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Application News
GCMS

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Screening of Agricultural Products for Pesticide Residues using GCMS-QP2010 in Full-Scan Mode, GCMSsolution Software and Shimadzu Pesticides Mass Spectral Library

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GCMS is commonly used for screening of agricultural products for the presence of pesticide residues. This is due to the ability of GCMS to separate the pesticide residues from the other compounds in the agricultural products, and to give mass spectra of the pesticides for confirming the identity of the pesticides. For this purpose, the ionization mode of the GCMS is usually set to Electron impact Ionization, and the acquisition mode is set to Full Scan.

Electron impact Ionization (EI) combined with Full Scan acquisition is good for identification of a compound because the ionization produces an extensive fragmentation for most organic compounds, which in turn gives a characteristic mass spectrum for the compound analyzed.

However, the extensive fragmentation in EI mode and the complexity of the sample matrix (that is, the presence of many other compounds in the vegetable samples) also result in a GCMS chromatogram which can easily contain more than 100 peaks (See **Figure 1**).

Finding the pesticides peaks in such a chromatogram can be a tedious task if we have to analyze the mass spectra one by one. Especially when the target peaks are much smaller than the interference peaks.

GCMSsolution software (Ver. 2) offers a solution to the problem through its Compound Finder feature. An example of the application of Compound Finder is illustrated in this report.

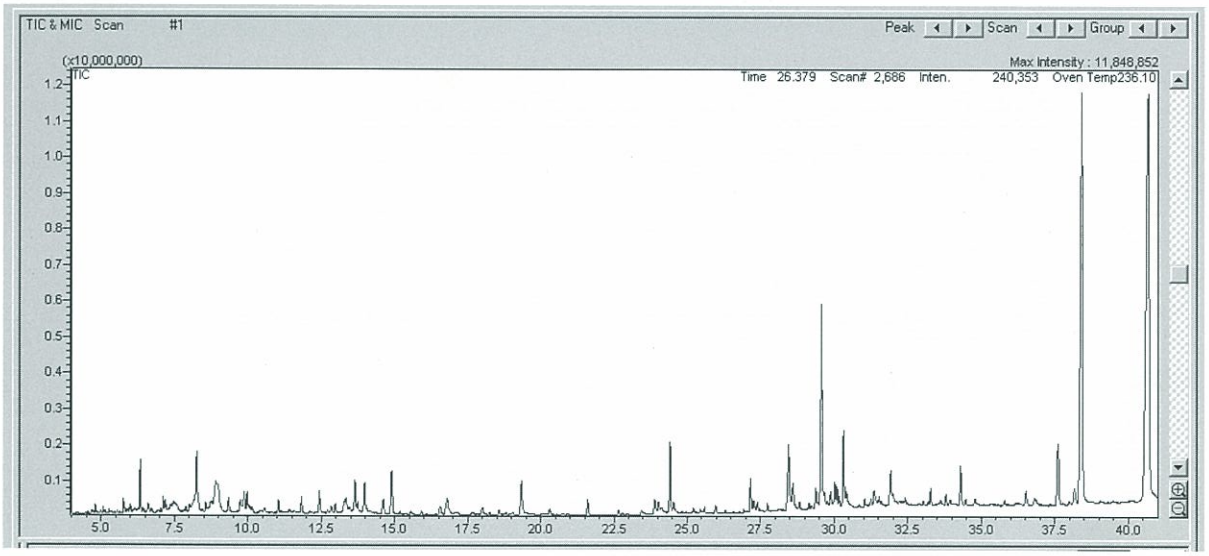

Figure 1. Shown here is a total ion chromatogram obtained from the GCMS (Scan) analysis of a vegetable extract and the search results using Compound Finder.

■ Compound Finder

When the goal of the analysis is to determine whether specific target compounds are present in the sample, the Compound Finder can be used to quickly find the peaks due to these compounds in the complex chromatogram. Compound Finder automatically compares the mass spectra of all the peaks detected in the chromatogram to a set of reference mass spectra of the target compounds. Compound Finder then determines the retention times of the target compounds based on the similarity of the detected peaks' mass spectra and the reference mass spectra.

Since Compound Finder detects the target pesticides *based on mass spectral comparison*, it is more reliable than simply comparing the retention times of the peaks in the sample chromatogram to the standard compounds' retention times alone for a few reasons. First, it is not uncommon for the retention time of a compound to be shifted from the standard retention time in the presence of interfering peaks. Second, retention times may also shift due to instrumental or analysis factors, such as the use of columns of different selectivities or of different lengths. The application of Compound Finder also does not require any instrumental calibration when different analytical conditions are used.

■ Screening of pesticides in sample using Compound Finder

In this application example, Compound Finder is used to determine whether the pesticides Methiocarb and Myclobutanil are present in a vegetable extract (the chromatogram of the sample is shown in **Figure 1**).

The reference mass spectra of the Methiocarb and Myclobutanil are taken from the ***Shimadzu Pesticides Mass Spectral Library***. Compound Finder determines the retention times of the Methiocarb and Myclobutanil peaks in the chromatogram once peak integration is performed (see **Figure 2**).

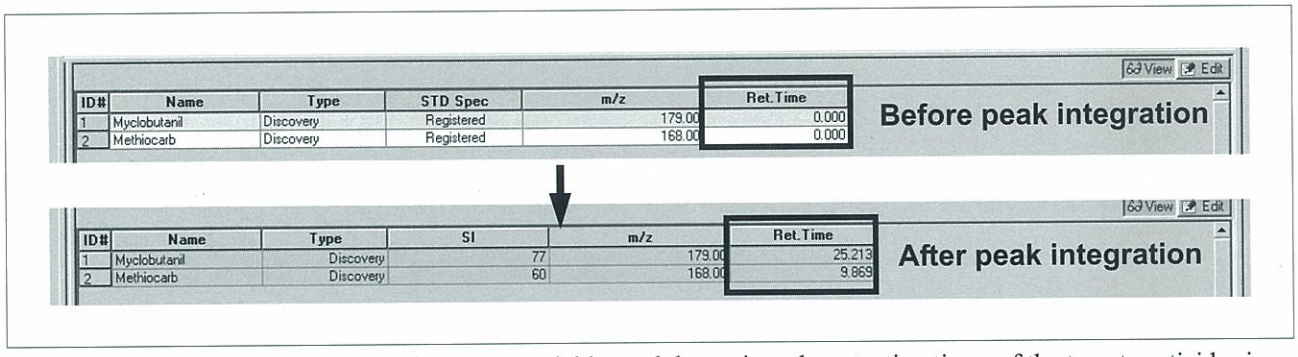

Figure 2. Compound Finder detects the target pesticides and determines the retention times of the target pesticides in the sample chromatogram.

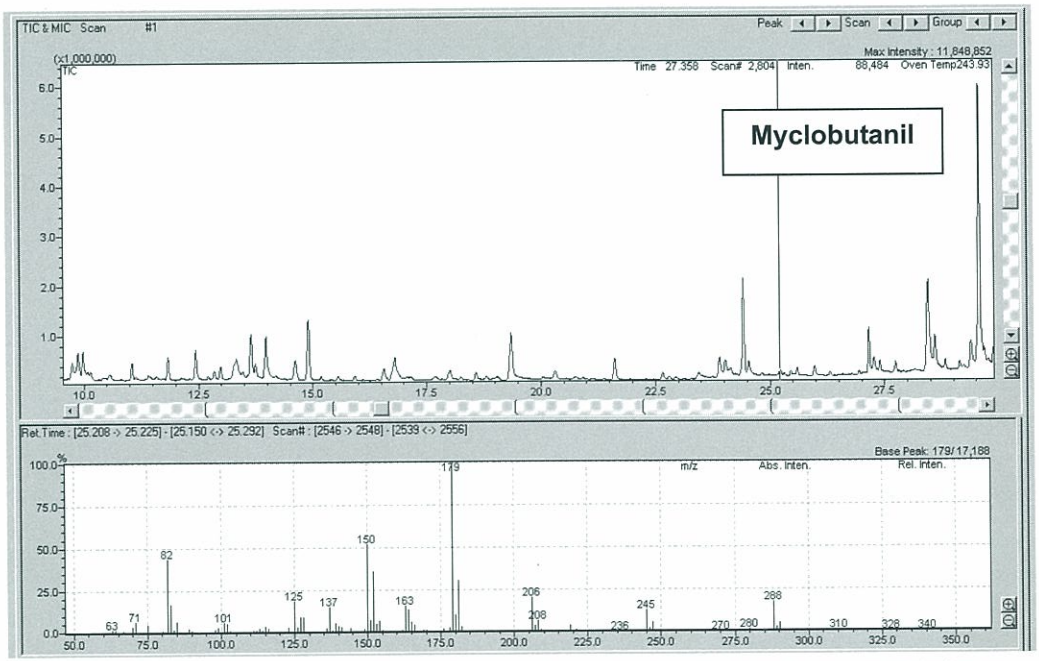

Figure 3. Enlarged chromatogram (top) showing the peak identified by Compound Finder as Myclobutanil, and the mass spectrum (bottom) of the peak.

Further confirmation of the identity of the detected peaks can be performed by using the standard Similarity Search function of GCMSsolution. **Figure 5** shows the Similarity Search results, comparing the mass spectrum of the peak detected by Compound Finder as Myclobutanil (at retention time 25.2 min) and the reference mass spectrum of Myclobutanil in the Shimadzu Pesticide Library.

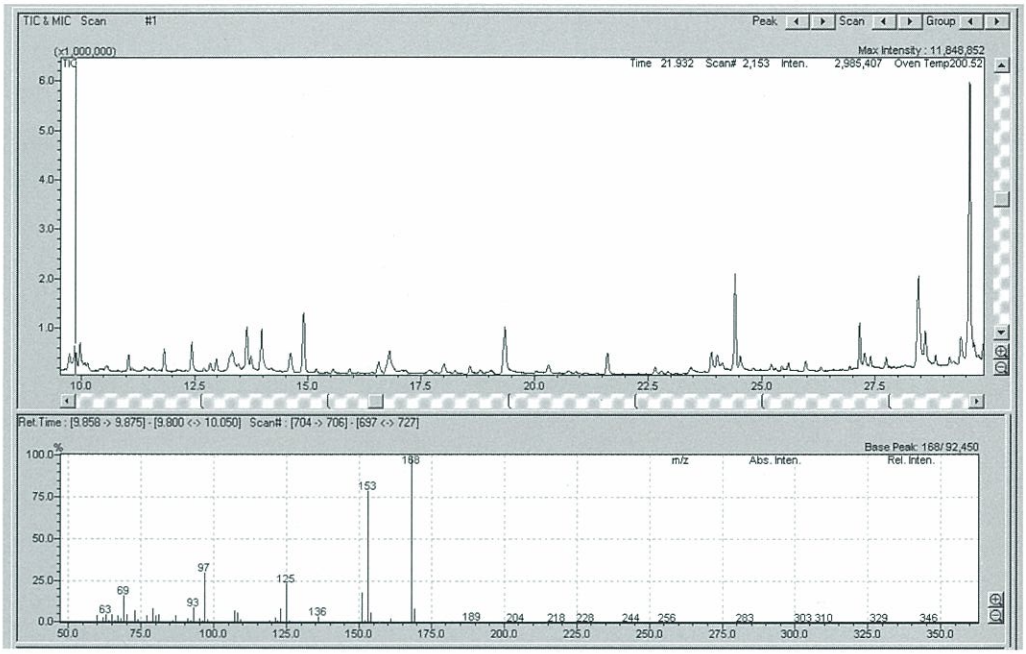

Figure 4. Enlarged chromatogram (top) showing the peak identified by Compound Finder as Methiocarb, and the mass spectrum (bottom) of the peak.

Figures 3 & 4 show the enlarged views of the chromatogram where Myclobutanil and Methiocarb peaks are determined by Compound Finder, and the full-scan mass spectrum of each peak. As shown here, the target peaks are among the minor peaks in the chromatogram.

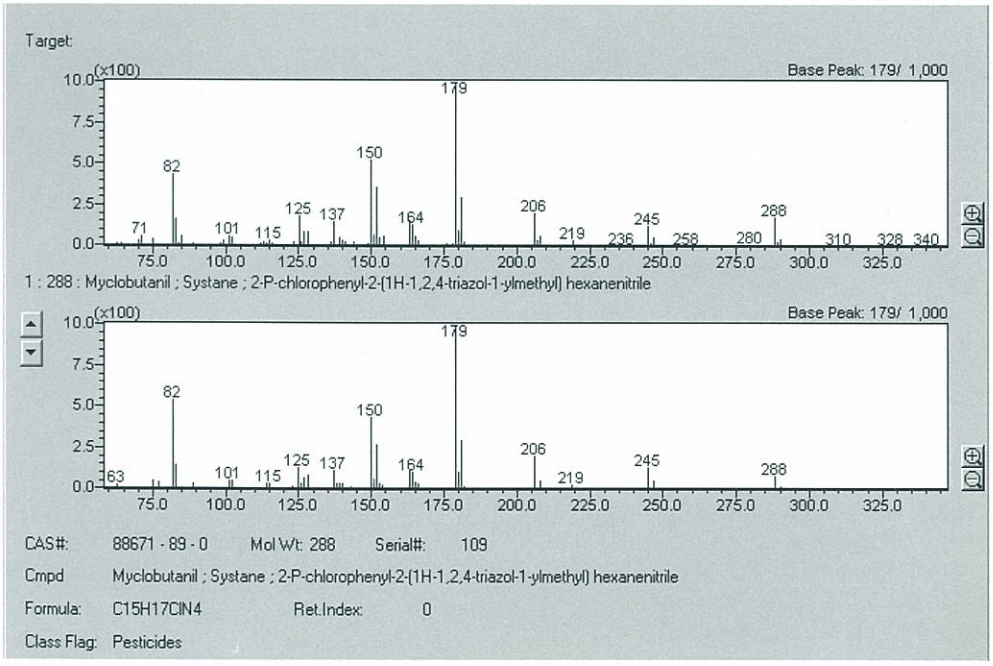

Figure 5. A comparison between the mass spectra Myclobutanil reference mass spectrum in the Shimadzu Pesticide Library and the mass spectrum of the identified peak.

■ Instrument Parameters

Instrument:	GCMS-QP2010
Column:	Rtx-5SilMS, 30m, 0.25mm, 0.25μm
Injection vol.:	3μL
Injector temp.:	250C
Injection:	Splitless with High Pressure Injection (270kPa, 2min)
Carrier gas:	Helium
Flow control:	Constant Pressure
Inlet pressure:	76.1 kPa
Column temp.:	70°C(2min)-25°C/min-150°C-3°C/min-200°C-8°C/min-280°C(10min)
Ionization:	Electron Impact (EI)
Acquisition mode:	SCAN
Mass range:	60-350 amu
Scan interval:	0.5 sec

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