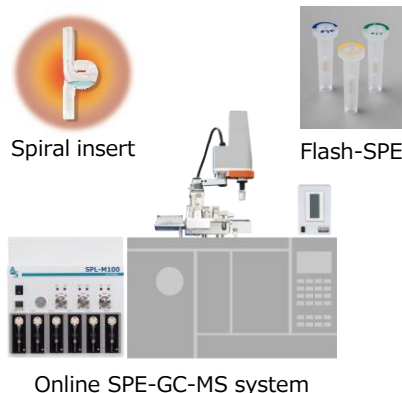


オンライン固相誘導体化-GC-MSシステムによる 尿中メタボローム分析

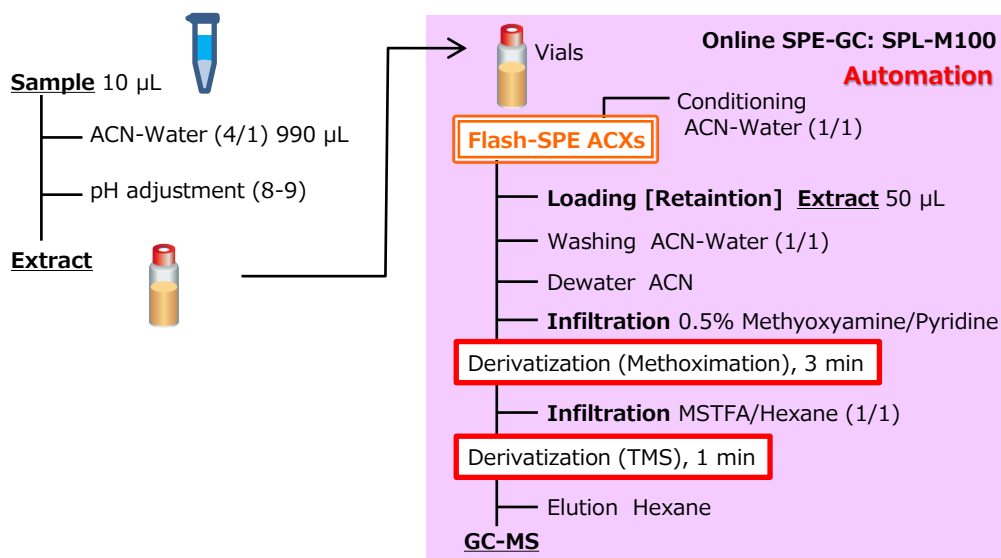
Introduction

固相誘導体化法（Solid-phase derivatization: SPD）は、分析対象成分を固相に保持し溶媒通液によって脱水処理を行うことで時間のかかる遠心乾固や凍結乾燥をすることなく誘導体化反応を行う手法です。

本アプリケーションでは尿中メタボローム分析における前処理方法および分析条件を紹介します。



SPD pretreatment workflow



Analytical condition

SPE-GC interface

SPE cartridge

PTV injection port

Insert type

Temp.

Gas chromatograph

Inlet mode

Flow mode

Pre-column

Column

Oven Temp.

Transfer line Temp.

Mass spectrometer

Acquisition mode

Data acquisition

SPL-M100 (AiSTI SCIENCE)

Flash-SPE

LVI-S250 (AiSTI SCIENCE)

Spiral insert

220°C(0.5 min)-50°C/min-290°C(23 min)

Split 1:50

Constant flow, 1.0 mL/min

0.25 mm i.d. x 0.5 m

Vf-5ms, 0.25 mm i.d. x 30 m, df=0.25 µm

100°C(2 min)-10°C/min-320°C(2min)

290°C

Scan (m/z 70-600)

3.0-26 min



SPL-M100
for SPE-GC system

Sample

尿

Information

AiSTI SCIENCE

Product

オンライン SPE-GC
SPL-M100

固相カートリッジ
Flash-SPE

大量注入口装置
LVI-S250



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HP : www.aisti.co.jp

Results

Phosphoric acid_3TMS

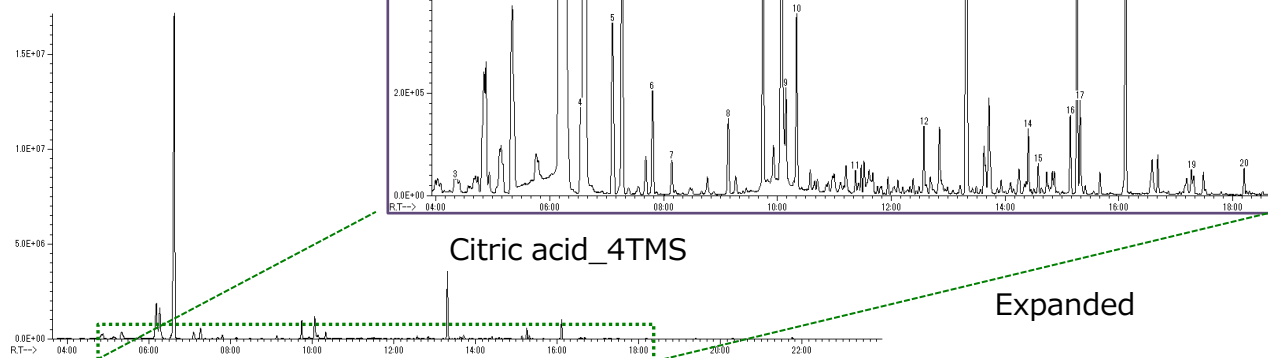


Figure: Total ion current chromatogram

Table 1: Result of recovery test (n=5)

Sample	No.	Norleucine_2TMS	Adipic acid_2TMS	Sample	No.	Norleucine_2TMS	Adipic acid_2TMS
Standard solution	S1	1,284,451	84,108	Urine	Feces_K1	1,400,444	99,683
IS concentrations:	S2	1,243,248	77,955	Dilution:	Feces_K2	1,415,794	98,689
20 μ M in vials	S3	1,169,708	73,754	100 times	Feces_K3	1,395,904	99,474
	S4	1,183,814	73,368	Spike period of IS:	Feces_K4	1,415,822	98,843
	S5	1,131,803	73,641	After deprotenization,	Feces_K5	1,391,498	96,746
	Ave.	1,202,605	76,565	20 μ M in vials	Ave.	1,403,892	98,687
	RSD, %	5.1	6.0		RSD, %	0.8	1.2
				(K/Sx100)	Recovery, %	117	129
				Mouse Feces	Feces_A1	1,451,203	106,679
				Dilution:	Feces_A2	1,521,654	103,472
				100 times	Feces_A3	1,477,161	99,614
				Spike period of IS:	Feces_A4	1,434,121	100,407
				Before extraction	Feces_A5	1,533,918	98,048
				2 mM in urine	Ave.	1,483,611	101,644
				(20 μ M in vials)	RSD, %	2.9	3.4
				(A/Sx100)	Recovery, %	123	133
				(A/Kx100)	Recovery, %	106	103

Table 2: Result of repeatability test (RSD%, n=5)

No.	Metabolites	1	2	3	4	5	Ave.	RSD, %
1	Alanine_2TMS	180,899	188,241	182,938	187,713	183,349	184,628	1.7
2	Ethanolamine_3TMS	486,231	499,319	511,776	491,099	499,867	497,658	2.0
3	Glycine_3TMS	113,691	111,586	113,681	113,461	115,306	113,545	1.2
4	Serine_3TMS	137,104	142,004	135,803	142,205	140,908	139,605	2.1
5	Threonine_3TMS	12,057	13,193	12,294	12,865	12,957	12,673	3.8
6	3-Aminoisobutyric acid_3	35,108	34,875	34,061	39,025	35,149	35,644	5.4
7	Threonic acid_4TMS	46,558	43,938	43,553	46,164	45,096	45,062	2.9
8	Creatine_3TMS	71,004	68,064	69,480	69,156	73,464	70,234	3.0
9	p-Hydroxyphenylactic aci	7,510	7,593	7,418	7,995	8,074	7,718	3.8
10	Aconitic acid_3TMS	18,060	17,740	18,623	18,335	17,708	18,093	2.2
11	Citric acid_4TMS	319,248	313,843	321,899	328,974	324,604	321,714	1.8
12	L-Lysine_4TMS	26,531	26,314	27,691	25,908	27,728	26,834	3.1
13	Tyrosine_3TMS	67,041	67,289	65,806	67,529	66,123	66,758	1.1
14	Gluconic acid_6TMS	23,901	21,094	22,881	23,351	20,520	22,349	6.6
15	Glucopyranuronic acid_5	90,468	97,764	91,691	99,144	93,550	94,523	4.0
16	Uric acid-4TMS	358,265	365,984	362,302	356,388	382,576	365,103	2.9