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Application News

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Gas Chromatography

Packed Column Analysis of Mentha Oil Using Nexis™ GC-2030 (FID)

The Nexis GC-2030 gas chromatograph has recently begun to support packed columns. The detectors compatible with packed columns are a flame ionization detector (FID) and a thermal conductivity detector (TCD).

As an example of glass-packed column FID analysis using the Nexis GC-2030, this article introduces a purity test of mentha oil performed in accordance with the Japanese Pharmacopoeia.

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Packed Column Support System

A glass column which can be installed into the Nexis GC-2030, a system compatible with packed columns, is the same as that used for the GC-17 or GC-2010, and it can be shared between these instruments.

Using the FID-2030Packed kit (P/N: S221-85191-41), the FID-2030 can be easily modified to become a packed FID by:

- Replacing the standard nozzle for the FID with one for a packed FID;
- Removing the FID's capillary column adaptor.

Fig. 1 shows an example of glass column installation.

Fig. 1 Example of Glass Column Installation

Extendable to Dual Line

A packed column analysis of samples with a column temperature program may cause greater baseline drift due to the elution of the liquid phase from the column, which can affect identification or quantitative processing.

The FID-2030 (compatible with packed columns) can be extended to a dual line system comprising the reference line, which has a sample vaporization chamber, column and detector, for elimination of baseline drift, as well as the sample line for analyzing samples.

Example of analysis - Analysis of Mentha Oil

In this example, mentha oil was analyzed to quantify l–menthol in accordance with the purity test prescribed by the Japanese Pharmacopoeia. To the sample diluted with ethanol, the internal standard substance (ethyl n-caprylate) was added to make 1 µL of sample solution, which was used for analysis.

Analysis Conditions

Table 1 lists the configuration of the instrument used for analysis and the analysis conditions. As prescribed by JP, the column flow rate was adjusted so that the retention time of the internal standard substance (ethyl n-caprylate) is around 10 minutes.

Table 1 Instrument Configuration and Analysis Conditions

Model	: Nexis GC-2030 /AOC-20i +SINJ-2030+FID-2030Packed Kit
Injection Mode	: Direct
Injection Volume	: 1.0 µL
Injection Temp.	: 200 °C
Carrier Gas	: N_2
Carrier Gas Control	: Constant flow rate[*1]
Column	: PEG 6000 25% CW 60/80 AW-DMCS (2 m × 3 mm I.D.)
Column Temp.	: 150 °C
Detector	: Flame ionization detector (FID)
Detector Temp.	: 200 °C
Detector Gas	: H_2 32.0 mL/min, Air 200 mL/min (no makeup gas required)

*1 The carrier gas flow should be adjusted so that the retention time of the internal standard substance is 10 minutes. The carrier gas flow setting may vary depending on the degree of column packing.

Chromatogram and Quantitative Results

The chromatogram of the sample solutions are shown in Fig. 2.

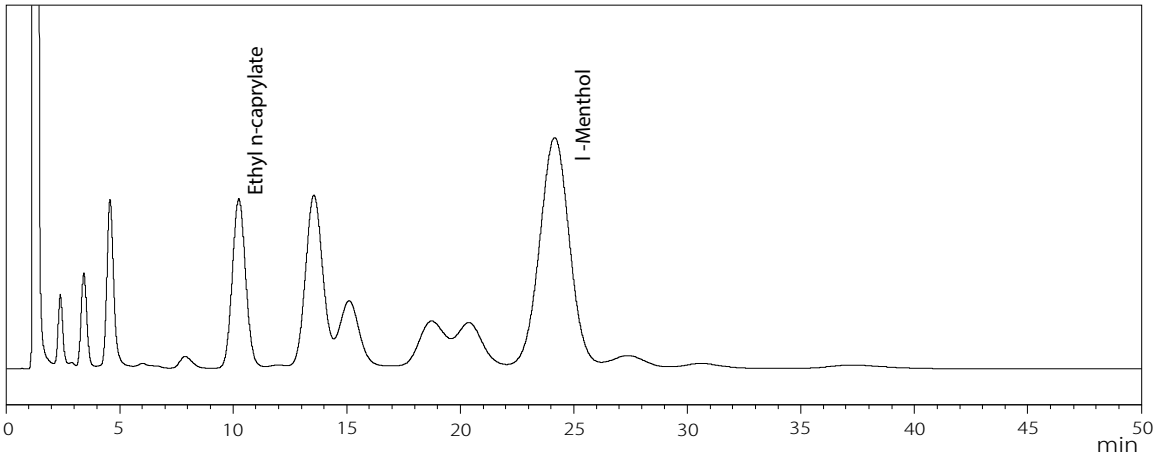

Fig. 2 Chromatogram of Sample Solutions

Quantitative Results for l-Menthol

The analysis results for the sample solutions and the quantitative results for l–menthol are shown in Table 2.

Table 2 Quantitative Results for l-Menthol

Sample solution	IS area	Menthol area	Quantitative value (mg)
n=1	185,015,464	542,461,986	166.961
n=2	188,204,772	552,155,381	167.065
n=3	188,572,117	553,166,607	167.045
n=4	187,792,875	550,934,516	167.061
n=5	188,802,576	553,789,578	167.029
Average value	187,677,561	550,501,614	167.032
%RSD	0.819	0.840	0.025

Conclusion

With a glass packed column connected to the FID modified for packed columns in the Nexis GC-2030, mentha oil was analyzed using a single line FID.
Good repeatability of measurements of the sample solution was demonstrated, and favorable results with reduced variations in the quantitative value were obtained.

Optional Information on Nexis GC-2030

The Nexis GC-2030 system makes a wide variety of useful options available.
<IQ/OQ documentation> IQ/OQ validation documentation for the Nexis GC-2030 supports latest format(E-format.)
IQ/OQ validation documentation (E-format) service controls procedures for OQ inspection parameters, setting values, control criteria and OQ results, all of which are integrated in a summary table, and it also provides an equipment qualification plan (EQP), a document for planning and checking IQ/OQ inspection items. In addition to the IQ inspection report and OQ inspection report, our service has allowed customers to receive the IQ/OQ inspection summary report (EQR). The use of this summary report helps to ensure that customers can verify the inspection procedures and results and can smoothly respond to regulatory agencies when they require a corporate audit to be performed. The E-format validation report can be provided as PDF files.

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