

# GCMS Solutions for Pharmaceuticals

## Testing for Residual Solvents in Pharmaceuticals: HS-GCMS System with FID Detector

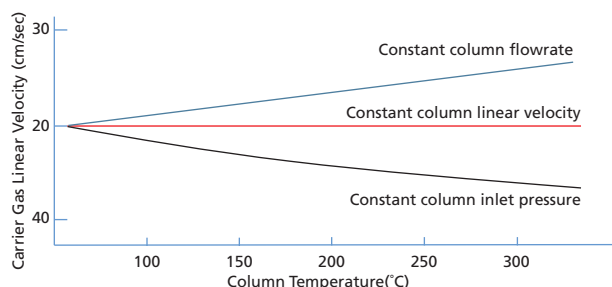
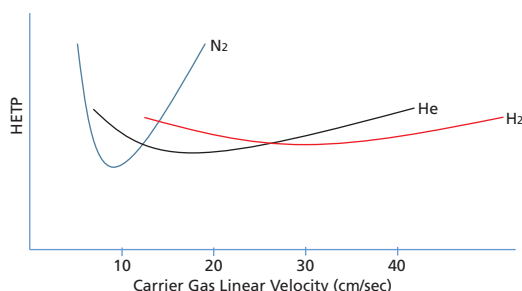
The headspace-GC (FID) method specified in the Japanese Pharmacopoeia is normally used to test for residual solvents in pharmaceuticals. However, unknown peaks not from target solvents sometimes appear during testing. GC-MS can be useful for qualitatively analyzing those peaks.

Since GC-MS systems can be equipped with an FID detector, the same system can be used for both GC-MS and GC analysis.

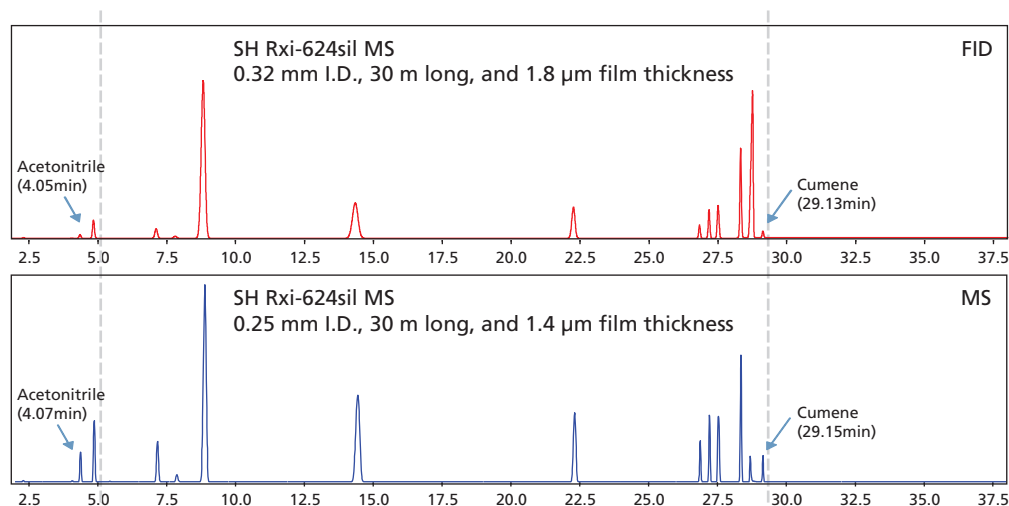


GCMS-QP2020 + HS-20 + FID-2010 Plus Detector

The constant linear velocity control mode is able to maintain an optimal linear velocity. Because the separation efficiency (HETP) of a column varies depending on the column oven temperature, this mode is ideal for temperature-programmed chromatography. The advanced flow controller (AFC) in the GC-2010 Plus system includes a constant linear velocity control mode as standard functionality. Provided the columns have the same phase ratio, it is possible in this mode to obtain GC-FID and GC-MS chromatograms with similar patterns by simply specifying the linear velocity setting.



After using a GC-FID system to acquire data from a Class 2 standard solution A, data was acquired using a GC-MS system with the same linear velocity condition (35 cm/sec). A comparison of the resulting chromatograms shows that the difference in retention times between FID and MS data was 0.02 minutes for acetonitrile, which has a short retention time, and 1.2 seconds for cumene, which has a long retention time. The ability to easily obtain similar chromatogram patterns from FID and MS data acquired using columns with the same phase ratio under the same constant linear velocity conditions means that unknown peaks that appear in FID data can be qualitatively analyzed by comparing the FID data to the MS data.



## Compliance with Data Integrity Requirements: GCMS-LabSolutions Analytical Data Management System

Many users, especially in the pharmaceutical industry, require compliance with regulations and guidelines, such as the CSV, PIC/S GMP, and Japanese Ministry of Health, Labour and Welfare ER/ES guidelines and FDA 21 CFR Part 11. They also are requiring more appropriate and efficient means of maintaining and managing instruments and analytical data. Therefore, starting with version 4.45, GCMSsolution is now compatible with LabSolutions DB/CS, so that LabSolutions can be used to manage analytical files and users for GC-MS systems.

- **Using LabSolutions for Integrated Management of Analytical Instruments, Including GC-MS Systems**

LabSolutions DB/CS systems can reduce data management costs by integrating the management of analytical files and user information for other analytical instruments as well.

- **Excellent Traceability Functionality**

The outstanding functionality for traceability supports maintaining audit trails for analytical files. Analytical files and operation histories are safely managed in a LabSolutions database, so that the necessary information can be easily accessed at any time.

- **Efficient Operation Under High-Security Conditions**

The system includes functionality for automatically locking the system after no operations have been performed for a given period, and for switching between different users during a series of consecutive analyses. These functions help ensure that the system can be operated efficiently under high-security conditions.

### Integrated Management of GC and GC-MS Systems from One PC

LabSolutions DB + GCMSsolution



- **GC-MS System with GC Detector**

The GCMS model, whether or not an HS unit or FID detector is connected, can be changed according to customer preferences. For more information about system configurations, contact your Shimadzu representative.

### Integrated (Networked) Management of Multiple Analytical Instruments

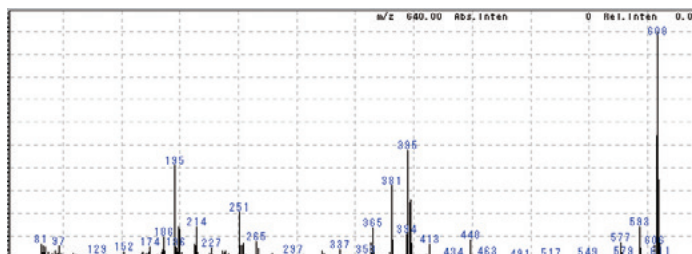
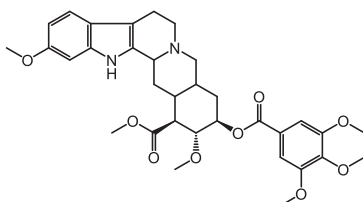
LabSolutions CS + GCMSsolution



## System for Confirming Synthesized Compounds: DI-2010 Direct Sample Inlet Unit



Direct sample injection (DI) is a method of bypassing the gas chromatograph (GC) and injecting samples directly into the ion source. The technique is especially useful for applications such as measuring the mass spectra of synthesized compounds. It is also easy to use; simply attach a direct inlet unit to the GC-MS system configuration normally used. GC is not well suited to analyzing compounds that thermally decompose easily or that are difficult to vaporize, but using the DI method makes it easy to confirm the mass spectra of such compounds.



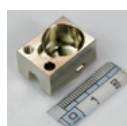
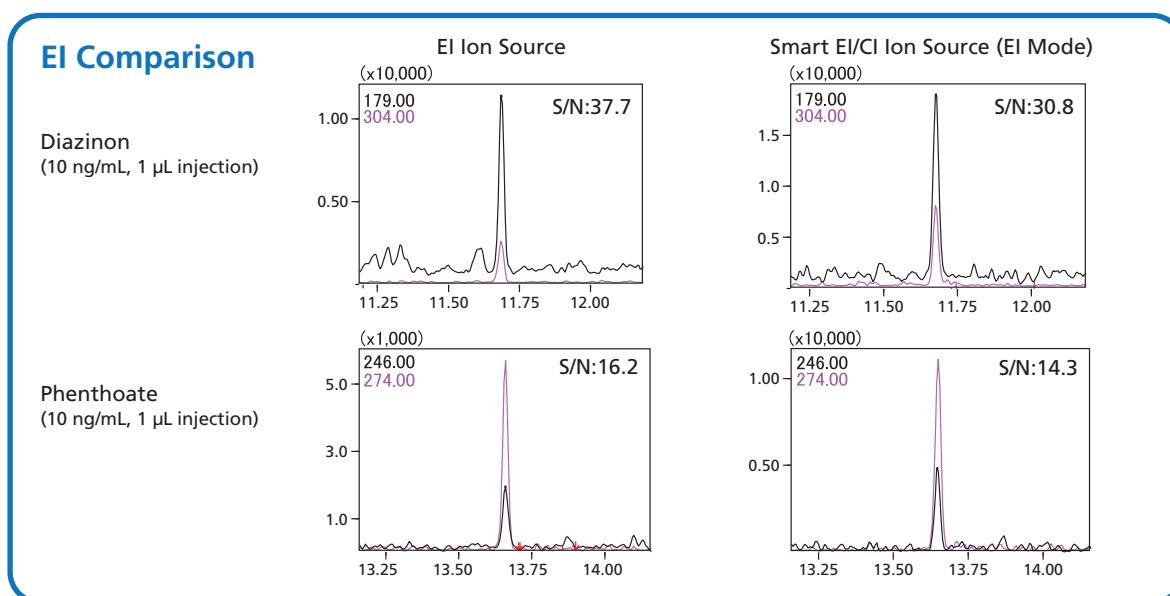
Example of Using DI-El to Analyze Reserpine

## Qualitative Analysis of Unknown Compounds: Smart EI/CI Ion Source

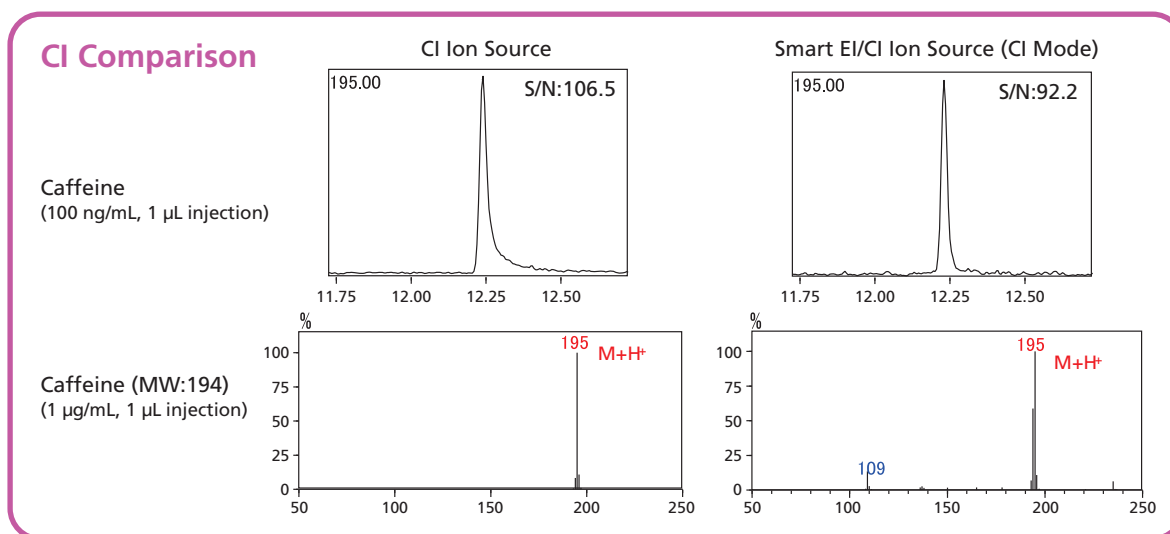
Using a mass spectral library is an extremely convenient and reliable technique for identifying compounds. However, sometimes impurities and newly synthesized compounds are not registered in the library. Positive chemical ionization (CI) has conventionally been used to obtain molecular ion information for qualitative analysis of such compounds, but that requires exchanging ion sources when switching between EI and CI modes.

In contrast, the newly developed Smart EI/CI ion source can be used to acquire data using either EI or CI modes without exchanging ion sources.

- The high sensitivity of the revolutionary ion source results in no noticeable difference compared to using dedicated ion sources, either for EI or CI modes.
- Easily switchable between EI and CI modes without exchanging ion sources.
- In combination with a DI direct sample inlet unit, mass spectra can be acquired easily in either EI or CI mode.



**Smart EI/CI Ion Source**  
Switchable without exchanging ion sources



In the CI mode, using isobutane as the reagent gas is recommended, because it makes it easier to detect protonated molecules. The Smart EI/CI ion source is an optional product. It can be used with GCMS systems capable of chemical ionization.

## High-Sensitivity Analysis of Genotoxic Impurities: GCMS-TQ8050

Genotoxic (mutagenic) impurities must be controlled to low concentration levels. Therefore, high-sensitivity analytical instruments are required for their analysis. Triple quadrupole gas chromatograph mass spectrometers (GC-MS/MS) offer significantly higher selectivity by separating components by mass in two stages. Consequently, they are able to selectively detect trace quantities of genotoxic impurities with high sensitivity.

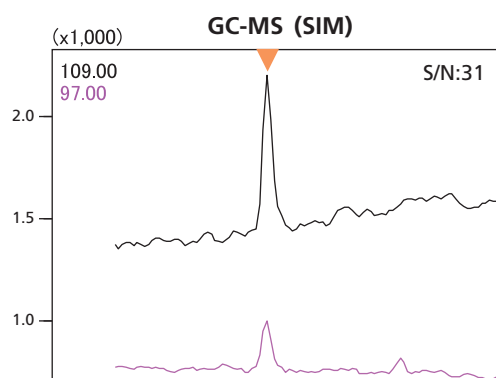
To maximize the benefits of OFF-AXIS ion optics, including both high ion transmission performance and outstanding noise elimination performance, the GCMS-TQ8050 now features a detector with higher amplification performance that achieves the world's highest sensitivity\*.

\* As of August 2016, according to a Shimadzu survey

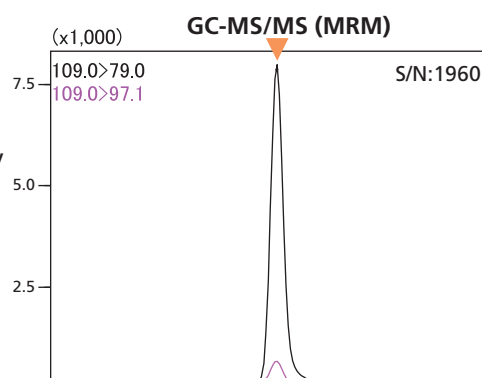
The GCMS-TQ8050 supports measurements as a single-MS system (scan and SIM modes). It is also compatible with almost everything supported by the GCMS-QP2020, including those mentioned above (FID detector, software, DI unit, and Smart EI/CI ion source). Thus, users can configure systems according to their purposes.



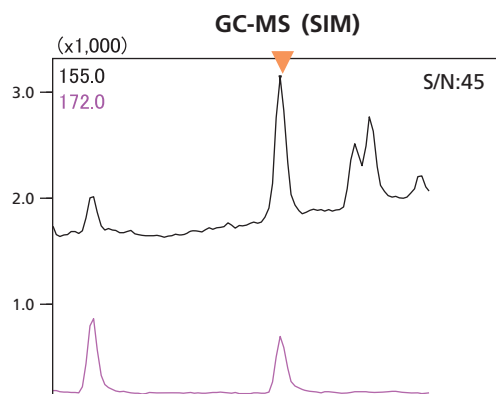
Triple Quadrupole  
Gas Chromatograph Mass Spectrometer  
GCMS-TQ8050



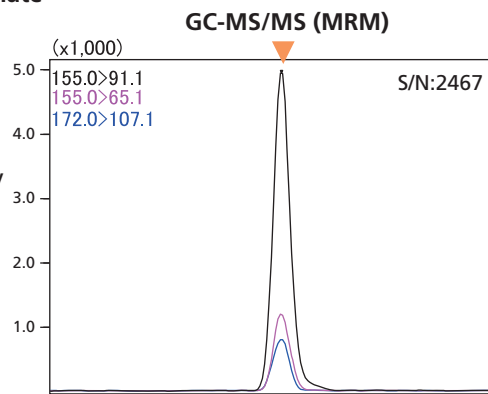
60 times  
higher sensitivity



Ethyl methanesulfonate



50 times  
higher sensitivity



*n*-Propyl *p*-toluene sulfonate

Solution concentration: 0.01 µg/mL [equivalent to a 1 ng/mg (1 ppm) concentration in pharmaceutical drug substances]



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