Applications note

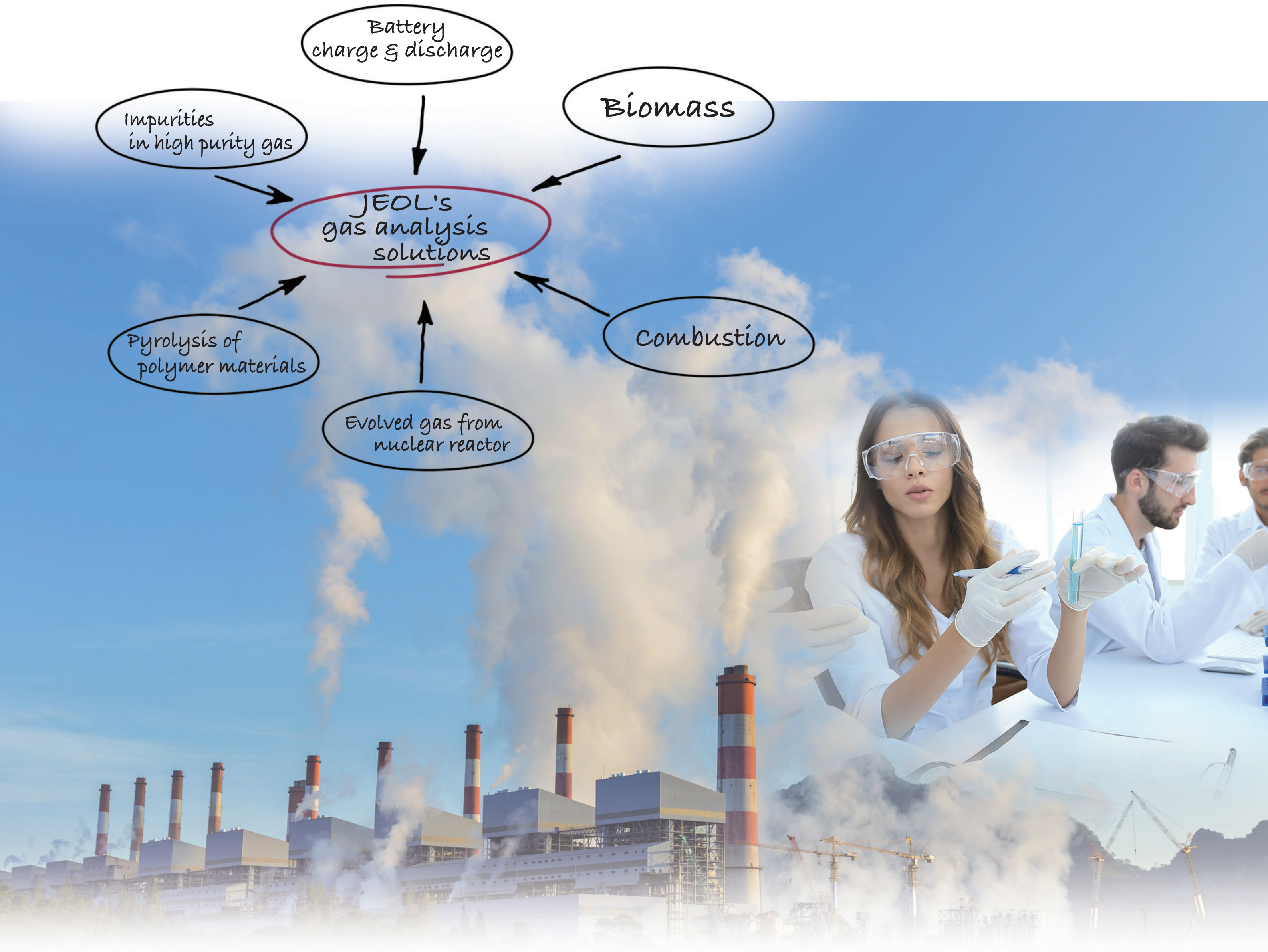
Gas analysis solutions

Solutions for Innovation

Gas analysis

Gas analysis Solutions with JEOL Mass Spectrometers

GC-QMS, High performance GC-TOFMS,
Multi-turn TOFMS, TG/DTA-MS System,
Pyrolyzer-GC/MS System

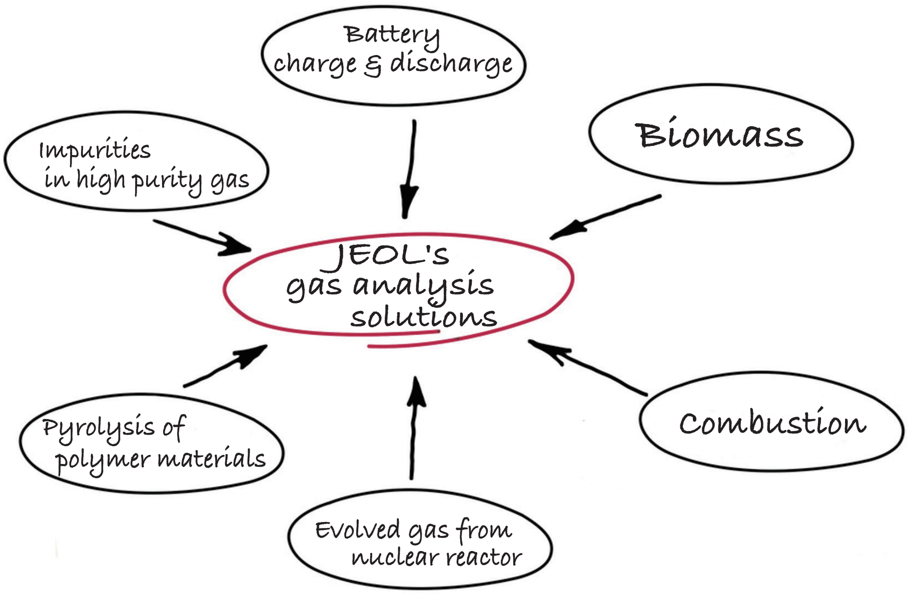


JEOL Ltd.

Gasanalysis

Introduction

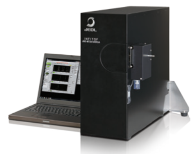
Gas analysis is important for numerous applications including, characterization of impurities in high-purity gases, semiconductor manufacturing, gases released during battery charge and discharge cycles or from biomass, combustion, pyrolysis of polymer materials, and nuclear reactors.
JEOL mass spectrometers offer gas analysis solutions to match the needs of various processes and research fields.



A comparison of JEOL gas analysis systems

GC-QMS
JMS-Q1500GC

High Performance
GC-TOFMS
JMS-T200GC
AccuTOF™ GCx-plus



Multi-Turn TOFMS
JMS-MT3010HRGA
INFITOF

Mass resolution	2,000	10,000	30,000 *1	Applications
Separation of nitrogen (N_2) and carbon monoxide (CO_2) (*m/z* 28)	✗	✓✓	✓✓✓	Quantitation of carbon monoxide (CO) as an impurity in a high purity gas. Tracking a reaction that generate or consume CO. (Quantitation of CO without being affected by nitrogen (N_2) background.)
Separation of nitrous oxide (N_2O) and carbon dioxide (CO_2) (*m/z* 44)	✗	✗	✓✓✓	Simultaneous analysis of nitrous oxide (N_2O) and carbon dioxide (CO_2).
Detection of hydrogen related ions				
Hydrogen molecular ion ($H_2^{+\cdot}$; *m/z* 2)	✓✓✓	✗	✓✓✓	Quantitaion of hydrogen (H_2) as an impurity in a high purity gas. Thermal desorption of H2 from metal. (EGA-MS, TG/DTA-MS)
Hydron (hydrogen atomic ion) (H^+, *m/z* 1)	✗	✗	✓✓✓	Tracking a reaction involving hydrogen radicals.
Transportability	✓	✗	✓✓	On-site analysis in the vicinity of the gas source.
High flow rate introduction	✓✓✓ *2	✓	✓✓ *2	Tracking a rapid reaction. Monitoring combustion.
Prolonged continuous monitoring	✓✓✓	✓	✓✓✓	QA/QC of a high purity gas. Prolonged monitoring of a reaction process.
EGA (evolved gas analysis) (with multi-functional pyrolyzer)	✓✓✓	✓✓✓	* *3	Testing thermophysical properties of an organic or inorganic material.
TG/DTA-MS	✓✓✓	✓✓✓	✓✓✓	
GC/MS with a packed column	✓✓✓	✗	✗	Analysis of impurities in a high-purity gas.
GC/MS with a capillary column	✓✓✓	✓✓✓	* *3	

Legend
✓✓✓: Excellent
✓✓: Good
✓: Possible
X: Not possible

*1 Mass resolution is variable according to the flight cycles.
*2 Optional high volume rotary pump or scroll pump is required.
*3 Please inquire.

Quadrupole Mass Spectrometer

JMS-Q1500GC is the fifth generation of the JEOL's GC-QMS system. It employs large hyperbolic quadrupole and high capacity vacuum pumping system, enabling stable quantitative analysis of hydrogen. Since it achieves high sensitivity under high carrier gas flow rate, it is ideal for GC/MS analysis with packed column, on-line connection with thermogravimetry (TG) and temperature programmed desorption (TPD).

GC-QMS
JMS-Q1500GC

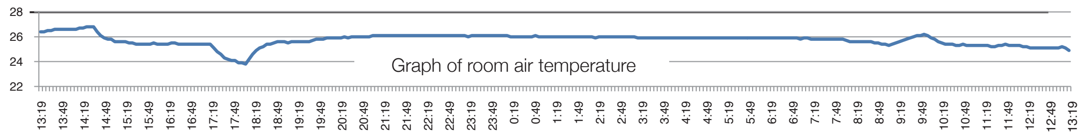
The quadrupole GC-MS system can analyze gas, liquid and solid samples and detect volatile compounds in the specimen and/ or generated from the specimen.
A variety of applications are available to analyze inorganic gases, organic compounds in solution and additives included in materials such as resin.

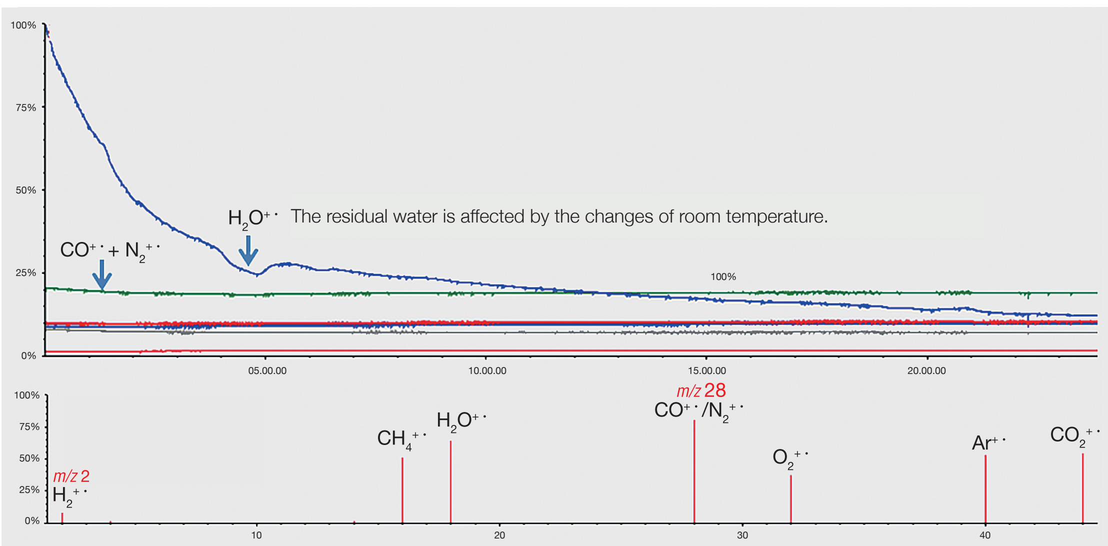
Analysis example — Prolonged monitoring of a standard gas

Sample : Standard gas; 10 ppm each of H_2, O_2, N_2, CH_4, Ar, and CO_2 in He

m/z 28 : $CO^{+\cdot}/N_2^{+\cdot}$ ⇒ Both species contribute to the peak height because they are not separated at the mass resolution of a quadrupole mass spectrometer.

m/z 18 : $H_2O^{+\cdot}$ ⇒ Water in the vacuum chamber and tubing.





Maximum time for one measurement is 24 hours. Monitoring longer than 24 hours is possible by automatically repeating the measurement.

Analysis example

Analysis of trace hydrogen in a gas mixture by using packed-column gas chromatography

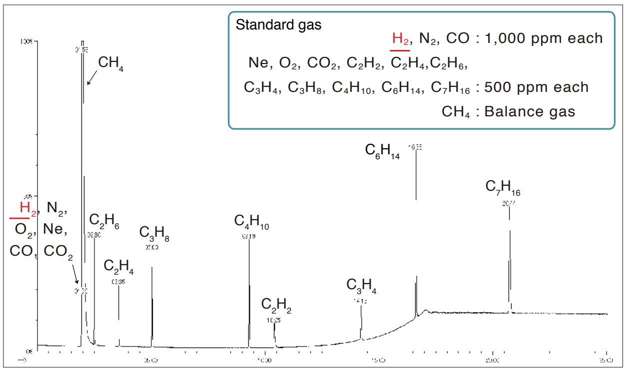
Chromatography maximizes the utility of the system for the analysis of gas mixtures. JMS-Q1500GC with high capacity vacuum pumping system can easily handle high carrier gas flow late of packed GC columns, which are essential for the analysis of trace components in gas mixture.

Analysis condition

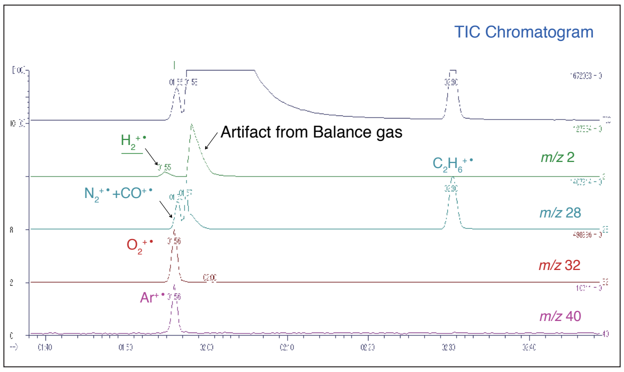
MS Condition	
Ionization mode	EI (17 eV, 30 μA)
Mass range	*m/z* 2-200
Ion source temperature	200 °C
GC interface temperature	200 °C

GC condition	
Injection mode	Direct to packed column
Inlet temperature	200 °C
Column	A proprietary packed column
Oven temperature	40 °C (5 min) → 8 °C /min → 220 °C
Sample injection	6-port valve Loop size:250 μL

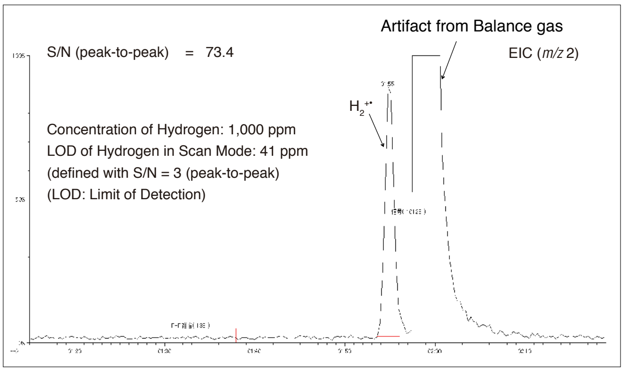
TIC Chromatogram (scanning mode)



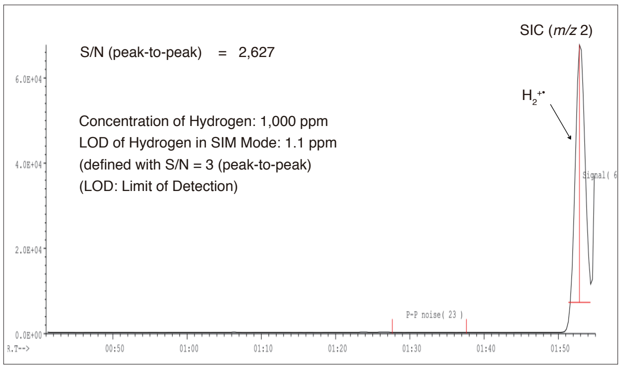
Extracted Ion Chromatogram (scanning mode)



Extracted Ion Chromatogram (scanning mode) of $H_2^{+\bullet}$ (*m/z* 2)



Selected Ion Chromatogram (SIM mode) of $H_2^{+\bullet}$ (*m/z* 2)



Scan measurement for identifying unknown compounds and selected ion monitoring (SIM) for target compound quantitation can be performed simultaneously. (SCAN/SIM function)

High Performance Gas Chromatograph Time-of-Flight Mass Spectrometer

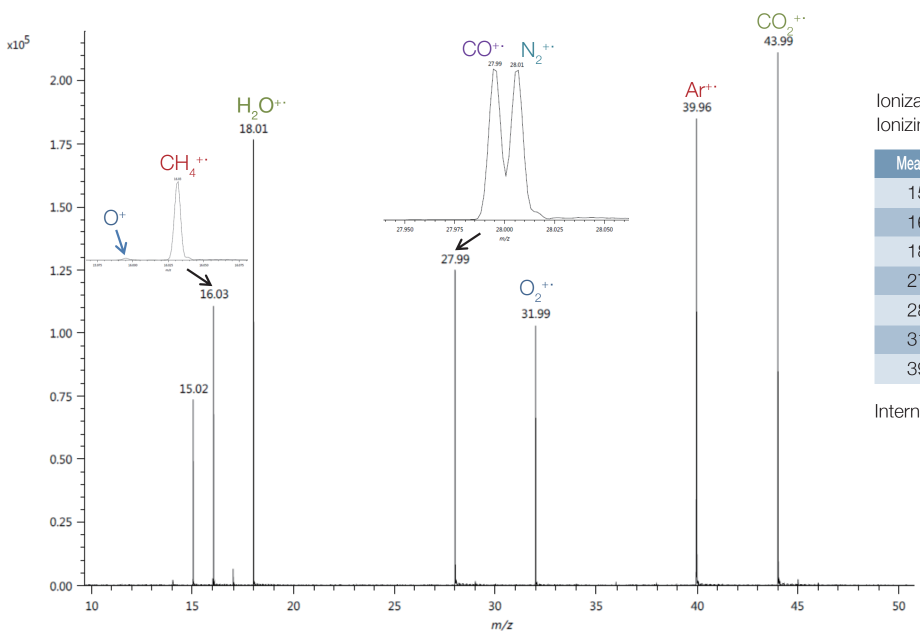
JMS-T200GC AccuTOF™ GCx-plus is the 5th generation of JEOL's successful AccuTOF™GC series of GC-HRTOFMS systems. With the patented ion transfer system that achieves high ion transmission without using radio-frequency-driven ion guide, high sensitivity is achieved even for low molecular weight gas components. It is challenging to achieve high mass-resolving power at low *m/z* region on a short flight path time-of-flight mass analyzer. JMS-T200GC AccuTOF™ GCx-plus employs high speed (4 gigasamples/s) digitizer and newly developed high speed ion detector, achieving high mass-resolution and excellent mass spectral peak shape even in low *m/z* region.
Several ion sources are available including Electron Ionization (EI, standard), and optional Chemical Ionization (CI), Field Ionization (FI), and Photoionization (PI) sources. This offers the widest variety of analyses for volatile compounds.

High performance GC-TOFMS
JMS-T200GC AccuTOF™ GCx-plus

The High Performance Gas Chromatograph Time-of-Flight Mass Spectrometer can acquire mass spectra with high sensitivity and high speed while providing high mass resolving power and mass accuracy for samples introduced by gas chromatography.

Analysis example

Accurate Mass Measurement: mass spectrum of a standard gas mixture.



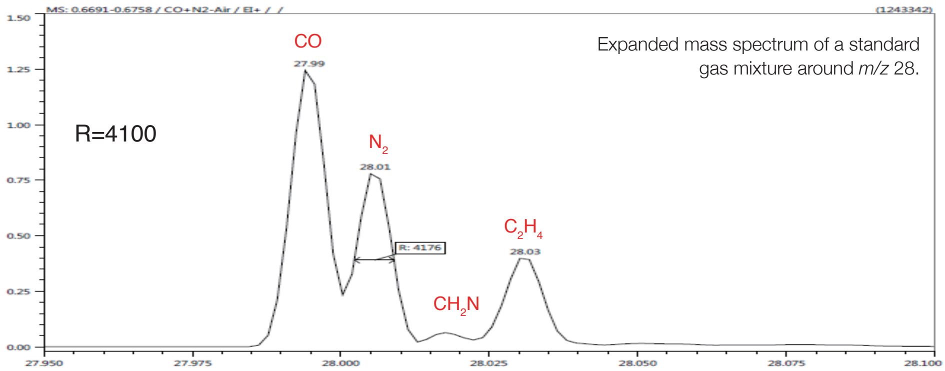
Ionization : EI
Ionizing voltage : 22 V

Measured mass	Elemental composition	Calculated mass	Error (mDa)
15.9951	O^+	15.9944	0.7
16.0314	$CH_4^{+\cdot}$	16.0308	0.6
18.0107	$H_2O^{+\cdot}$	18.0100	0.7
27.9950	$CO^{+\cdot}$	27.9944	-0.6
28.0061	$N_2^{+\cdot}$	28.0056	0.5
31.9898	$O_2^{+\cdot}$	31.9893	0.5
39.9620	$Ar^{+\cdot}$	39.9618	0.2

Internal mass calibration with *m/z* 43.9893 ($CO_2^{+\cdot}$)

Analysis example

Separation of isobaric components at *m/z* 28



Expanded mass spectrum of a standard gas mixture around *m/z* 28.

Even at *m/z* as low as 28, mass resolution sufficient to resolve CO and N_2, and excellent mass spectral peak shapes are achieved.

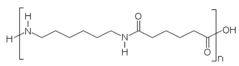
Analysis example

Evolved gas analysis (EGA) of nylon 66 (PA66) by using a multi-functional pyrolyzer

When synthetic polymers are heated to high temperature, low molecular weight gas evolve. With JMS-T200GC AccuTOF™ GCx-plus, elemental composition of each evolved gas component can be elucidated through accurate mass measurement. High mass-resolution extracted ion chromatograms facilitate to monitor time course change of each evolved gas component.

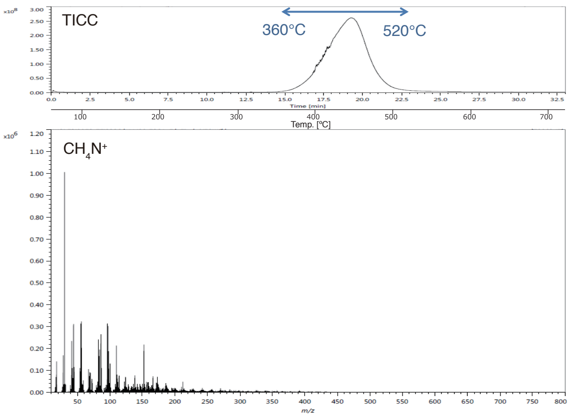
Analysis conditions

Instrument	JMS-T200GC "AccuTOF™ GCx" PY-2020iD (Frontier Laboratories Ltd.)
Py Conditions	
Pyrolysis Furnace Temperature	60°C => 20°C/min => 700°C (a minute)
Py-GC-ITF Temperature	350 °C
GC Conditions	
Inlet temperature	350°C
Injection mode	Split 50 :1
Columns	Deactivated fused silica tube (5 m x 0.25 mm)
Oven Temperature Program	350°C (33 min)
Carrier Gas flow	1 mL/min (He, Constant flow mode)
MS Conditions	
Ionization method	EI(+) ; 25 V, 200 µA
Interface Temperature	350°C
Ion source Temperature	250°C
Spectrum recording intervals	2.5 Hz (0.4 sec / Spectrum)
m/z range	10 ～ 800
Drift Compensation	*m/z* 28.0056 (N_2^{+})
Sample Weight	0.28 mg



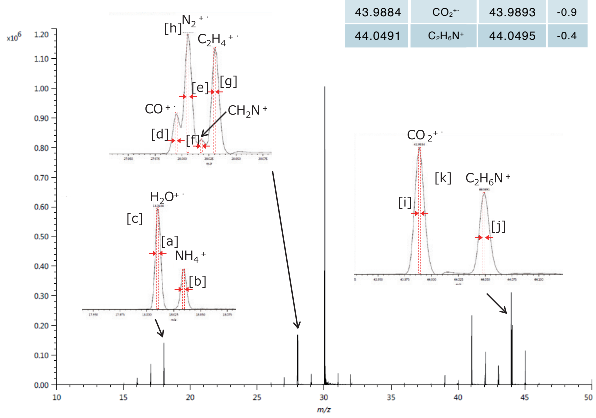
PA66 (Nylon 66)

EGA total ion current thermogram and mass spectrum

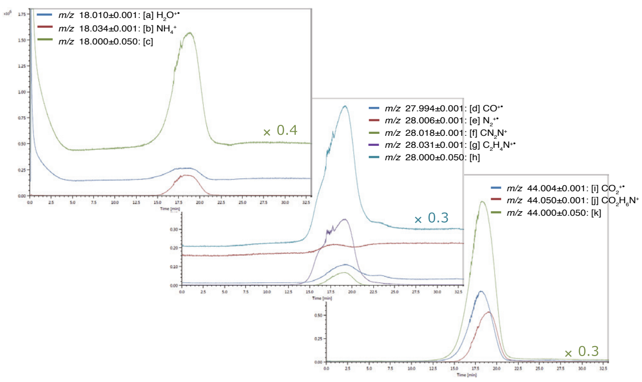


Mass spectrum

Measured Value	Elemental Composition	Calculated Mass	Erorr [mDa]
18.0104	$H_2O^{+\cdot}$	18.0100	0.4
18.0344	NH_4^{+}	18.0338	0.6
27.9950	$CO^{+\cdot}$	27.9944	0.6
28.0180	CH_2N^{+}	28.0182	-0.2
28.0308	$C_2H_4^{+\cdot}$	28.0308	-0.0
43.9884	$CO_2^{+\cdot}$	43.9893	-0.9
44.0491	$C_2H_6N^{+}$	44.0495	-0.4

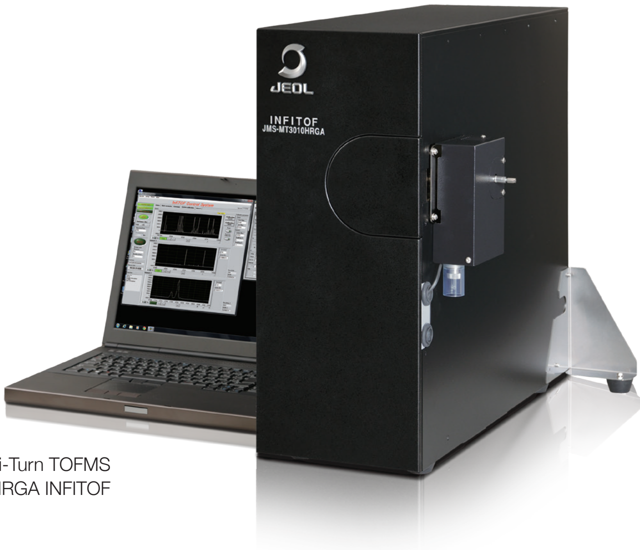


High mass-resolution extracted ion chromatograms



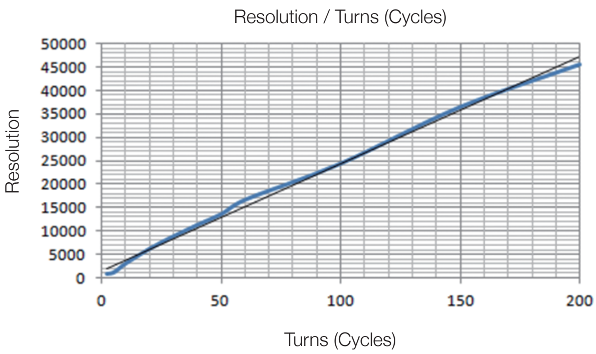
Multi-Turn Time-of-Flight Mass Spectrometer

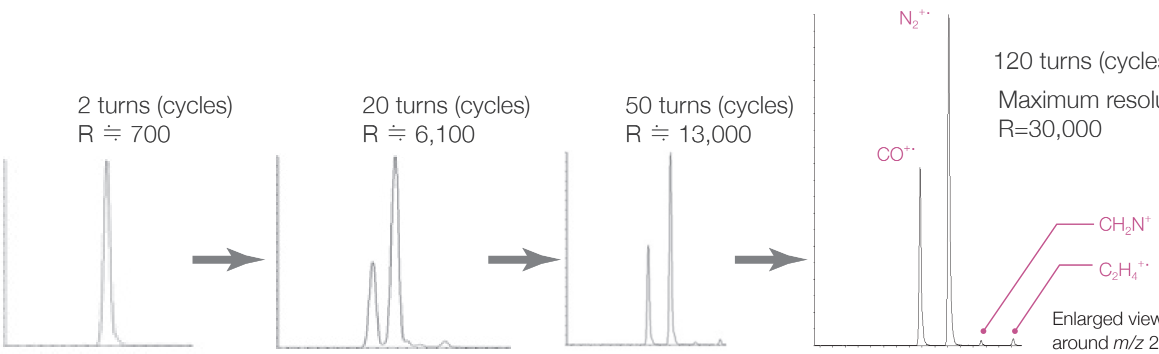
Multi-Turn Time-of-Flight Technology is a breakthrough technology for compact high-resolution mass spectrometers. This compact TOF-MS is designed in response to cutting edge research needs for in-situ realtime analysis of small gas samples. The system offers high mass resolving power for the analysis of advanced materials. With a wide mass range (*m/z* 1-1000), the system is ideal for the research related to next-generation energy sources and hydrogen fuel.



Multi-Turn TOFMS
JMS-MT3010HRGA INFITOF

Analysis example — Mass resolution at *m/z* 28



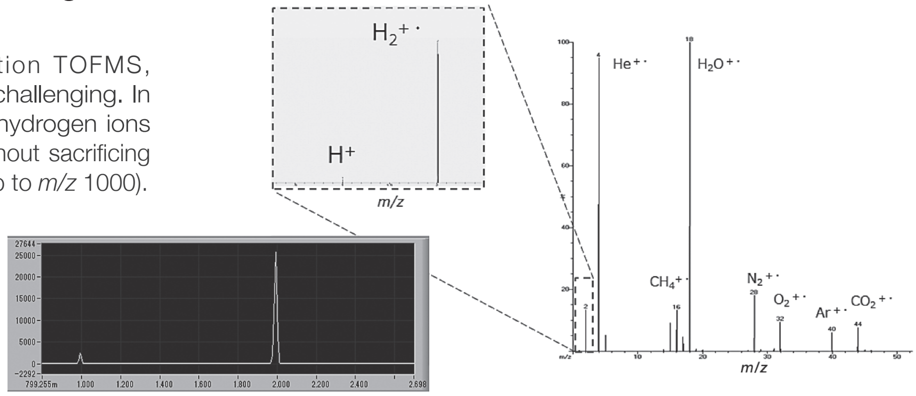


Enlarged view of the Mass Spectrum around *m/z* 28

Analysis example

Detection of hydrogen

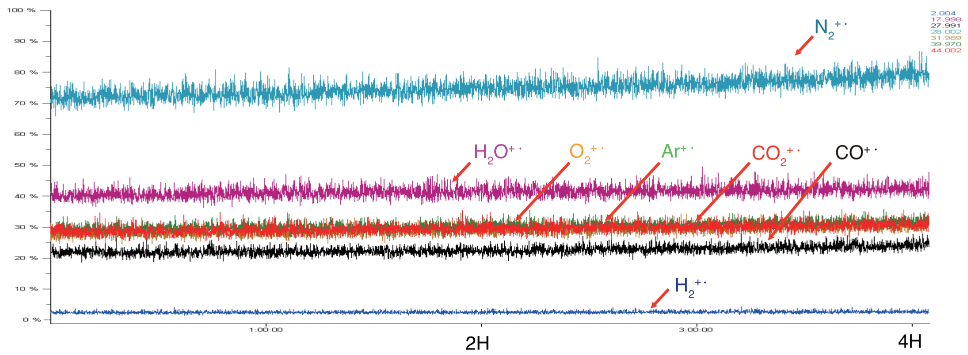
With conventional high-resolution TOFMS, detecting hydrogen ions is highly challenging. In contrast, the INFITOF can detect hydrogen ions (H^+ at *m/z* 1 and $H_2^{+\cdot}$ at *m/z* 2) without sacrificing the ability to detect larger species (up to *m/z* 1000).



Analysis example

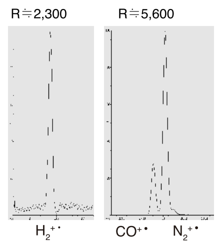
Continuous monitoring of low concentration gas mixture containing hydrogen; Resolution ≈ 5,000 (4 hours)

Stable signal over a 4-hour period for gases including hydrogen Measurement Condition: Ionization: EI, Ionization voltage: 25 eV, Analyzer : 14 turns (cycles), R ≥ 5,000 (*m/z* 28)



Components	
2.01565 (H_2)	10ppm
18.01057 (H_2O)	Vacuum residue
16.03130 (CH_4)	10ppm
27.99492 (CO)	10ppm
28.00615 (N_2)	10ppm
31.98983 (O_2)	10ppm
39.96238 (Ar)	10ppm
43.98983 (CO_2)	10ppm

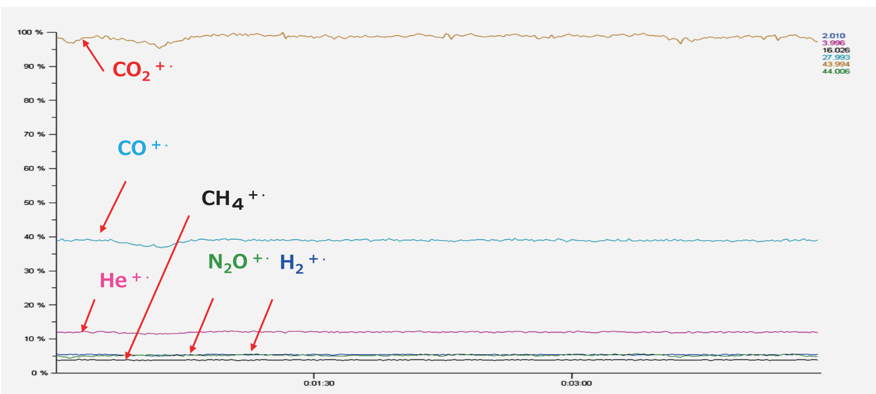
Balance gas: He



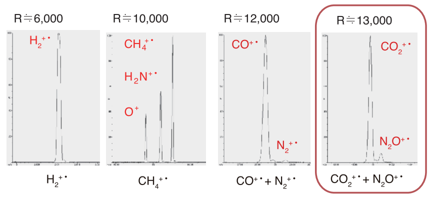
Analysis example

Continuous monitoring of high concentration gas containing hydrogen and N_2O; Resolution ≈ 12,000

Stable analysis of high concentration gas in the % order. Complete separation of N_2O (*m/z* 44.00106) and CO_2 (*m/z* 43.98983) is highly effective for the analysis of combustion products.
Measurement condition : Ionization : EI, Ionization voltage : 25 eV, Analyzer : 30 turns (cycles), R ≥ 12,000 (*m/z* 28)

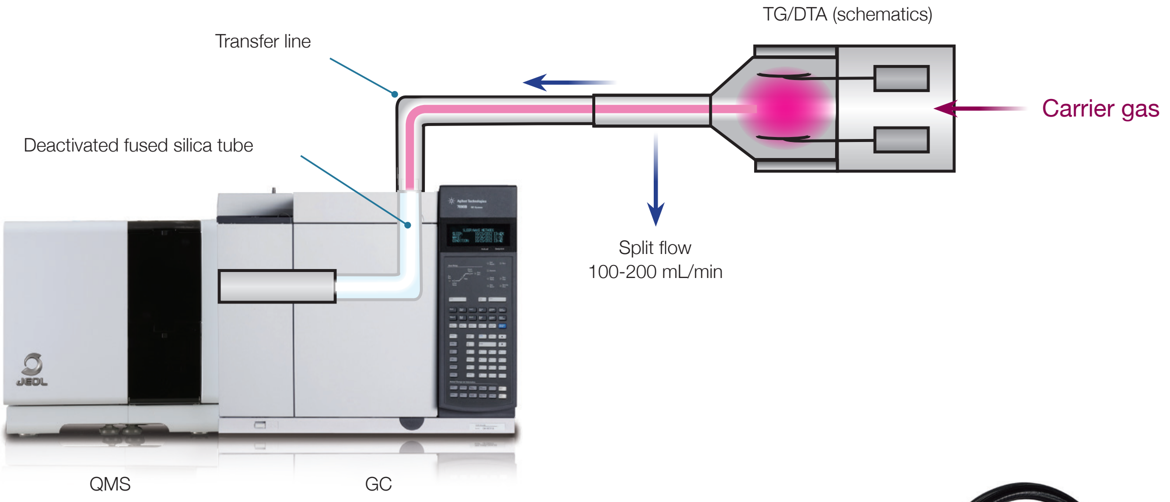


Components	
2.01565 (H_2)	3%
4.002603 (He)	Balance gas
16.03130 (CH_4)	1,000ppm
27.99492 (CO)	0.6%
43.98983 (CO_2)	1%
44.00107 (N_2O)	1,000ppm

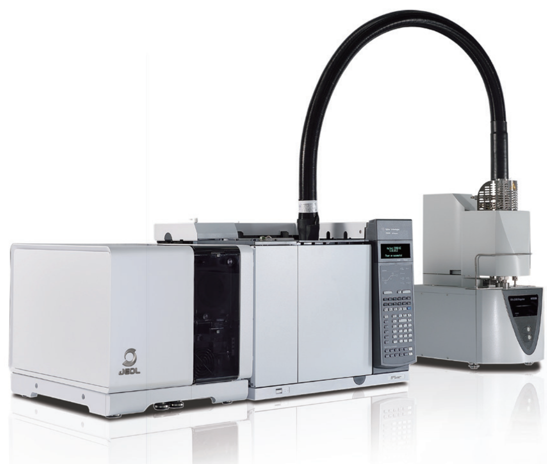


TG/DTA-MS System

TG/DTA (Thermogravimetry / Differential Thermal Analysis) system records the mass changes of and endothermic/exothermic heats to/from the specimen while the temperature of the specimen is increased. By combining the TG/DTA system with a mass spectrometer, components evolved from the specimen can be detected and identified.



JMS-1500GC QMS combined with a TG/DTA manufactured by Hitachi High-Technologies Corporation.



JMS-1500GC QMS combined with a TG/DTA manufactured by NETZSCH-Gerätebau GmbH.

JMS-T200GC AccuTOF™ GCx-plus TOFMS combined with a TG/DTA manufactured by NETZSCH-Gerätebau GmbH.

Pyrolyzer-GC-MS System

A Pyrolyzer-GC-MS System, which combines a Pyrolyzer with a GC-MS, is ideal for the structural analysis of synthetic polymers by pyrolisis and thermal extraction of semi-volatile components in materials. Pyrolysis GC/MS is widely used for materials analysis.

GC-QMS with a multi-functional pyrolyzer,
manufactured by Frontier Laboratories Ltd.

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