

Solutions for Innovation

Scientific / Metrology Instruments JEOL GC-MS Primer - A Guidebook -

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What is a Gas Chromatograph - Mass Spectrometer (GC-MS)? What can we know with gas chromatography / mass spectrometry (GC/MS)? What can a GC-MS be used for? Is electron ionization (EI) the only ionization method for GC/MS? Is there any other ionization method? What are the other ionization methods used for? What kinds of mass spectrometer are available for GC-MS? What is the difference in mass resolution between QMS/TQMS and TOFMS? When is accurate mass analysis [elemental composition elucidation] required? What is the difference in obtaining data between QMS/TQMS and TOFMS? How can a quantitative analysis be performed with GC/MS? Application examples of JMS-Q1600GC UltraQuad™ SQ-Zeta Application examples of JMS-TQ4000GC UltraQuad[™] TQ Application examples of JMS-T2000GC AccuTOF™ GC-Alpha What are the sample preparation devices connected to GC-MS? JEOL Mass Spectrometers - product lineup

JEOL Ltd.

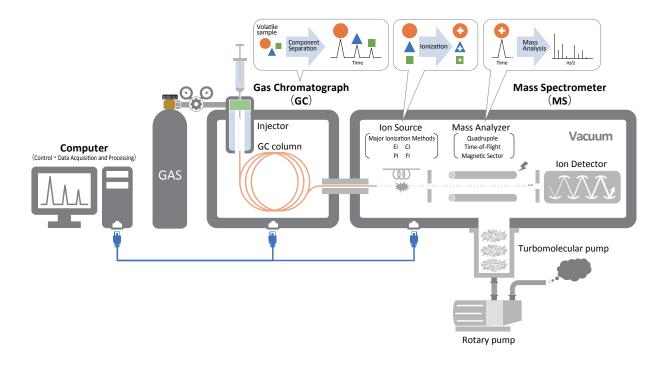
JEOL GC-MS Primer - A Guidebook -

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What is a Gas Chromatograph – Mass Spectrometer (GC-MS)?

It is a combination of Gas Chromatograph (GC) and Mass Spectrometer (MS) in which the MS acts as a detector for the GC.

On-line connection of Gas Chromatograph and Mass Spectrometer



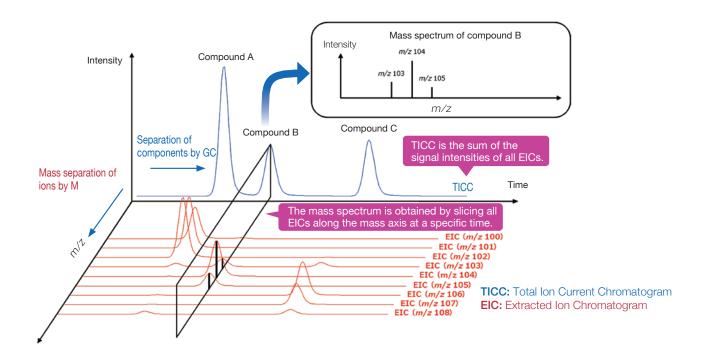
Ans.

What can we know with the gas chromatography / mass spectrometry (GC/MS)?

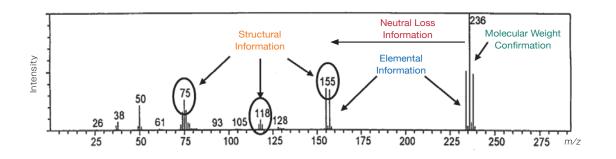


Components of a mixture can be separated by using gas chromatography. Every component is ionized. The mass (m/z) of the ions is measured and a mass spectrum can be generated. From these results, the following information can be obtained.

A mixture sample is separated using the gas chromatograph. Each separated component is analyzed using the mass spectrometer



Information obtained from a mass spectrum



- Confirmation of molecular weight obtained by observing M⁺⁺ or [M+H]⁺
- Structural elucidation using fragment ions, identification by using mass spectral library search
- Estimation of the presence/absence and number of certain elements by isotopic peak intensity ratio, estimation of the presence/absence of nitrogen.
- Elemental composition and double bond equivalence by accurate mass
- Mass and elemental composition information of neutral losses (e.g. functional group)
- Quantification by quantity (intensity) of ions

What can a GC-MS be used for?



It is used in a wide range of fields, as shown below, for identification and/or quantification of chemical species.

Application Range

- Natural product chemistry / Organic synthetic chemistry
- Chemistry / Materials, etc.
- Environmental analysis
- Biochemistry / Medical / Pharmaceutical / Forensics, etc.
- Other

Confirmation of molecular weight, structural analysis

Analysis of synthetic polymers, materials, additives, petrochemicals Analysis of POPs (persistent organic pollutants dioxin, PCB, agrochemicals, etc.) Analysis of water, atmosphere, and indoor air

Doping test, analysis of pharmaceuticals, illicit drugs, explosives, etc.

Analysis of flavors and fragrances, gas analysis, etc.

Is electron ionization (EI) the only ionization method for GC/MS? Is there any other ionization method? What are the other ionization methods used for?



In addition to electron ionization (EI), ionization methods for volatile samples suitable for GC/MS include chemical ionization (CI), photoionization (PI), and field ionization (FI). With EI, there are some compounds whose molecular weight is difficult to confirm. CI, PI and FI are effective for analyzing such compounds.

Ionization methods for volatile samples : EI, CI, PI, and FI

El (Electron Ionization)

→ Hard ionization method

El mass spectra of a large number of compounds have been archived as a library. Searching against an El mass spectral library such as NIST and Wiley Registry provides identification of the compound. However, it may be difficult to confirm the molecular weight of some compounds because the El method tends to dissociate intramolecular bonds. If the molecular ion of the compound cannot be detected, the accuracy of identification could be low.

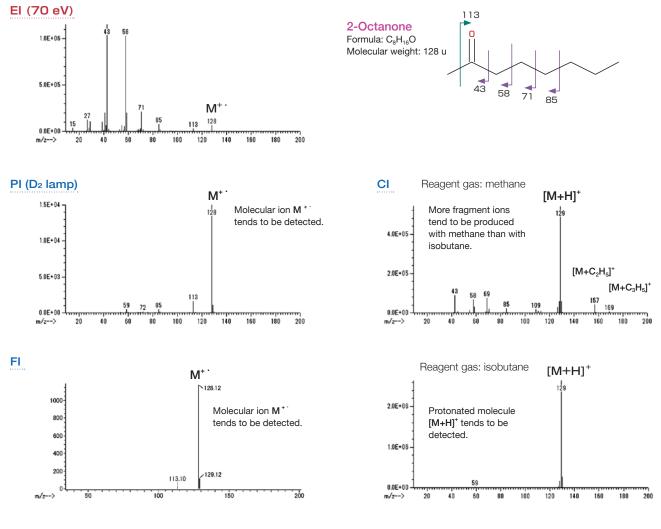
- CI (Chemical Ionization)
- PI (Photoionization)

Soft ionization methods

■ FI (Field Ionization)

These ionization methods may be used to confirm the molecular weight of a compound. Structural information for a particular compound may not be available. A suitable ionization method needs to be selected according to a sample.

Performing soft ionization allows for confirmation of the molecular weight.
 Performing both El & soft ionization improves the qualitative capability.



Comparison of mass spectra by different ionizations [2-Octanone]

CI → Ionization using the ion-molecular reaction with reagent ions.
 → A classical soft ionization method.

Selection of a reagent gas suitable for the analyte is important: methane, isobutane, ammonia, etc. Ability to donate protons: methane > isobutane Methane tends to produce more fragment ions.

- → Available on JEOL's quadrupole MS (QMS), triple quadrupole tandem MS (TQMS), and time-of-flight MS (TOFMS)
- \blacksquare PI \rightarrow Ionization by irradiating with a vacuum-ultraviolet (VUV) light
 - → The low ionization energy of 8 to 11 eV suppresses fragmentation.
 - → Some compounds may be fragmented during the ionization process or may not be ionized. (Depends on the wavelength range of the light source and the ionization energy of the compound.)
 - \rightarrow No reagent gas is required.
 - → Available on JEOL's QMS, TQMS, and TOFMS as an EI/PI combination ion source
- **FI** \rightarrow lonization using a high electric field
 - → Gives less excess internal energy and produces less fragmentation than EI, CI, and PI.
 - → The softest ionization among all ionizations for volatile compounds
 - \rightarrow No reagent gas is required.
 - → Available on JEOL's TOFMS as an EI/FI/FD combination ion source

* For details concerning the ionizations, please refer to the Ionization Methods for JEOL Mass Spectrometers -A Guidebook-.

What kinds of mass spectrometer are available for GC-MS?



The mass spectrometers used in GC-MS include Quadrupole Mass Spectrometer (QMS), Triple Quadrupole Mass Spectrometer (TQMS), and Time-of-Flight Mass Spectrometer (TOFMS).

(QMS)

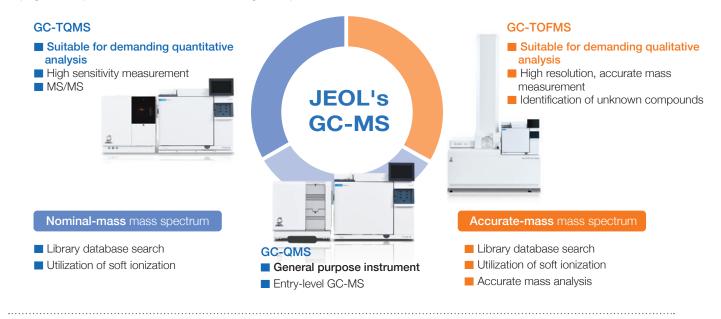
This instrument is easy to operate, robust and versatile, making it suitable for routine analysis.

(TQMS)

This instrument, allowing for two-step mass separation by tandem mass spectrometry (MS/MS) has high selectivity and it is suitable for the quantitative analysis of samples with complex matrices and interferences.

(TOFMS)

Majority of TOFMS's (including JEOL's) have high mass resolving power and high mass accuracy, and are capable of elemental composition elucidation by accurate mass measurement. A high mass-resolution TOFMS can identify unknown compounds without relying on library search and is suitable for non-target analysis.



What is the difference in mass resolution between QMS/TQMS and TOFMS?



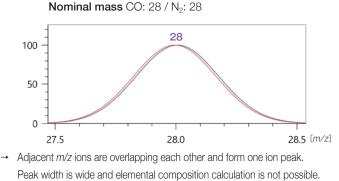
While QMS and TQMS can measure nominal mass, TOFMS has a mass resolution that provides mass accuracy in the order of 1/1000.

When accurate mass analysis is required, high resolution TOFMS is an optimal choice.

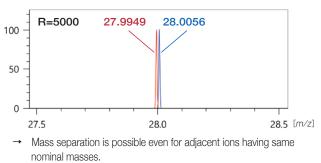


m/z 28 Carbon monoxide (CO: 27.9949) Nitrogen (N₂: 28.0056)

Difference of two exact masses: 0.0107 Da



Accurate mass CO: 27.9949 / N2: 28.0056



Elucidation of elemental composition is possible.

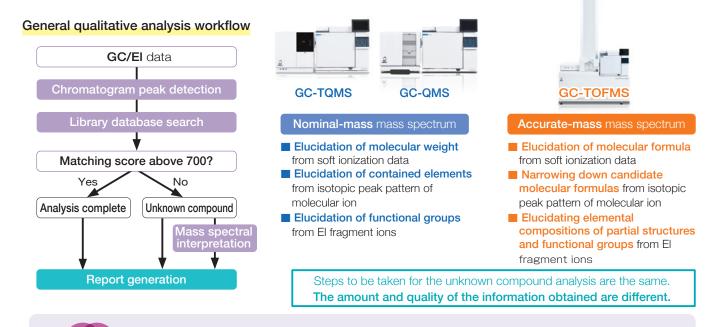
When is accurate mass analysis [elemental composition elucidation] required?



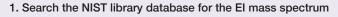
The first step in GC/MS qualitative analysis is to search the NIST mass spectral library.

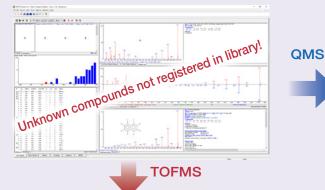
When it is difficult to judge whether or not molecular ions are present in the EI mass spectrum, you can improve confidence in identification by detecting molecular ions from soft ionization data. However, for unknown compounds, conclusive identification can be difficult.

By performing accurate mass analysis, we are able to know the elemental composition (its molecular formula) and opt for further structural analysis.

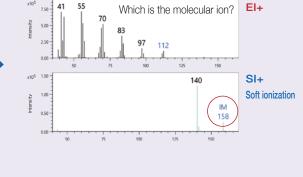


Qualitative analysis workflow for unknown compounds





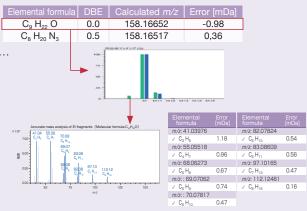
2. Search for molecular ions of unknown compounds Determine the molecular weight with soft ionization (SI).



2. Search for molecular ions of unknown compounds Soft ionization and accurate mass analysis allows you to determine the molecular formula.
→ But if you have more than one candidate molecular formula...
3. Narrowing down candidate molecular formulas by isotope peak pattern
→ If you still have more than one candidate...

4. Checking whether the elemental compositions of the El fragment ions can be derived from the candidate molecular formula

If the candidate molecular formula is correct, the elemental compositions of El fragment ions can also be obtained, allowing for further elucidation of structure.



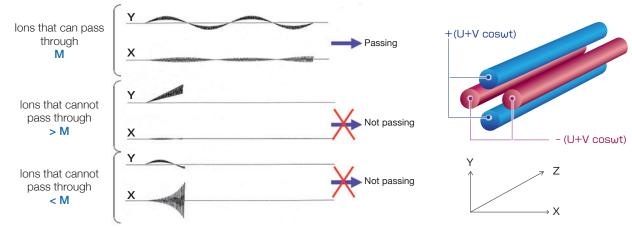
Note: Integrated analysis software - msFineAnalysis for JEOL GC-TOFMS and msFineAnalysis iQ for JEOL GC-QMS/TQMS - performs the qualitative analysis workflow above automatically.

What is the difference in obtaining data between QMS/TQMS and TOFMS?



QMS/TQMS uses a transmission quadrupole mass analyzer to separate ions, and switches between scan mode and SIM mode depending on the purpose. TOFMS always acquires a mass spectrum.

Quadrupole mass analyzer (QMS/TQMS): $m/z \propto RF$ voltage (V), DC voltage (U)

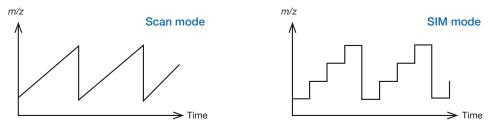


Scan mode

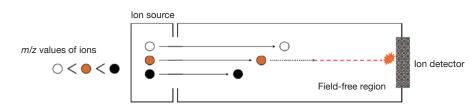
In the transmission quadrupole mass analyzer, by appropriately setting the RF voltage (V) and DC voltage (U) applied to the quadrupole, only ions with a specific m/z can be transmitted. Mass spectra can be sequentially obtained by repeatedly scanning the RF voltage (V) and DC voltage (U) while maintaining the ratio of V and U constant. From the data obtained, the mass spectrum at any time and the EIC of any m/z can be obtained as shown in P. 2. Qualitative analysis (identification) of a compound can be performed from the mass spectrum, and quantitative analysis can be performed from EIC.

SIM (Selected Ion Monitoring) mode

Measurement is performed by repeatedly changing the RF voltage (V) and DC voltage (U) in a stepwise manner so that one or more preset m/z ions can be sequentially transmitted. High sensitivity can be obtained because the time during which a specific m/z ion is transmitted is much longer than in scan mode. The data obtained consists only in chromatograms of pre-specified m/z ions (SIM chromatograms) and cannot be used for qualitative analysis. This data is used for quantitative analysis of trace substances whose sensitivity is insufficient in scan mode.



Time-of-Flight Mass Spectrometer : $m/z \propto [time-of-flight]^2$



When ions with different *m/z* are accelerated at the same voltage at the same time, all the ions acquire the same kinetic energy and fly in a field-free region.

Smaller m/z ions fly faster, and larger m/z ions fly slower, so mass spectra can be obtained by sequentially detecting the ions that reach the detector. Since a single ion flight cycle can be completed in tens of microseconds to hundreds of microseconds, theoretically, it is possible to measure more than 10,000 mass spectra per second, but in reality, an appropriate number of mass spectra are summed and then recorded. It is possible to record up to 50 mass spectra per second.

How can a quantitative analysis be performed with GC/MS?

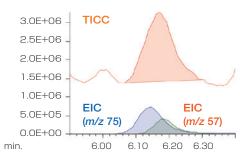


An analytical method for investigating the concentration of a target component contained in a sample. Quantitative analysis is made possible by creating EICs of the ions derived from the target components of data measured by QMS/TQMS scan modes and by TOFMS. If the sensitivity is insufficient in scan mode, the measurement is performed in the SIM mode with QMS/TQMS. If the sensitivity and selectivity are insufficient even in SIM mode, it is necessary to perform measurement in SRM mode (described later) of TQMS.

Quantitative analysis from scan mode data and TOFMS data

EICs are created by specifying the m/z of ions with strong intensity and specific to the components to be analyzed. Then, the quantitative value can be calculated from the area of the peaks on the EICs.

Note: When two components overlap as shown in the figure, they can be quantified by creating EICs of m/z 57 and 75, which are specific to the components.



Quantitative analysis by SIM (Selected Ion Monitoring)

If the sensitivity is insufficient in scan mode measurement, measurement needs to be performed in SIM mode. Ions with strong intensity and specific to the component to be analyzed are identified by measuring the standard sample in advance in scan mode. Then, measurement can be performed for the samples for calibration curve and the actual samples under the SIM measurement conditions that monitor only these ions.

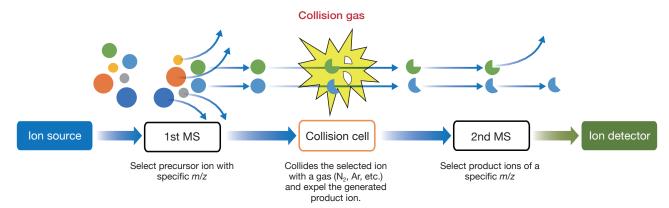
Quantitative analysis by SRM (Selected Reaction Monitoring)

SRM is a measurement mode specific to TQMS. It is highly selective and is effective for multi-component analysis where complete component separation by GC is not possible, and for sample analysis where it is difficult to ensure the reliability of quantitative results due to a large number of matrices.

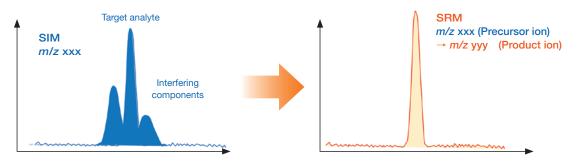
A TQMS (Triple Quadrupole Mass Spectrometer) is a tandem mass spectrometer consisting of two quadrupole mass analyzers, the 1st MS and the 2nd MS.

Specific precursor ions selected in the 1st MS are made to collide with the collision gas, and the specific product ions generated are selected in the 2nd MS and monitored.

Since the mass separation of specific ions is performed twice in this way, more selective and reliable quantification results can be obtained than in the SIM mode in which mass separation is performed only once with the 1st MS.



SRM enables highly sensitive and reliable quantitative analysis even for samples containing a large number of matrices, where the *m/z* of the target analyte and the interfering components overlap and cannot be accurately quantified using SIM mode.



Application examples of JMS-Q1600GC UltraQuad[™] SQ-Zeta

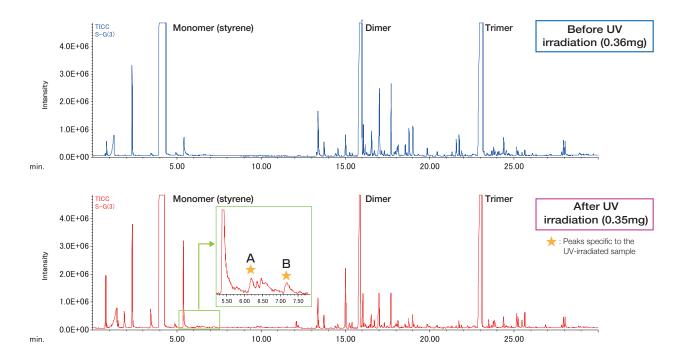
Qualitative Analysis

Ultraviolet (UV) photodegradation analysis of styrene foam using a pyrolyzer

Styrene foam samples before and after UV irradiation were measured by pyrolysis (Py)/GC/MS. El and PI were used for ionization.

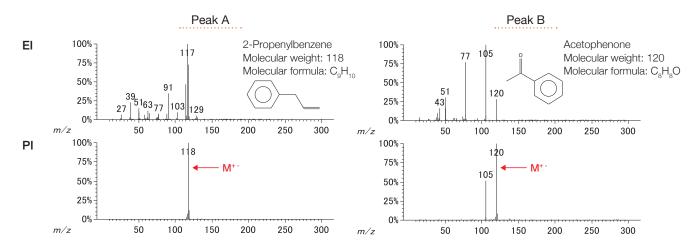
TICC (EI)

 \cdot The peaks specific to the UV-irradiated sample were found.



Mass spectra at the peaks specific to the UV-irradiated sample

• The molecular weights suggested by NIST library search for the El mass spectra were confirmed by Pl as the molecular ions were detected as base peaks in the Pl mass spectra.





Quantitative Analysis

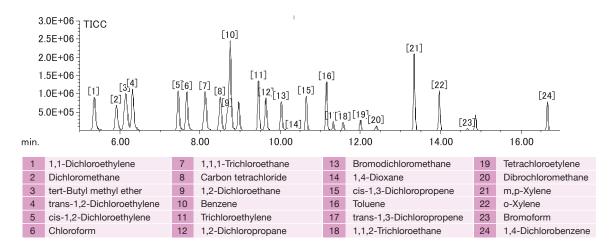
Analysis of volatile organic compounds (VOCs) in water

VOCs in water were analyzed using headspace (HS)-GC-MS, by referring to JIS (Japanese Industrial Standards) K 0125:2016 which describes the testing methods for VOCs in industrial water and waste water. The calibration curves were established by measuring the standard samples (0.05, 0.1, 0.5, 1, 5, 10, 50 µg/L).

TICC (EI)

 \cdot Shown below is the TICC from the 5 μ g/L standard sample.

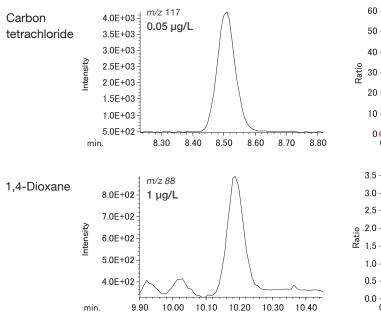
· 24 VOC components were detected with good chromatographic separation.



Calibration curves

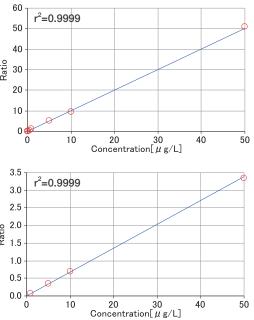
 \cdot The EICs and calibration curves for 1,4-dioxane and carbon tetrachloride, which are difficult to detect among the VOCs, are shown.

· Good chromatographic peak shapes and good linearities of the calibration curves were achieved.



SIM chromatograms

Calibration curve



Application examples of JMS-TQ4000GC UltraQuad[™] TQ



Simultaneous quantitative analysis of pesticide residues in food completed in 15 minutes

One of the major challenges in the analysis of pesticide residues in food is to process as many samples as possible within a limited time. The JMS-TQ4000GC UltraQuad[™] TQ is equipped with 2 SRM modes – a high-speed mode and a high-sensitivity mode – based on "short collision cell" and "ion accumulation" technologies. In high-speed mode, up to 1000 ions can be quantitatively measured per second.

SRM mode	High-speed mode	High-sensitivity mode
SRM speed	1,000 channels/sec	100 channels/sec
Ion accumulation period	1 ms	10 ms
Application example	Simultaneous analysis of 300 agrochemical residues with Fast GC conditions	Simultaneous analysis of 300 pesticide residues based on the method provided by the Ministry of Health, Labour and Welfare of Japan.

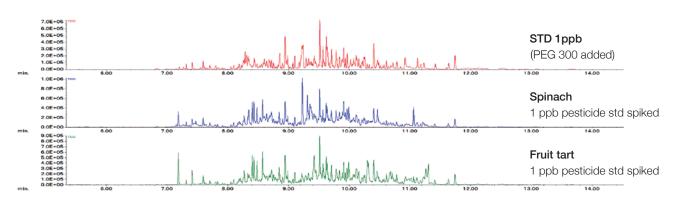
ligh-speed mode: chieved by "short collision cell" echnology

High-sensitivity mode: achieved by "ion accumulation" technology

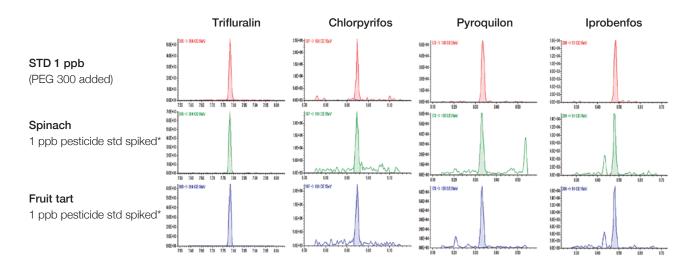
The JMS-TQ4000GC with Fast GC conditions* enabled a simultaneous analysis of 10 ppb pesticides (with an ordinary injection volume of 2 µL) in 15 minutes. Furthermore, sensitivity down to 1 ppb was confirmed.

*Conditions to shorten GC analysis time by using a shorter and narrower GC column and faster GC oven temperature ramping.

TICC of 292 pesticides (1 ppb each)



SRM chromatograms of 1 ppb pesticides



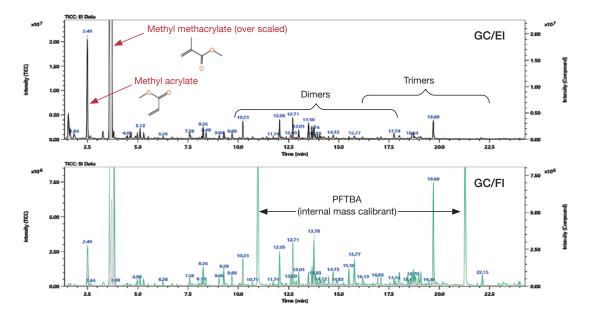
Both standard and spiked samples (*) showed SRM chromatograms with good separation and peak shape, even when the sample concentration was as low as 1ppb.

Application examples of JMS-T2000GC AccuTOF[™] GC-Alpha

Integrated analysis of acrylic resin using pyrolysis/GC/MS

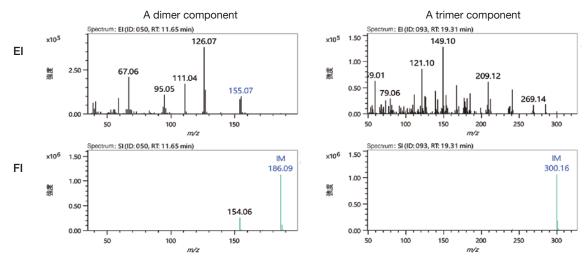
Commercial acrylic resin was measured by pyrolysis (Py)/GC/MS. El and Fl were used for ionization.

TICC of acrylic resin obtained by pyrolysis/GC/MS



The majority of the pyrolysis products of acrylic resin are not registered in library databases, and cannot be identified by library search alone. In addition, there are many components for which molecular ions are difficult to detect using El alone. These molecular ions were detected with high relative intensity using Fl.

Mass spectra of a dimer component (left) and a trimer component (right)



• GC-TOFMS allows for qualitative analysis of compounds that are not registered in the library database (unknown compounds).

• Moreover, msFineAnalysis, a software for automated and integrated qualitative analysis, enables easy analysis of unknown compounds.

Refer to MSTips No. 300 for detail of this application. https://www.jeol.co.jp/en/applications/detail/1874.html



What are the sample preparation devices connected to GC-MS?



GC-MS can analyze components in gas, liquid, and solid samples - in combination with various other sample preparation devices. Here, we introduce popular sample preparation devices, Head Space Sampler (HS), Pyrolyzer (Py), and the thermogravimetry/differential thermal analysis (TG/DTA) system.

Headspace sampler (HS)

A sample (liquid or solid) is placed in a sealed vial and heated. Volatile components in a sample are extracted and distributed between the gas phase and the sample. Then, a portion of the gas phase containing volatile components is introduced into a GC-MS.

Pyrolyzer (Py)

Py/GC/MS is a method used to analyze evolved gases or pyrolysis products generated by heating a sample in a pyrolyzer furnace. It is mainly used for the analysis of polymeric materials, including the identification of polymers and quantification of additives in resin.

Thermogravimetry / Differential Thermal Analysis (TG/DTA)

The TG/DTA system can be used to observe weight changes during the sample heating process and thermophysical properties such as heat generation and heat absorption that the chemical reactions accompany. In addition, by connecting to an MS, gaseous components generated during the heating process can be analyzed.

TG (Thermogravimetry)

A method to measure the weight change of a sample by heating.

DTA (Differential thermal analysis)

A method used to detect the temperature difference between a sample and a reference substance, and observe the exothermic/ endothermic reaction in the sample.

eatures of each sample preparation device

	HS	Ру	TG/DTA
Maximum heating temperature	250 °C	1,000 °C	1,600 $^{\circ}\text{C}^{*}\text{varies}$ depending on the model.
Maximum sample volume	Approx. 20 g (Container volume: up to ~20 mL)	Several tens of milligrams (Container volume: up to ~80 µL)	Approx. 1 g (Container volume: up to ~400 µL)
Sample state	Solid, liquid, gas	Solid, liquid	Solid, liquid
Usage and analysis target	Volatile components in the sample	Polymers and additives	Volatile components that evolve in sync with weight change



JMS-T1600GC UltraQuad™ SQ-Zeta and MS-62071STRAP headspace autosampler



JMS-T1600GC UltraQuad™ SQ-Zeta and EGA/PY-3030D multishot pyrolyzer (Frontier Lab)



JMS-T2000GC AccuTOF™ GC-Alpha and STA-449 TG/DTA (Netzsch)

JEOL Mass Spectrometers - product/lineup

Gas Chromatograph - Quadrupole mass spectrometer (GC-QMS) JMS-Q1600GC UltraQuad™ SQ-Zeta

The UltraQuad[™] SQ-Zeta is our 6th generation high-end GC-QMS based on JEOL's 50 years of MS technology and experience. In addition to the standard high-semisitivity El ion source (IDL 5 fg or below), the JMS-Q1600GC can be equipped with the Enhanced Performance Ion Source (EPIS). Moreover, an integrated qualitative software system, "msFineAnalysis iQ" for GC-QMS, which applies the methodology in the msFineAnalysis software for GC-TOFMS, is available. The SQ-Zeta is the ultimate general-purpose GC-MS for a wide variety of analytical needs, from quantitative applications such as environmental analyses, water quality control and agrochemicals, to qualitative applications such as materials and aroma analyses.



High Performance Gas Chromatograph - Time-of-Flight Mass Spectrometer JMS-T2000GC AccuTOF[™] GC-Alpha

The JMS-T2000GC AccuTOF™ GC-Alpha is a high performance GC-MS system that simultaneously realizes high mass resolution, high mass accuracy, high sensitivity, high speed data acquisition, a wide dynamic range, and a wide mass range. Two types of multi-ionization combination ion sources - El/FI/FD combination ion source and an El/PI combination ion source - are available. Soft ionization methods can produce molecular ions from unknown compounds whose molecular ions cannot be produced by El, and facilitate the elucidation of their elemental compositions. The system comes with the automatic data analysis software "msFineAnalysis" for integrated analysis of the data measured by the multi-ionization combination ion sources, thus providing fast automatic qualitative analysis which does not solely depend on mass spectral library databases.



Ultra-High Mass-Resolution MALDI-TOFMS System JMS-S3000 SpiralTOF[™]-plus 2.0

The JMS-S3000 SpiralTOFTM-plus 2.0 is a MALDI-TOFMS* incorporating JEOL's unique SpiralTOFTM ion optical system. With capabilities superior to conventional MALDI-TOFMS, the JMS-S3000 provides state-of-the-art analytical solutions for a wide range of research needs, including functional synthetic polymers, materials chemistry, and biomolecules.

* Matrix-Assisted Laser Desorption/Ionization Time-of-Flight Mass Spectrometer



Gas chromatograph - Triple Quadrupole mass spectrometer (GC-QMSMS) JMS-TQ4000GC UltraQuad™ TQ

The JMS-TQ4000GC GC-QMSMS is equipped with a short collision cell developed by JEOL's original technology, which achieves both high sensitivity and high throughput, while improving upon the already excellent features of JEOL's QMS. Changing the accumulation time allows multi-component simultaneous analysis in the same concentration range of calibration curve. High-sensitivity analysis with high reproducibility can be performed for pesticide residues in food, pesticides in water, and dioxins.



A Mass Spectrometer Dedicated for Dioxin Analysis JMS-800D UltraFOCUS™

The JMS-800D UltraFOCUS[™], conforming to international standards on dioxin analysis, including EPA, EN, and JIS methods, focuses on analysis of ultra-trace amounts of dioxins, PCBs, PBDEs, and POPs components. Equipped with a socket-type ion chamber, a standard sample inlet system with automatic ON/OFF function, and a photo-multiplier detector, the JMS-800D is optimum for detecting dioxins with ultrahigh sensitivity and ultrahigh selectivity.

(Note: not available in some countries or regions. Please inquire our local sales representative for details.)



Atmospheric Pressure Ionization High-Resolution Time-of-Flight Mass Spectrometer JMS-T100LP AccuTOF[™] LC-Express

The AccuTOF™ LC-Express, the fourth generation of the successful AccuTOF™ LC series, is a simple, robust and versatile atmospheric pressure ionization high-resolution time-offlight mass spectrometer (API-HRTOFMS). It can provide solutions for a wide variety of fields with JEOL's unique ionization technologies, DART and ColdSpray, in addition to the standard electrospray ionization (ESI), the most widely used ionization technique for LC/MS.

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