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Application News

Liquid Chromatograph Mass Spectrometer LCMS-9050/9030
Kit for Direct Probe Ionization Mass Spectrometer DPiMS™ QT

Analysis of Fungicide on the Peel Surface of Imported Oranges (2) —Identifying Substances by MS/MS Analysis—

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User Benefits

- Whether or not a target substance is present on the surface of the sample can be judged with only simple pretreatment.
- The combination of DPiMS QT and LCMS -9050 can identify substances in a sample with a simple pre-treatment, taking advantage of the high MS accuracy of the Q-TOF mass spectrometer and MS/MS analysis.

■ Introduction

Due to the long transport time of agricultural products imported to Japan from overseas, post-harvest pesticides are applied after agricultural products are harvested to prevent mold or decay during transport. However, Japan prohibits the importation, use, or sale of foods with unapproved post-harvest agricultural chemicals. Therefore, inspection technologies that require only simple pretreatment and operating steps to determine whether or not post-harvest pesticides were used are expected to achieve shorter inspection times and result in shorter overall transport times.

Application News 01-00411 "Analysis of Fungicide on the Peel Surface of Imported Oranges (1)" describes using simple operations with LCMS-9050 and DPiMS QT systems to detect the fungicide enilconazole on the peel surface of imported oranges. This article shows an example of analyzing the same pretreated samples by MS/MS tandem mass spectrometry to identify substances based on a comparison of fragment ions in addition to accurate mass values obtained by MS analysis.

■ Pretreatment and Measurement Conditions

The experiment determines whether enilconazole applied to imported oranges (Fig. 1) can be detected using an LCMS-9050 liquid chromatograph mass spectrometer in combination with a DPiMS QT (Fig. 2).

The orange peel was pretreated by the same steps as described in Application News 01-00411 "Analysis of Fungicide on the Peel Surface of Imported Oranges (1)."

Probe actuation parameter settings and the method settings for MS and MS/MS analysis are indicated in Tables 1 and 2. Only the positive mode was used for this analysis, however LCMS-9050 systems are also capable of high-speed switching between positive and negative modes to flexibly analyze multiple types of substances.

Fig. 1 LCMS-9050 and DPiMS™ QT

■ MS Analysis

Fig. 3 shows the results from MS analysis of a standard sample and orange peel surface extraction. Enilconazole was detected from both samples, with accurate mass within ±1 ppm of theoretical values ($[M+H]^+$: *m/z* 297.0555). In addition, the results also showed characteristic isotope distributions. Those results alone provide highly accurate information indicating that the substance is enilconazole.

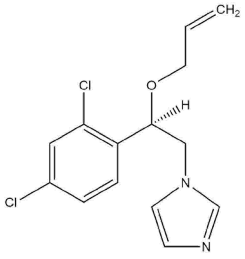

Fig. 2 Enilconazole Structural Formula

Table 1 Probe Actuation Parameters

Ionization Position:	–37 mm
Ionization Stop Position:	160 msec
Sample Acquisition Position:	–46.0 mm
Sample Acquisition Stop Time:	30 msec
Probe Speed:	300 mm/s
Probe Acceleration:	0.86 G

Table 2 Analytical Conditions

DL Temp.:	250 °C
Heat Block Temp.:	50 °C
Interface Voltage:	+2.50 kV (ESI – positive mode)
Scan Range:	*m/z* 50 to 1000 (MS) *m/z* 50 to 350 (MS/ MS)
Collision Energy (CE):	25.0 (MS/MS)
Measurement Time:	0.5 min

■ MS/MS Analysis

In this experiment, fragment ions were also compared by MS/MS analysis. Fig 4 shows the results from MS/MS analysis for each sample that selected a precursor ion of *m/z* 297.0555 (theoretical mass of protonated form of enilconazole). The enilconazole standard sample shows the characteristic fragment ion pattern as Fig. 4-A ▼. In the sample of the substances extracted from the orange peel surface, the result also shows characteristic fragment ion pattern that matches the standard sample results (Fig. 4-B ▼).

From these results, the substance detected at *m/z* 297.0555 from the orange peel surface can be identified as the enilconazole.

■ Conclusion

This application showed a combination of LCMS-9050 and DPiMS QT systems can be used to quickly and easily confirm the presence/absence or identify target substances extracted from sample surfaces. The technique described in this article can also be used to analyze all sorts of other samples that can fit inside the folded plate, which can be expected to be useful in a wide range of fields.

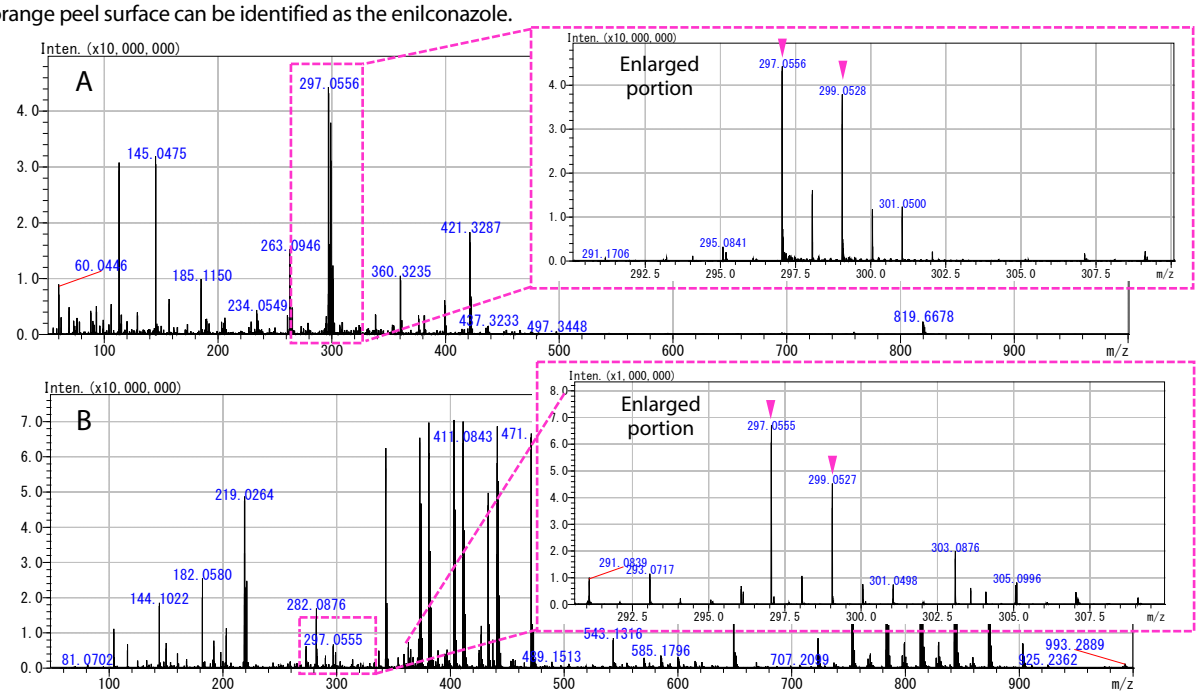

Fig. 3 MS Spectra
A: MS Spectrum of 0.5 ppm Enilconazole Standard Sample; B: MS Spectrum of Substance Extracted from Orange Peel Surface

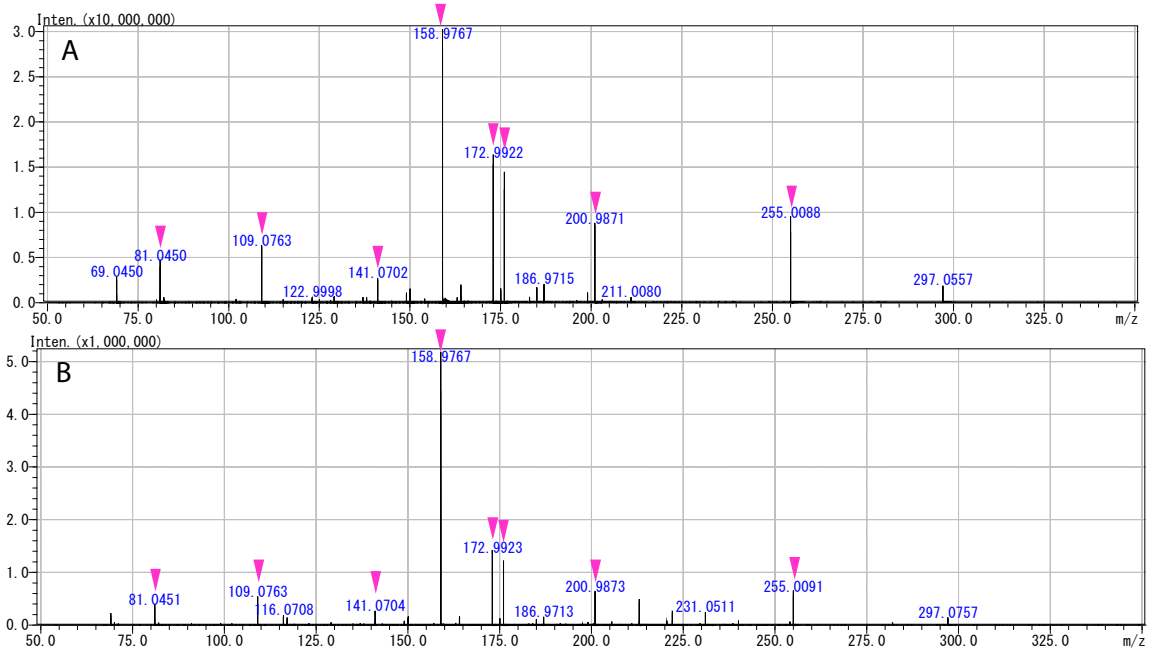

Fig. 4 MS/MS Spectra of the Precursor Ion *m/z* 297.0555 (Theoretical Mass of Enilconazole Protonated Form)
A: MS/MS Spectrum of 0.5 ppm Enilconazole Standard Sample; B: MS/MS Spectrum of Substance Extracted from Orange Peel Surface
▼: Fragment Ions from Enilconazole

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