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DPiMS-2020 Direct Probe Ionization Mass Spectrometer

Contaminant Analysis by DPiMS™-2020 (3): Detection of Sleeping Pills in Beverages

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User Benefits

- The PESI method enables quick and simple analysis without complex sample preparation.
- A simple technique for judgment of the presence of sleeping pills in beverages was developed.
- Simple and high sensitivity screening for the presence of contamination by foreign substances is possible.

■ Introduction

A diverse range of incidents involving contamination or adulteration of beverages and foods has been reported, including contamination by familiar chemical substances, and for this reason, an extremely large number of chemical substances must be considered as analysis targets in inspections of beverages and foods in which contamination is suspected. To conduct this kind of analysis efficiently, it is necessary to develop a quick screening technique for the presence of contaminants.

In this article, a technique that enables highly sensitive detection with minimal sample preparation was developed using a DPiMS-2020 analytical instrument, which employs the PESI-MS method, assuming contamination of beverages by sleeping pills as one example of contamination. The results of a study of the possibility of applying this technique as a simple contamination screening technique for drugs and poisons are also reported.

■ Analysis of Sleeping Pills Dissolved in Distilled Water

The targets of the analysis were four preparations A to D, which are sleeping pills in tablet form. In addition to the soporific component (i.e., the active ingredient of the sleeping pill), tablets also contain additives called excipients, which are used as fillers or diluents. Therefore, the analysis also focused on those components.

First, samples for study were prepared by dissolving and diluting the four sleeping pills in distilled water. As the sample preparation procedure of the analysis, an equal amount of 2-propanol was added and mixed with the sample substance. 9 µL of the sample solution was then placed on a dedicated sample plate, and the analysis was conducted using a DPiMS-2020 direct probe ionization mass spectrometer (Fig. 1). Tables 1 and 2 show the probe drive conditions and the mass spectrometer conditions, respectively.

Table 1 Probe Drive Conditions

Ionization position	-37 mm
Ionization stop time	200 msec
Sampling position	-46.3 mm
Sampling stop time	50 msec
Probe speed	250 mm/s
Probe acceleration	0.63 G

Table 2 Mass Spectrometry Analysis Conditions

DL temperature	250 ˚C
Heater block temperature	30 ˚C
Interface voltage	±2.45 kV
Scan speed	5000 µ/s
Data acquisition time	0-1 min: Negative ion mode 1-2 min: Positive ion mode

As a result of this analysis, the protonated molecules of the active ingredients contained in each of the tablets were detected with a detection intensity corresponding to the content in the tablet, as shown in Table 3. As also shown in the table, among the additives, ions originating from lactose were also detected with remarkable intensity as the sodium adduct (*m/z* 365), potassium adduct (*m/z* 381), and sodium adduct dimer ions (*m/z* 707). In the following experiment, these ions were used as monitor ions.

Fig. 1 Appearance of DPiMS™-2020

Table 3 List of Target Compounds

Sleeping pill	Target compound	Detection ion
Preparation A (0.25 mg)	A	*m/z* 343, 345 (pos.)
	Lactose	*m/z* 365, 381 (pos.)
Preparation B (1 mg)	B	*m/z* 343, 345 (pos.)
	Lactose	*m/z* 365, 381 (pos.)
Preparation C (2 mg)	C	*m/z* 314 (pos.)
	Lactose	*m/z* 365, 381 (pos.)
Preparation D (10 mg)	D	*m/z* 308 (pos.)
	Lactose	*m/z* 365, 381 (pos.)

■ Analysis of Spiked Samples

Next, samples spiked with sleeping pills were analyzed. These samples were prepared by spiking 200 mL of each four kinds of commercially-available beverages (α, β, γ, δ) with one tablet of one of the sleeping pills. An equivalent amount of 2-propanol was added to these samples and mixed, and 9 µL of the solution was placed on a dedicated sample plate for use in the analysis. As reference, data were also collected for unspiked (blank) beverages.

Using the spectra acquired from this analysis, we attempted to judge whether detection was possible or not, focusing on the active ingredient and the additive lactose (Table 3).

As one example of the analysis results obtained here, Fig. 2 shows the mass spectrum of beverage α spiked with preparation C. In comparison with the results of the blank beverage analyzed under the same conditions, ions originating from the spiked substance (positive ion mode: *m/z* 365, 381) were strongly detected.

A similar analysis of the other samples was also conducted. Table 4 summarizes the results of detection of the sleeping pills in each of the beverages. In beverages α and δ, only the active ingredient of D was detected, as this tablet has a high content of the active ingredient, and the active ingredients of the other tablets were not detected. On the other hand, when beverages β and γ were spiked with the sleeping pills, ions of 3 substances originating from the sleeping pills, which were not detected in beverage α, could be detected with comparatively good sensitivity.

When beverage α was spiked with preparations A, B, or C, ions originating from the active ingredients in the sleeping pills could not be detected, but judgment of the presence of contamination was possible by detection of lactose, which is used as an additive in the sleeping pills.

■ Conclusion

In this article, we attempted to use the PESI-MS method to determine whether beverages were contaminated with components originating from sleeping pills.

The results revealed that there are differences in the detectability of substances originating from sleeping pills, depending on the beverage. This study also showed that the results can be divided into cases in which ions originating from the active ingredient in the sleeping pill are easily detected, and cases in which components derived from additives (excipients) are easily detected, depending on the beverage.

<References>
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(2) Misato Wada et al. Application of probe electrospray ionization mass spectrometry to the analysis of poisons and drugs in adulterated foods and beverages, Jpn. J. Forens. Sci. Technol.

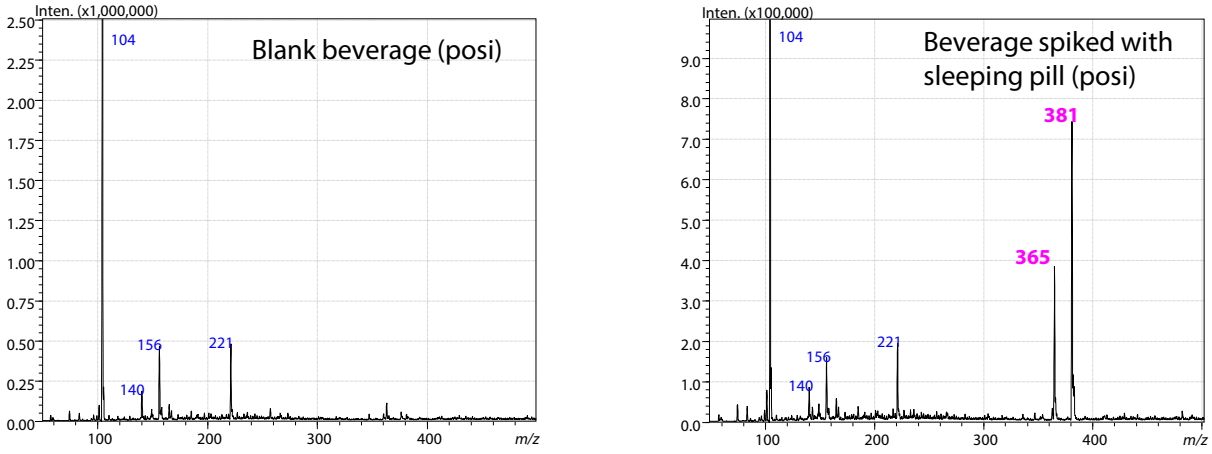

Fig. 2 Mass Spectra of Beverage Spiked with Sleeping Pill and Blank (Unspiked) Solution

Table 4 Detectability of Sleeping Pills and Excipient (Lactose) in Beverages

Beverage	Sleeping pill							
	Preparation A		Preparation B		Preparation C		Preparation D	
	Component A	Lactose	Component B	Lactose	Component C	Lactose	Component D	Lactose
Distilled water	✓	✓	✓	✓	✓	✓	✓	✓
α	×	✓	×	✓	×	✓	✓	✓
β	✓	–	✓	–	✓	–	✓	–
γ	✓	–	✓	–	✓	–	✓	–
δ	×	–	×	–	×	–	✓	–

✓ : detected, × : not detectable, – : could not be determined

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