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Determination of methamphetamines in human saliva by GC-MS and two step injection on-column derivatization

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1. Overview

Two-Step Autoinjector for Analyzing Amphetamine-Type Stimulants and Other Amine-Type Drugs in human saliva.

2. Introduction

To Analyze drugs using GCMS,samples are derivatized for high sensitivity,Typically samples are derivatized by placing them in a glass tube or vial and reacting them with a derivatizing agent,but the entire process requires about 30 minutes.However,the two-step injection mode provides a much easier method for derivatization,where the sample and derivatizing agent are each injected into the GC sample injection port and derivatized within column.
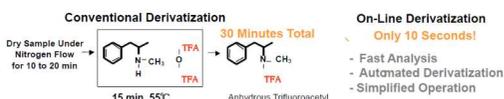
This instrument is designed to accomplish the two-step sample injection method described above.When this method was perfomed manually,two microsyringes were used to inject the test sample and derivatizing agent separately,but this system suctions both the derivatizing agent and test sample into a single,then injects the derivatizing agent and waits a few seconds before injecting the sample
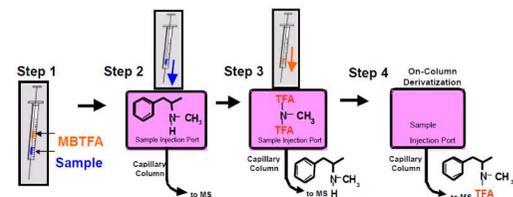
3. Methods and Materials

Sample extraction: Analytical sample was extracted by ATLAS-USIS,a analysis treatment laboratory automatic system.

LLE   Emulsification detection   Remove Supernatant   Adjust PH   Dry up   End, Constant volume
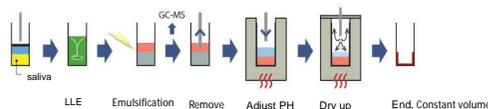
Shimadzu ATLAS-USIS

Shimadzu GCMS-QP2020

4. Result

Two-Step Mode Parameters

MBTFA Injection mode: 3(MBTFA fast injection)
MBTFA Suction Volume:1.0μL
Volume of Air Suctioned:2.0μL
Volume of air when injecting sample:1.0μL
Wait time after injecting sample before injecting MBTFA:4.0sec

GC-MS conditions

Injection mode: Splitl mode
Split ratio:10:1
Injection Port Temp:250 ℃
Column: SH-Rxi-5 Sil MS, 30m × 0.25mm × 0.25μm
Column oven temp.: 80℃(1.5min)_5℃/min_120℃_20℃/min_180℃(3min)
Carrier gas: Helium
Control Mode:Pressure
Column flow rate:1mL/min
Purge flow rate:3mL/min
Ionization mode: EI
Interface temp.: 250℃
Ion source temp.: 200℃
Measurement Mode:Fasst(Scan+Sim)
Scan Mass Range:m/z 45 to 310
Event time:0.3sec
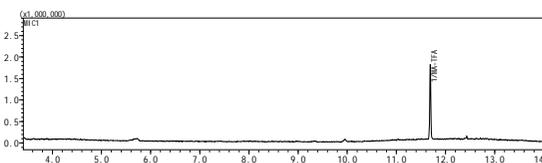
Figure 1 Representative Scan chromatogram (1ppm)

Table 1 Compound Information

| ID	R.T（min）	Compound	Target	Ref.Ions
1	11.69	MA-TFA	154	110，118
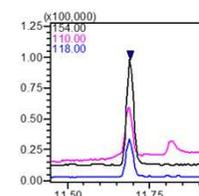
Figure 2 Representative SIM chromatogram(50ppb)

Table 2 Representative calibration curve

ID	Compound	Linear	Coefficient(r)
1	MA-TFA	Y =2458.284X- 11251.11	0.9993
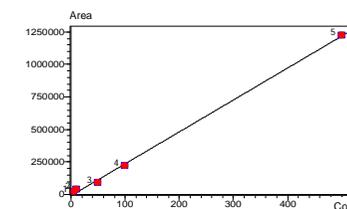
Figure 3 Representative calibration curve

Table 3. Repeatability results（n=5）

ID	Compound	Area					RSD%
		1	2	3	4	5	
1	MA-TFA	138876	137850	152139	146471	141544	4.13

Table 4. Recovery results（n=3）

ID	Spiked Amount	MA-TFA			AVE-Recovery（%）
		Recovery1（%）	Recovery2（%）	Recovery3（%）	
1	45ng	77.68	66.27	61.42	68.48
2	150ng	78.19	89.52	62.08	76.60

5. Conclusions

A Gas Chromatography and Mass Spectrometry and two step injection on-column derivatization method for determination of methamphetamines in human saliva was developed.The saliva samples were extracted by ATLAS-USIS.a new analysis treatment laboratory automatic system. Two step injection mode provides a much easier method for derivatization,where the sample and derivatizing agent are each injcted into the GC sample injection port and derivatized within the column.As a result, Methamphetamines TFA derivative was determined in 15 min with a limit of detcetion of 0.5 ng/mL and the peak area relative standard deviation was 4.13% when inject 50ng/mL standard solution 5 times in succession.The recoveries were in the range of 68.48~76.60% at the spiked levels of 45~150ng. The method showed a good linearity in detection of neurotransmitters and the linear correlation coefficients were greater than 0.999.Also the stability,and recovery were eligible for the analysis.This method was successfully applied to demermination of methamphetamines in human saliva.