

Optimization of metabolome analysis
by using Fast GC/MS [Intuvo9000GC-5977MSD]
coupled with online solid-phase analytical derivatization technique
(1) Repeatability and Durability test of Amino acid and Organic acid



Introduction

Conventional metabolome analysis in GC/MS requires from 4 hours to overnight for drying and 2 hours for derivatization after sample extraction. Online **solid-phase derivatization (SPD)** dramatically reduces the pretreatment time and stabilizes the time finishing derivatization until the start of a GC/MS analysis, thus it is expected to more stable data acquisition.

In addition, an easy-to-use instrument is desirable as it is possible to perform high-speed analysis and measure multiple samples in line with the speeding up of pretreatment especially for beginners of GC/MS analysis. In this note, reproducibility and durability tests of amino acids and organic acids were conducted using a system that connects SPL-M100 as an online SPAD system and Intuvo 9000GC + 5977B MSD capable of high-speed analysis.

Instruments

Fast GC/MS and Online SPAD system



Intuvo 9000GC-5977B MS with SPL-M100

Benefit

[Intuvo9000GC]

- Fast analysis
- Easy handling
- Using Guard chip

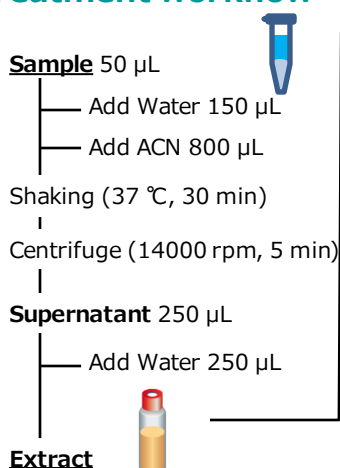
Guard chip

- Role as precolumn
- Easy changing
- Not required column cutting

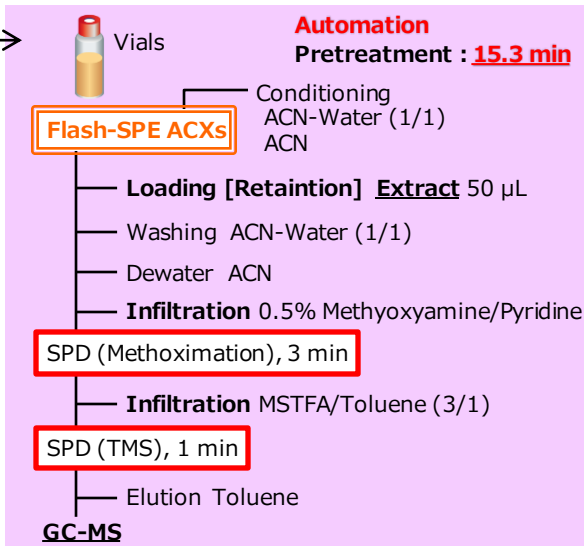
[SPL-M100]

- Extremely shorter pretreatment time
- Sequenced pretreatment and injection

Pretreatment workflow



Online SPE-GC : SPL-M100



Analytical condition

GC Intuvo9000C

Column	DB-5MS, 15 m \times 0.25 mm I.D. , df=0.25 μ m
Oven Temp.	80 $^{\circ}$ C (1 min)-20 $^{\circ}$ C/min-220 $^{\circ}$ C-30 $^{\circ}$ C/min-310 $^{\circ}$ C (3 min) (total 14 min)
MMI Temp.	220 $^{\circ}$ C (0.5 min)-70 $^{\circ}$ C/min-290 $^{\circ}$ C
Liner	Ultra inert liner, single tapered, low pressure drop, glass wool
Injection mode	Split (50:1)
Guard chip Temp.	Oven track
Carrier gas flow	1.0 mL/min (Constant flow)
Transfer line Temp.	290 $^{\circ}$ C

MS 5977B

Mode	Scan (m/z 70-470)
Ion source Temp.	250 $^{\circ}$ C
Quadrupole Temp.	150 $^{\circ}$ C

Sample



Information

第15回 メタボロームシンポジウム
「高速GC/MSとオンライン固相誘導体化を用いたメタボローム分析の最適化」

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AiSTI SCIENCE

Product

Online SPE-GC
SPL-M100
Solid phase cartridge
Flash-SPE
GC large volume injection port
LVI-S250



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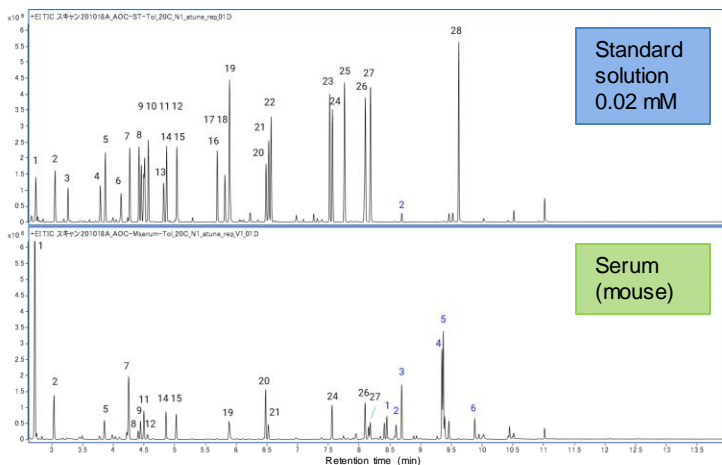
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Results and Discussion

(1) Repeatability test

Total ion chromatograms and the result of the repeatability test are shown by analysis of standard solution (amino acids and organic acids) and mouse serum.

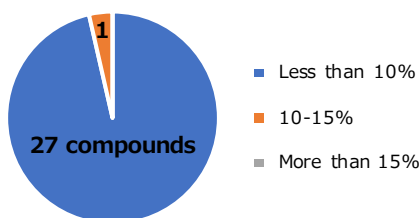


- | | |
|------------------------|--|
| 1. Lactic acid 2TMS | 18. Aspartic acid 3TMS |
| 2. Alanine 2TMS | 19. Pyroglutamic acid 2TMS |
| 3. Oxalic acid 2TMS | 20. Glutamic acid 3TMS |
| 4. Malonic acid 2TMS | 21. Phenylalanine 2TMS |
| 5. Valine 2TMS | 22. Tartaric acid 4TMS |
| 6. Benzoic acid TMS | 23. Shikimic acid 4TMS |
| 7. Leucine 2TMS | 24. Citric acid |
| 8. Isoleucine 2TMS | 25. Quinic acid |
| 9. Proline 2TMS | 26. Lysine 4TMS |
| 10. Maleic acid 2TMS | 27. Tyrosine 3TMS |
| 11. Glycine 3TMS | 28. Cysteine 3TMS |
| 12. Succinic acid 2TMS | 1. Gluconic acid 6TMS |
| 13. Fumaric acid 2TMS | 2. Palmitoleic acid TMS |
| 14. Serine 3TMS | 3. Palmitic acid TMS |
| 15. Threonine 3TMS | 4. 9,12-Octadecadienoic acid (Z,Z)-, TMS |
| 16. Malic acid 2TMS | 5. 9-Octadecenoic acid (E), TMS |
| 17. Methionine 2TMS | 6. Arachidonic acid TMS |

Total ion chromatograms of standard solution (amino acids and organic acids) and mouse serum

Repeatability (RSD) distributions of standard solution and mouse serum

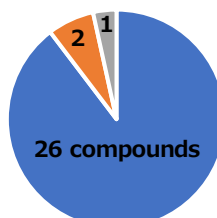
Standard solution (0.02mM)



The successful result was obtained in 16 sequential analyses (a mixture of 28 standards).

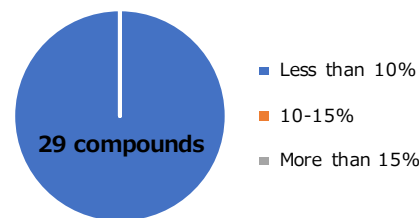
Serum (mouse)

First run (lot 1)



100 sequential analyses were performed twice, using different lots and on different dates. No change of liners or guard chips was performed during this period.

Second run (lot 2)



(2) Durability test

The change in detection response is shown below (the result of the first run). No particular trends were observed in these responses. A stable result was also obtained in the second run. These results show that this is a very robust system that does not require liner replacement in 200 sequential analyses of real samples.

Serum (mouse, lot 1)

