SHIMADZU

Application News

Liquid Chromatography Mass Spectrometry No.C69

Ultra High Performance Liquid Chromatography / Mass Spectrometry Using Open Solution Software

With the demand for faster research and development and improved data quality, the spread of Ultra High Performance Liquid Chromatography (UHPLC) is rapidly advancing. The development of minute particle size packing material and high-pressure tolerance liquid chromatographs have made these higher speeds possible, but in order to further improve throughput, the importance of a high performance mass spectrometer coupled with well-developed, user-friendly software is also recognized. The LCMS-2020, with its high scanning speed (up to 15,000 u/sec) and high-speed polarity switching (15 msec polarity switching), demonstrates the performance that satisfies the demands of UHPLC. The powerful user interface of Open Solution makes data review and verification easy, supporting the chemist who wishes to make rapid yet accurate decisions related to his ongoing research.

■ Analysis and Data Processing Using Open Solution Software

Open Solution is a superb software for routine high-speed analysis. For example, when conducting confirmation of synthetic compounds in pharmaceutical product research and development, compounds having different molecular weights and various physical properties are required to be measured using the same analytical conditions in a short period of time. Under these circumstances, the shorter the time spent on analytical separations and data verification, the better, so software that provides simplified sample login and LCMS operation, and quickly indicates pertinent information becomes a real asset. Fig. 1 shows an Open Solution sample login screen, and Fig. 2 shows an example of a Results View page. The compounds shown in Table 1 are the test compounds that were analyzed for this application news article. The compounds are easily confirmed using the mass spectral data displayed in the Results View window by comparing theoretical and actual *m/z* values.

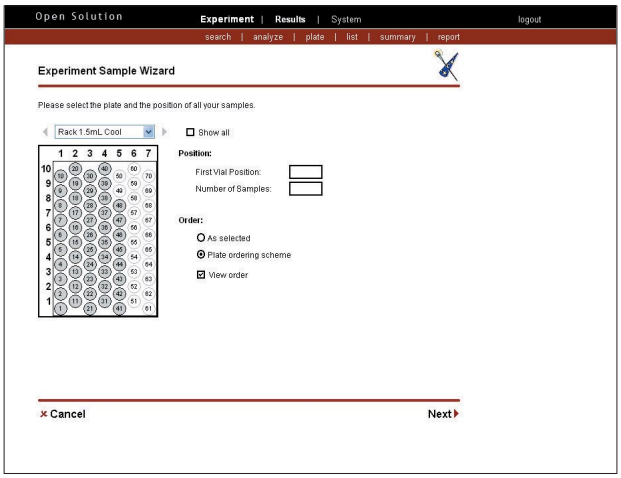

Fig. 1 Open Solution (Sample Log In Screen)

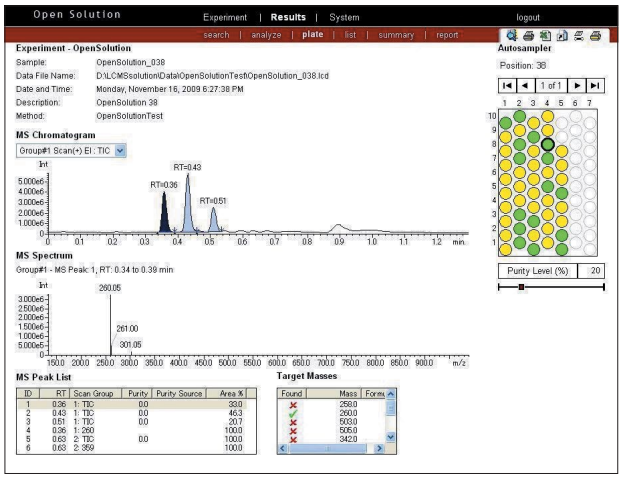

Fig. 2 Open Solution (Results View)

Table 1 Test Compounds (*: negative ion detection, others: positive ion detection)

No.	Compound	*m/z*	No.	Compound	*m/z*	No.	Compound	*m/z*
1	Atenolol	267	11	Doxepin	280	21	Isopropylantipyrine	231
2	Procaine	237	12	Dipyridamol	505	22	Alprazolam	309
3	Lidocaine	235	13	Desipramine	267	23	Triazolam	343
4	Atropine	290	14	Imipramine	281	24	Cilostazol	370
5	Yohimbine	355	15	Nortriptyline	264	25	Nifedipine	347
6	Chlorpheniramine	275	16	Amitriptyline	278	26	Diazepam	285
7	Propranolol	260	17	Dibucaine	344	27	Warfarin	309
8	Alprenolol	250	18	Verapamil	455	28	*Cefuroxime	423
9	Tetracaine	265	19	Reserpine	609	29	*Chloramphenicol	321
10	Diphenhydramine	256	20	Carbamazepine	237	30	*Nitrendipine	359

■ Ultra Fast Analysis of Drug Mixture Using LCMS-2020

We conducted UHPLC analysis of a solution containing a mixture of 30 drug compounds. A concentration of 5 µg/mL was used for substances analyzed using positive detection, and 50 µg/mL for substances analyzed using negative detection. Fig. 3 shows the mass chromatograms (displayed using Data Browser of LCMSsolution Ver. 5). For the liquid chromatograph, the Prominence UFLCXR was used. All of the constituents were eluted within 0.7 min using a Shim-pack XR-ODSII column (30 mm L × 1 mm I.D., 2.2 µm, P/N 228-59907-91).

Even after 700 successive analyses, stable measurement is achieved (Fig. 4).

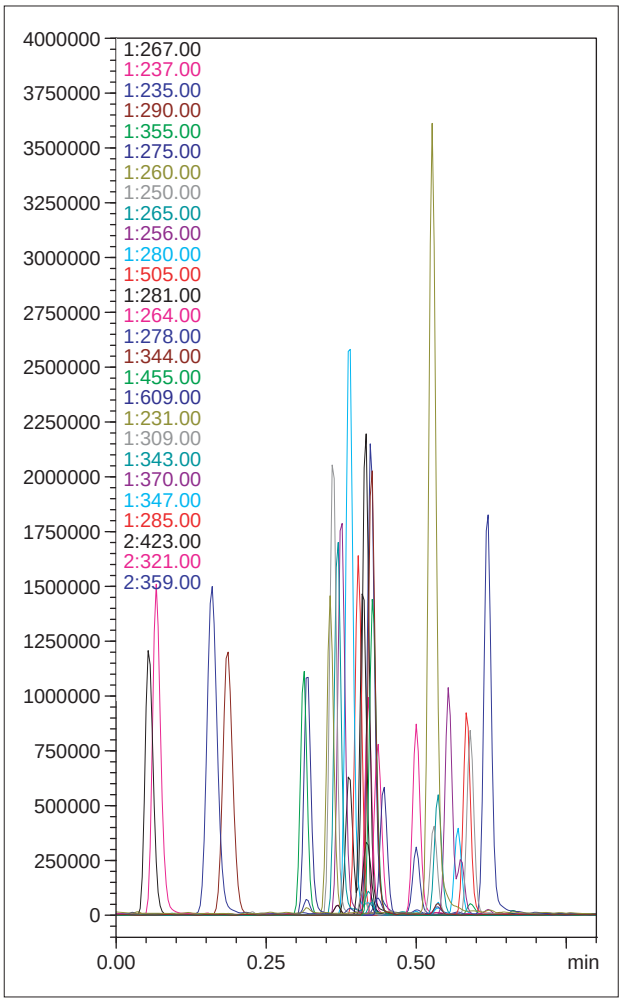

Fig. 3 Chromatograms of Drugs (Injection 1)

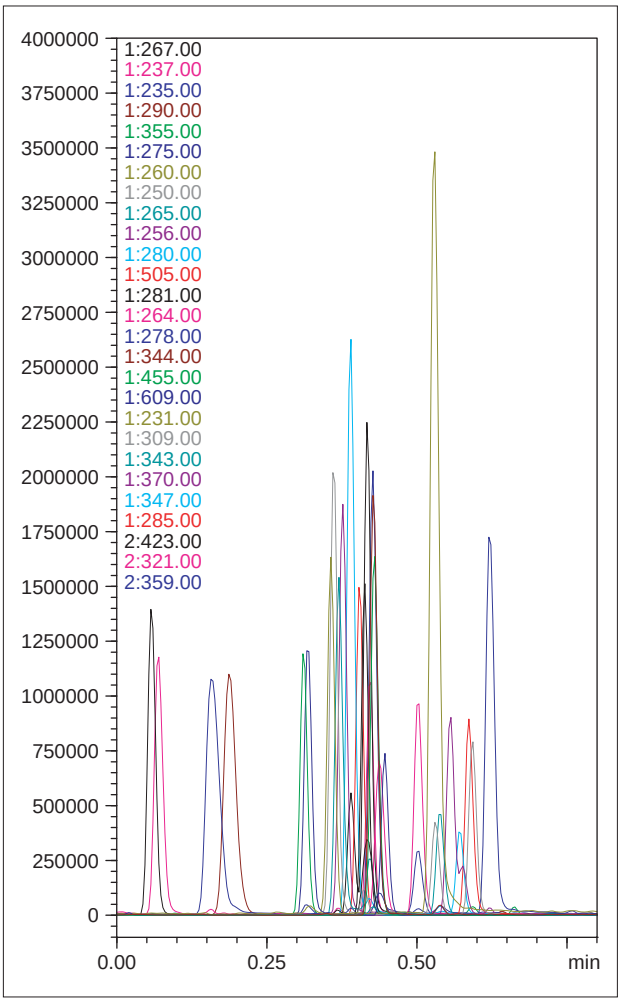

Fig. 4 Chromatograms of Drugs (Injection 700)

Table 2 Analytical Conditions

Column	: Shim-pack XR-ODS Ⅱ (30 mmL. × 1.5 mmI.D., 2.2 µm)	MS	: LCMS-2020
Mobile Phase	: A: 0.1 % formic acid in water B: 0.1 % formic acid in acetonitrile	Probe Voltage	: +4.5 kV (ESI-Positive mode), –3.5 kV (ESI-Negative mode)
Time Program	: 8 %B (0 min) - 95 %B (0.5 min) - 8 %B (0.51 min) - STOP(1.3 min)	Nebulizing Gas Flow	: 1.5 L/min
		Drying Gas Flow	: 20.0 L/min
Flowrate	: 1.2 mL/min	DL Temperature	: 250 ˚ C
Column Temperature	: 50 ˚ C	Block Heater Temperature	: 450 ˚ C
Injection Volume	: 1 µL	DL, Q-array Voltages	: default values
Rinsing Pump	: 3 sec (methanol)	Event Time	: 0.1 sec
Mixer Volume	: 100 µL	Scan Range	: *m/z* 150 – 1000

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