

## Confirmation of Synthesis of Organic EL Materials Using Direct Infusion Mass Spectrometry

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### User Benefits

- ◆ By injecting a sample using direct infusion into a single quadrupole mass spectrometer, confirmation of the synthesis of the main components can be simply achieved.
- ◆ In-source CID enables simple estimation of chemical structure as well as confirmation of molecular weight.

### Introduction

In chemical synthesis, confirmation of molecular weight by mass spectrometry is one of the common methods used for determining whether the synthesis was successful or not. When the composition of the analyte can be inferred in advance, such as for confirming synthesis, accurate mass measurement is not always necessary. In Application News No. 01-00266-EN, examples were introduced in which the synthesis of organic EL materials was confirmed and the molecular weight of the impurities contained was estimated using a high performance liquid chromatograph (HPLC) and a single quadrupole mass spectrometer.

This Application News describes an example in which the molecular weight was confirmed by direct infusion and single quadrupole mass spectrometer in order to more easily confirm the synthesis of the main component.

### Samples

The commercial organic EL material TAPC (Fig. 1) was selected as an analyte. A standard solution of 1 ppm TAPC was prepared with tetrahydrofuran and analyzed.

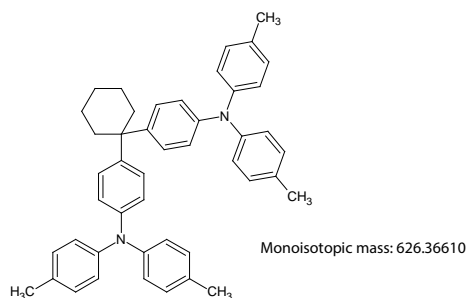


Fig. 1 TAPC (4,4'-Cyclohexylidenebis[N,N-bis(4-methylphenyl)benzenamine])

### Instruments and Analytical Conditions

The LCMS-2050 single quadrupole mass spectrometer (Fig. 2) was used in the analysis. It is compact, but it offers excellent ease of use and performance. The samples were injected into the mass spectrometer by direct infusion (continuous injection of the sample by syringe pump). Although the direct infusion is simple and the mass information of a compound can be obtained in a short time, it can lead to contamination of the instrument. However, the LCMS-2050 is highly robust, and even if it is contaminated, it is easily maintained, so it is suitable for direct infusion analysis. The analytical conditions are shown in Table 1.



Fig. 2 LCMS-2050 Single Quadrupole Mass Spectrometer

Table 1 Analytical Conditions

[Direct Infusion Condition]	
Flowrate:	0.1 mL/min
[MS Conditions] (LCMS-2050)	
Ionization:	ESI/APCI (DUIS™), Positive mode
Mode:	Scan ( $m/z$ 10-1000)
Interface Voltage:	+3.0 kV
Nebulizing Gas Flow:	2.0 L/min
Drying Gas Flow:	5.0 L/min
Heating Gas Flow:	7.0 L/min
Desolvation Temp.:	450 °C
DL Temp.:	200 °C
Qarray Voltage:	20/150 V

### Analysis by Direct Infusion

The standard solution of 1 ppm TAPC was analyzed by scan mode under the analysis conditions in Table 1. Fig. 3 shows the mass spectrum of the main peaks detected. The characteristic ions originating from TAPC were detected. It is considered that  $m/z$  626 is the molecular ion  $[M]^+$ ;  $m/z$  627 is the protonated molecule  $[M+H]^+$ , and  $m/z$  628 and 629 are isotopic ions.

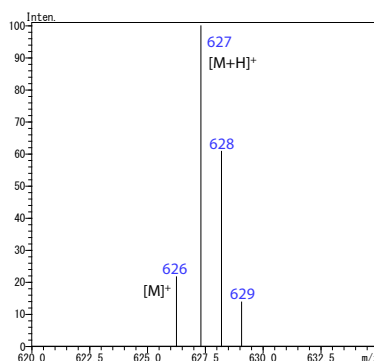


Fig. 3 Mass Spectrum of TAPC

In the LCMS-2050, it is possible to deliberately dissociate molecules in the ion source (in-source CID) at high Qarray voltages to obtain chemical structure as well as molecular weight. The standard solution of 1 ppm TAPC was analyzed by scan mode at 150 V of Qarray voltage. Fig. 4 shows the mass spectrum obtained using in-source CID. Several fragment peaks dissociated from TAPC were detected. In this way, simple structural analysis can be performed using in-source CID.

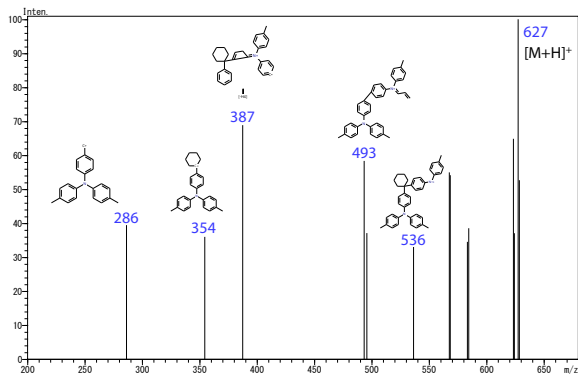


Fig. 4 Mass Spectrum Using In-Source CID

### Accurate Mass Measurement Using Q-TOF

As described above, molecular weights can be simply confirmed using a single quadrupole mass spectrometer. If more accurate qualitative analysis is required, a quadrupole time-of-flight (Q-TOF) mass spectrometer is useful. The standard solution of 1 ppm TAPC was analyzed by MS mode and MS/MS mode. The sample was injected by direct infusion into the Q-TOF mass spectrometer LCMS-9030. The data obtained were analyzed using LabSolutions Insight Explore™.

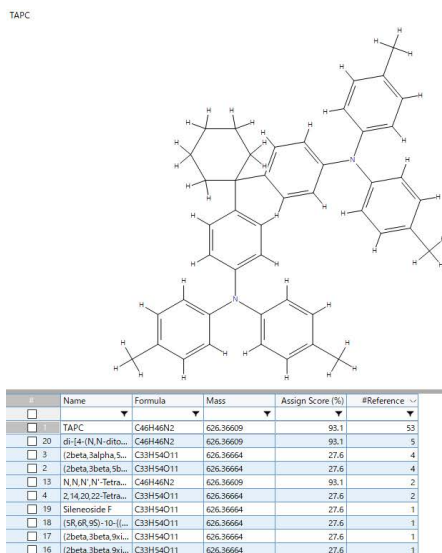


Fig. 6 Online Search (ChemSpider) Results

<Related Application News>

- Analysis of Organic EL Materials and Impurities Using Single Quadrupole Mass Spectrometer [Application News No.01-00266-EN](#)

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Fig. 5 shows the mass spectrum of the MS analysis. The composition was estimated to be C<sub>46</sub>H<sub>46</sub>N<sub>2</sub> within a mass accuracy of 1 mDa. Next, an online search was performed on the ChemSpider database using the data of MS/MS spectrum. As shown in Fig. 6, the mass accuracy of the fragment peaks was within 1 mDa, so TAPC was estimated with high accuracy.

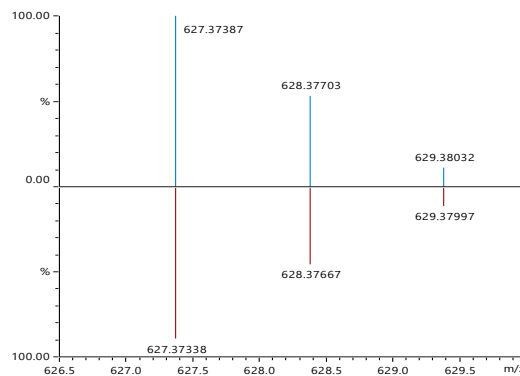


Fig. 5 Mass Spectrum by Q-TOF  
(Top: Measured mass spectrum, Bottom: Theoretical mass spectrum)

### Conclusion

This Application News describes an example in which molecular weight was confirmed by direct infusion and the single quadrupole LC-MS. To confirm the molecular weight of trace amounts of impurities, a combination of chromatographic separation using HPLC and mass spectrometer is useful. However, when the analyte is the main component that has high purity or high concentrations, the direct infusion is sufficient to obtain the molecular weight. If more accurate qualitative analysis is required, a Q-TOF mass spectrometer is useful.

