by the leak. Column 1 is the comparison result for VOC, column 2 is the background signal for each analyte, column 3 is the analyte concentration in the air sampled from the crawl space, column 4 is the analyte concentration obtained from the soil sample, column 5 is

the results of the second sample taken from air samples from the remediated clandestine methamphetamine ('P') laboratory. The following observations are possible. The correlation between the two techniques is shown in non-contaminated samples such as the ambient samples and the samples from the crawl space. Good correlations were obtained between the contaminated samples (column 4, soil gas) and aromatic hydrocarbons (benzene, toluene and C2-alkylbenzene), but not the other compounds. This is because the soil is saturated with hydrocarbons in the fuel, which creates mass overlaps with SIFT-MS products from the analytes included in the method. Under these conditions, when many multiple components exist at high concentrations, SIFT-MS is susceptible to interference. However, in the comparative analysis of residues in the 'P' lab, a good correlation between the two techniques and additional advantages were also found in SIFT-MS of all VOCs present. The GC/MS method does not report methanol because of the background of the canisters during the manufacturing process (Langford et al., 2014).

Table 2: Comparison of Analyte Concentrations in Two Commercial Samples Analysed at Hill Laboratories

Analyte	Ambient GC/MS	Ambient SIFT-MS	Crawl space GC/MS	Crawl space SIFT-MS	Soil gas GC/MS	Soil gas SIFT-MS	Cleaned 'P' lab GC/MS	Cleaned 'P' lab SIFT-MS
1,1,1-trichloroethane	<0.5	0.6	<0.5	0.8	<1.8	70	<0.5	1.1
1,2-dibromoethane	<0.5	0.3	<0.5	0.5	<1.8	11	<0.5	0.6
1,3-butadiene	<2	0.1	<2	0.0	<2	0.2	<2	0.0
C_2-alkylbenzenes[a]	<1.5	0.8	<1.5	1.9	589	525	<1.5	1.5
C_3-alkylbenzenes[b]	<2	0.9	<2	1.6	298	476	<2	1.1
acetone	[c]	5.9	[c]	8.6	[c]	25.5	8.0	5.0
acetonitrile	[d]	2.3	[d]	2.3	[d]	10.5	[d]	1.5
acrylonitrile	<0.5	0.2	<0.5	0.4	<1.8	5.2	<0.5	0.3
benzene	<0.5	0.6	<0.5	0.7	19.9	21	<0.5	0.7
butanone	<0.5	0.8	<0.5	0.6	<3	12	>14.4	18
carbon disulfide	<0.5	1.1	<0.5	1.4	<4	15	<0.5	1.2
carbon tetrachloride	<0.5	0.8	<0.5	1.0	<1.8	29	<0.5	1.2
chlorobenzene	<0.5	0.6	<0.5	0.8	<1.8	51	<0.5	1.4
chloroethene	<0.7	0.8	<0.7	1.0	<3	8.3	<0.7	0.7
dichlorobenzene	<1.5	3.6	<1.5	5.5	<5.4	41	<1.5	0.2
ethyl acetate	<0.5	1.1	<0.5	2.2	<1.8	280	<0.5	1.6
isooctane	<0.5	0.9	<0.5	1.2	230	458	<0.5	1.1
methanol	[d]	1.1	[d]	1.6	[d]	22.1	[d]	1148
methyl bromide	<0.5	0.4	<0.5	0.4	<1.8	9.7	<0.5	0.5
naphthalene	<0.5	2.7	<0.5	2.9	12.6	23	<0.5	0.7
propane	<0.5	0.6	<0.5	0.3	<1.8	18	<0.5	0.4

styrene	<0.5	1.4	<0.5	2.6	<1.8	13	<0.5	0.5
tetrachloroethene	<0.5	0.2	<0.5	0.4	<1.8	8.8	<0.5	0.3
tetrahydrofuran	<0.5	3.5	<0.5	4.0	<1.8	430	<0.5	1.8
toluene	1.7	1.6	11.1	8.7	430	323	0.8	1.3
trichloroethene	<0.5	0.2	<0.5	0.1	<1.8	3.3	<0.5	0.4

Note: [a]C_2-alkylbenzenes gives the total of ethylbenzene and the three xylene isomers for the SIFT-MS; speciation reported for GC/MS has been summed.
[b]C_3-alkylbenzenes gives the total for all isomers for SIFT-MS; speciation reported for GC/MS has been summed.
[c]Not reported for GC/MS due to interference from 2-methylbutane.
[d]Not reported for GC/MS.
(as cited in Langford et al., 2014)

4. Conclusions

Mass spectrometry is one of the most recognized analytical methods used in each field for the development of modern society, such as environment, medicine, and genetics. Qualitative and quantitative of substances are possible, and it has a long history of development starting in 1886. After the maturity stage in the 20th century, it has been continuously developed by many engineering students and scholars until the 21st century and the present day. Nowadays, GC-MS and LC-MS are used as mass spectrometers for chromatography using gas or liquid depending on the properties of the material. The analysis method referred to in this study called SIFT-MS is an analysis method that corresponds to the aforementioned CI (Chemical Ionization) which called previously in this study.

Multiple reagent ions (H_3O^+, NO^+, O_2^+) act simultaneously and independently, enabling clear distinction between isobars and isomers. According to these characteristics, it is possible to analyse materials that can be analysed in GC-MS and LC-MS at once without separately distinguishing them. Due to the small fragmentation, there is less fragmentation in the ion generation process, so the quantitative error is minimized.

Like this, mass spectrometry has been continuously developed since 1886 and is being used as an essential technology in each social field. At present, it has advanced to the stage of technology called SIFT-MS mentioned above. Compared to GC-MS and LC-MS, SIFT-MS maintains the shape of the parent molecule with less fragmentation, enabling accurate and fast analysis. Compared to the GC-MS method, the analysis time is as short as 1/10 of GC-MS. Currently, SIFT-MS technology cannot be recognized as an official test result, but its distribution is increasing due to the reduction in analysis time and the fact that it does not require professional manpower. In this study, the evolution of mass spectrometry and SIFT-MS, a new technology, were investigated through data. In our society, qualitative and quantitative analysis of substances plays a very important role in the research and medical fields. For this reason, analysis methods have been improved and developed since a long time ago and have reached the present.

The development of these analytical methods is expected to continue in the future, and faster and more accurate qualitative analysis and mass spectrometry will be developed than the level currently reached. In addition, it is expected that hardware and software will be configured so that non-analyst experts can handle it easily, and it will be used as a technology that is more closely related to our lives.

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