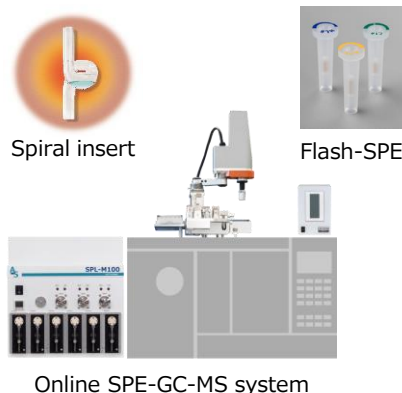


オンライン固相誘導体化-GC-MSシステムによる 唾液中メタボローム分析

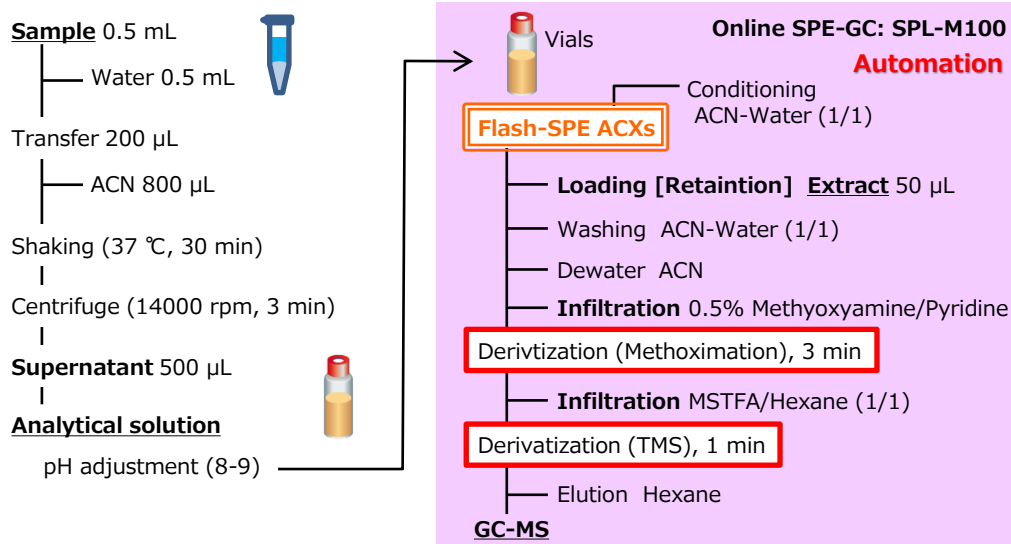
Introduction

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唾液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 $^{\circ}$ C(0.5 min)-50 $^{\circ}$ C/min-290 $^{\circ}$ 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 μ m

100 $^{\circ}$ C(2 min)-10 $^{\circ}$ C/min-320 $^{\circ}$ C(2min)

290 $^{\circ}$ C

Scan (m/z 70-600)

3.0-26 min



SPL-M100
for SPE-GC system

Sample



唾液

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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Results

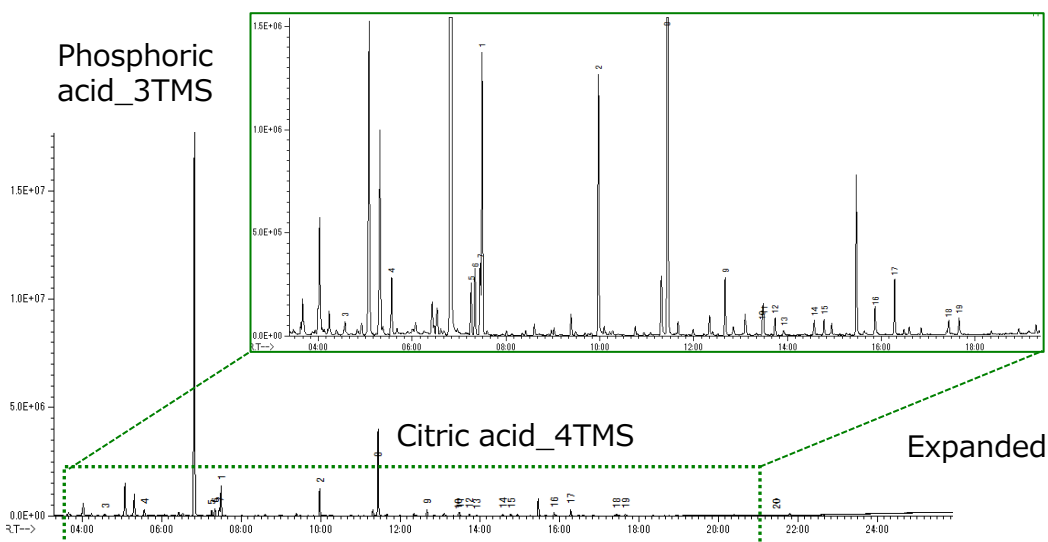


Figure: Total ion current chromatogram

Table: Result of recovery test (n=5)

Sample	No.	Norleucine_2TMS	Adipic acid_2TMS	Sample	No.	Norleucine_2TMS	Adipic acid_2TMS
Standard solution	S1	2,448,000	111,000	Saliva	Feces_K1	2,310,000	126,700
IS concentrations: 20 μ M in vials	S2	2,374,000	110,200	Dilution:	Feces_K2	2,025,000	115,300
	S3	2,313,000	102,700	10 times	Feces_K3	2,134,000	117,700
	S4	2,383,000	110,300	Spike period of IS:	Feces_K4	2,063,000	109,300
	S5	2,349,000	103,400		Feces_K5	2,197,000	120,900
	Ave.	2,373,400	107,520	After deprotenization, 20 μ M in vials	Ave.	2,145,800	117,980
	RSD, %	2.1	3.8		RSD, %	5.3	5.5
				(K/Sx100)	Recovery, %	90	110
				Saliva	Feces_A1	1,978,000	121,700
				Dilution:	Feces_A2	1,961,000	113,900
				10 times	Feces_A3	2,080,000	122,200
				Spike period of IS:	Feces_A4	1,968,000	111,400
					Feces_A5	2,054,000	122,300
				Before extraction, 200 μ M in saliva (20 μ M in vials)	Ave.	2,008,200	118,300
					RSD, %	2.7	4.4
				(A/Sx100)	Recovery, %	85	110
				(A/Kx100)	Recovery, %	94	100

Table: Result of repeatability test (RSD%)

No.	Metabolites	1	2	3	4	5	Ave.	RSD, %
1	Alanine_2TMS	124,100	108,500	112,100	113,300	114,200	114,440	5.1
2	Urea_3TMS	59,950	53,270	65,700	54,050	70,000	60,594	12.0
3	Proline_2TMS	481,000	418,600	437,000	432,700	454,600	444,780	5.4
4	Glycine_3TMS	354,300	319,700	323,500	322,100	340,900	332,100	4.5
5	Succinic acid_2TMS	49,020	46,330	45,910	44,520	46,790	46,514	3.5
6	5-Aminovaleric acid_3TMS	4,982,000	4,212,000	4,519,000	4,623,000	4,786,000	4,624,400	6.3
7	Putrescine_4TMS	457,300	404,100	422,900	413,200	438,100	427,120	4.9
8	Citric acid_4TMS	9,392	8,965	8,049	8,148	7,767	8,464	8.1
9	Ornithine_4TMS	74,060	62,220	63,890	63,740	68,900	66,562	7.3
10	Cadaverine_4TMS	131,400	113,400	119,100	116,700	124,200	120,960	5.8
11	Myristic acid_TMS	15,190	14,160	14,430	12,530	14,170	14,096	6.9
12	Lysine_4TMS	15,050	12,850	13,010	13,890	14,080	13,776	6.5
13	Tyrosine_3TMS	105,300	90,520	96,260	91,420	96,840	96,068	6.1
14	Palmitic acid_TMS	73,910	72,730	74,010	63,600	75,890	72,028	6.7
15	Uritic acid_4MS	122,400	105,400	111,000	111,300	108,700	111,760	5.7
16	Oleic Acid_TMS	17,690	18,570	16,440	15,330	16,250	16,856	7.6
17	Stearic acid_TMS	40,930	39,760	38,730	34,410	39,650	38,696	6.5