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Application News

Streamlining Impurity Management Using LabSolutions™ Detect

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User Benefits

- LabSolutions Detect automates impurity peak detection and identification, improving the efficiency of impurity management.
- By coupling a single quadrupole mass spectrometer (LCMS-2050), mass information can be acquired as qualitative information for impurity peaks.

■ Introduction

In the pharmaceutical development process, the qualitative and quantitative analysis and appropriate management of impurities are essential to ensure product safety. For example, during active pharmaceutical ingredient (API) synthesis, various impurities are generated throughout multi-step synthetic routes from starting materials to the final API. Accordingly, the ICH guidelines[1)] define threshold levels at which impurity identification is required, and such impurities must be controlled within acceptable limits. In addition, stability testing of pharmaceuticals is conducted to evaluate time-dependent changes under various exposure conditions in order to ensure product safety and determine shelf life. Degradation products generated under these exposure conditions must also be appropriately managed to monitor such changes over time. However, when multiple impurities and degradation products are managed manually, tasks such as peak detection and peak identification carry the risk of missed detections and human error. LabSolutions Detect, an anomaly peak detection support software, automates peak detection and peak identification for arbitrary peaks, including impurities and degradation products. In this article, case examples are presented in which peak detection and peak tracking were automated for multiple model samples assuming different impurity profiles, thereby streamlining impurity management. Furthermore, the combined use of a single quadrupole mass spectrometer (LCMS-2050) demonstrates that mass information can be acquired as qualitative information for each peak.

■ Target Compounds

As target compounds, a control sample free of impurities and five model samples assuming different impurity profiles were used; the model samples were artificially prepared as mixtures of different low-molecular-weight pharmaceuticals. The analytical conditions are summarized in Table 1. LabSolutions Detect automatically performs peak detection according to predefined criteria. In this study, the upper limit of the area percentage for impurity peaks was set to 0.1%, and peak detection and peak tracking were automatically performed for Samples 1–5 (Table 1).

Table 1 Analytical Conditions

Control	: Benzoic acid, Naproxen, Probenecid
Sample 1	: Control with added quinidine
Sample 2	: Control with added antipyrine, hydrocortisone
Sample 3	: Control with added antipyrine, furosemide
Sample 4	: Control with added antipyrine, indometacin
Sample 5	: Control with added indometacin
Mobile phase	
Pump A	: 0.1% formic acid in water
Pump B	: Acetonitrile
Column	: Shim-pack Scepter C18-120 *1 (100 mm × 3.0 mm I.D., 1.9 µm)
Injection Vol.	: 0.5 µL

LC Conditions

System	: Nexera X3
B Conc.	: 5% (0 min)→90% (5 min)→5% (5-10 min)
Column Temp.	: 40 °C
Flow rate	: 0.7 mL/min
Detection	: 254 nm (SPD-M40, STD cell)

MS Conditions

System	: LCMS-2050
Ionization	: ESI/APCI (DUIS), positive and negative mode
Mode	: SCAN (*m/z* 80-1000)
Nebulizing gas flow	: 2.0 L/min
Drying gas flow	: 5.0 L/min
Heating gas flow	: 7.0 L/min
DL Temp.	: 200 °C
Desolvation Temp.	: 450 °C
Interface voltage	: +3.0 kV / -2.0 kV

Criteria of peak detection : Upper limit of impurity (area %) : 0.1

*1 P/N : 227-31013-03

■ Streamlining Impurity Management by LabSolutions Detect

Fig. 2 shows the results of automatic impurity detection and identification performed by LabSolutions Detect in accordance with the criteria set as detection thresholds (Fig. 1). The blue-framed area in Fig. 2 shows the results for the control, in which three known compounds (Known 1–3) were detected. The red-framed area in Fig. 2 shows the results for Samples 1–5, where the area percentages of the detected impurities are automatically entered as red values. In addition, because peak identification is also automated, identical impurities are displayed in the same column. For example, in Sample 2, two impurities (New 2 and New 3) were detected, and New 2 was also detected in Samples 3 and 4. Furthermore, for the three known compounds (Known 1–3), the area percentages were automatically entered for Samples 1–5, indicating that these compounds were detected in all samples. Fig. 3 shows the individual chromatograms for Samples 1–5, in which automatically detected impurity peaks are highlighted in red, facilitating the identification of unknown impurities. In addition, a comparison of the chromatograms for Samples 1–5 is shown in Fig. 4. By combining these results with those shown in Fig. 2, the number of impurities detected across multiple chromatograms and their elution times can be visually confirmed. Moreover, mass information obtained using the LCMS-2050 can be utilized as qualitative information for each impurity.

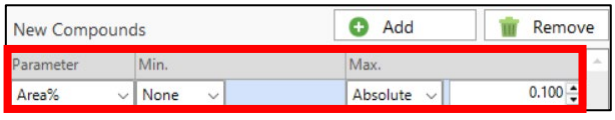

Fig. 1 Criteria for Unknown Impurities

	#	Data File Name	Overall Result	Known 1 Area%	Known 2 Area%	Known 3 Area%	New 1 Area%	New 2 Area%	New 3 Area%	New 4 Area%	New 5 Area%
Control	1	Control.lcd		14.003	18.525	67.472					
Target											
Sample 1	1	Sample 1.lcd	Outside the criteria	13.211	17.450	64.201	5.138				
Sample 2	2	Sample 2.lcd	Outside the criteria	12.712	17.162	62.129		4.339	3.658		
Sample 3	3	Sample 3.lcd	Outside the criteria	12.736	17.019	61.301		4.575		4.369	
Sample 4	4	Sample 4.lcd	Outside the criteria	12.651	16.862	61.024		4.608			4.855
Sample 5	5	Sample 5.lcd	Outside the criteria	13.031	17.433	64.114					5.422

Fig. 2 Impurity Management by LabSolutions Detect (area percentages of detected impurities are automatically entered as red values)

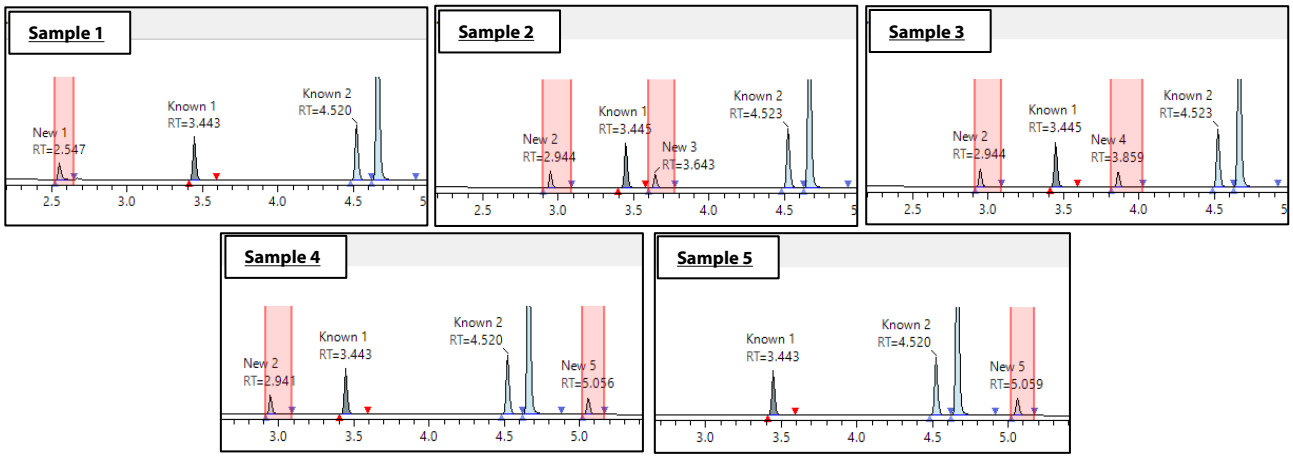

Fig. 3 Chromatograms of Samples 1–5 (automatically detected impurities are highlighted in red)

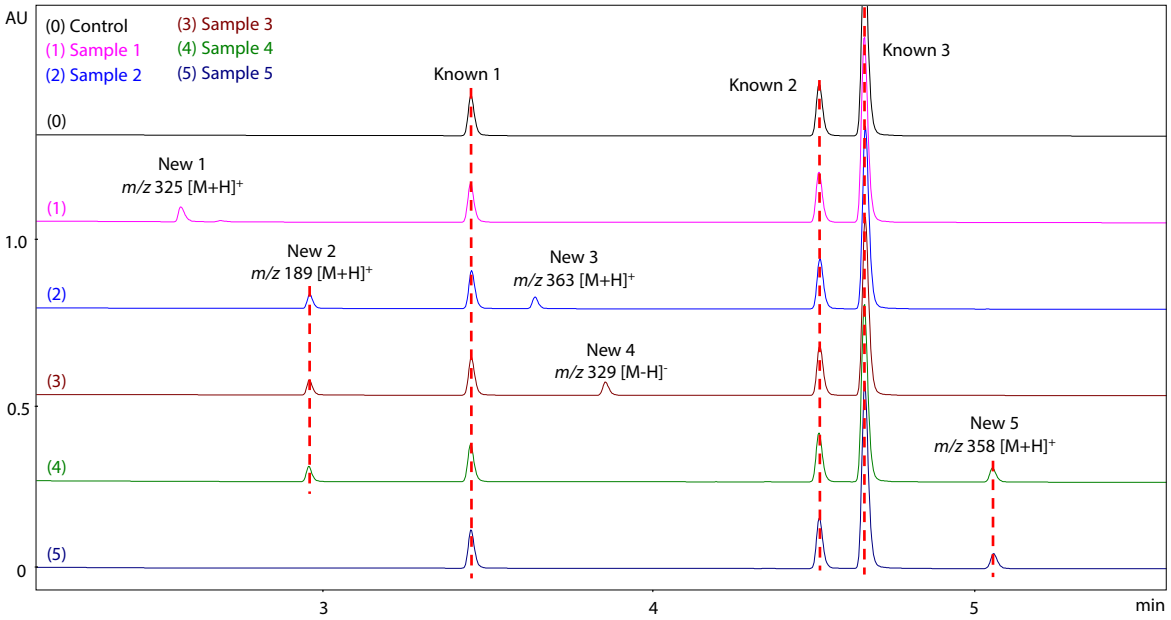

Fig. 4 Comparison of Chromatograms for Control and Samples 1–5

■ Conclusion

By automating impurity peak detection and identification using LabSolutions Detect, the risk of missed peaks and human identification errors can be reduced. In addition, coupling a single quadrupole mass spectrometer (LCMS-2050) enables acquisition of mass information as qualitative data for each peak.

Consequently, this approach contributes to improved efficiency and reliability in impurity management.

<References>

1) International Council for Harmonisation of Technical Requirements for Pharmaceuticals for Human Use : Q3A, Q3B

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