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Application Data Sheet

No.142

GC-MS
Gas Chromatograph Mass Spectrometer

Analysis of Nootropics Using GC-MS(/MS) - Part 2

By performing mass separation in two stages, GC-MS/MS analysis is able to separate and detect contaminants in medicinal toxicants and biological samples with high sensitivity. Consequently, it can simplify the process of determining whether or not biological samples contain medicinal toxicants and can significantly shorten the time required for data analysis. However, analysis in the MRM mode requires optimizing MRM transitions and collision energies (CEs). Due to the time and trouble required for that optimization process, users have been asking for a pre-optimized database.
Smart Forensic Database Ver. 2 is registered with information necessary for MRM analysis. In this new version, the number of registered medicinal toxicants was increased from 201 components to 486 components. This comprehensive enhancement in medicinal toxicant information, such as for drugs of abuse, psychotropic drugs, general pharmaceuticals, and pesticides, significantly increases the number of medicinal toxicants that can be monitored with a single measurement.
This Application Data Sheet describes an example of using Smart Forensic Database Ver. 2 to analyze nootropics in blood plasma.

Sample Preparation

Blood plasma extract samples were prepared using a combination of QuEChERS and dispersive solid phase extraction (dSPE) methods. 0.2 mL of blood plasma was mixed with 1.3 mL of distilled water and added to a previously prepared 5 mL test tube containing 0.5 g of a Q-sep QuEChERS extraction salt packet (AOAC 2007.01, Restek Corporation, P/N: 26238), stainless steel beads, and 1.5 mL of acetonitrile. The test tube was then promptly mixed. Next, the mixture was centrifuged for 10 minutes at 3000 rpm and the acetonitrile phase fraction was obtained. Then the extract fraction was added to the Q-sep QuEChERS cleanup kit (Restek Corporation, P/N: 26242). After mixing in a vortex mixer for 2 minutes, the mixture was centrifuged for 5 minutes at 3000 rpm. The blood plasma extract sample was prepared by drying the re-fractioned extract solution under a stream of nitrogen gas and redissolving it in ethyl acetate. The standard nootropics sample was added to the resulting extract sample so that the concentration in the blood plasma was 50 ng/mL.

Analytical Conditions

The analytical conditions registered in Smart Forensic Database Ver. 2 were used (Table 1). The simultaneous scan/MRM analytical method was created with the 401 underivatized components registered in the database specified as target compounds for MRM measurement. To compare contaminant separation with a single-GC/MS system, a simultaneous scan/SIM analytical method was also created for the 17 nootropic components measurable without derivatization.

Table 1: Analysis Conditions

GC-MS:	GCMS-TQ8040
Column:	SH-Rxi™-5Sil MS (30 m long, 0.25 mm I.D., df = 0.25 μm) (Shimadzu, P/N: 221-75940-30)
Glass Liner:	Deactivated splitless liner with wool (PN:221-48876-03)

GC	
Injection Temp.:	260 °C
Column Oven Temp.:	60 °C (2 min) → (10 °C/min) → 320 °C (15 min)
Carrier Gas:	Helium
Flow Control:	Linear velocity (45.6 cm/sec)
Injection Mode:	Splitless
High Pressure Injection:	250 kPa (1.5 min)
Injection Volume:	1 μL

MS	
Interface Temp.:	280 °C
Ion Source Temp.:	200 °C
Acquisition Mode:	Scan/MRM Scan/SIM
Scan Event Time:	0.1 sec
Scan Mass Range:	*m/z* 43 to 600
Scan Speed:	10,000 u/sec
MRM (SIM) Event Time	0.4 sec
Total Loop Time:	0.5 sec

Analytical Results

Chromatograms obtained by SIM and MRM-mode measurements of the blood plasma extract sample spiked with nootropics are shown in Fig. 1. In SIM mode, it is extremely difficult to determine whether or not nootropics are contained, due to overlapping with ions derived from blood plasma contaminants, whereas all components were clearly detected in the MRM mode.

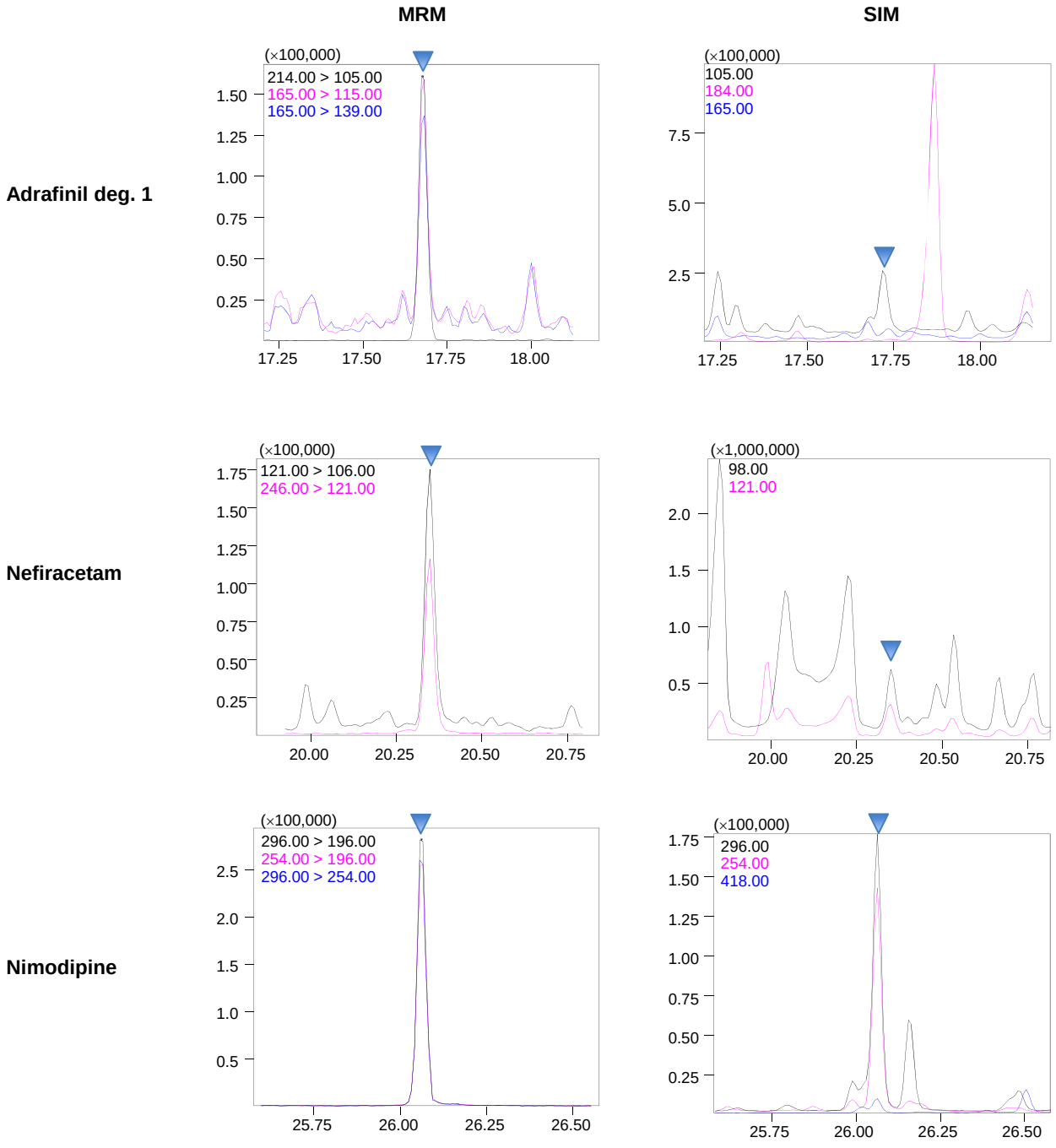

Fig. 1: MRM and SIM Mass Chromatograms of Nootropics in Blood Plasma Extract Samples (50 ng/mL Concentration in Blood Plasma)
(Left: MRM, Right: SIM)

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