Application Note : 1206

Phencyclidine(PCP) Confirmation in Oral Fluids by Laser Diode Thermal Desorption (LDTD) – MS/MS

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Introduction

Drug testing in Oral Fluids is a constantly evolving analysis procedure which benefits from increasingly sensitive methods of detection. Testing for drugs of abuse in oral fluids can strongly benefit the criminal justice field as a less invasive and cost-effective approach for drug detection when compared to blood or urine sampling. In a clinical environment, oral fluids can be used in patient screening for rapid confirmation of the presence or absence of orally administered drugs.

The LDTD ion source uses an infrared laser diode to desorb samples that have been dried onto a 96-well LazWell™ plate. The rapid desorption produces neutral species which are carried into a corona discharge region to undergo an efficient protonation and are subsequently transferred directly into the mass spectrometer for detection.

Oral Fluid Collection

- The Intercept® device by OraSure is used for saliva collection. Standard curves and QC's are prepared in the Oral Fluid Calibration Buffer.

Figure 1: Intercept® Oral Fluid Drug Test

LDTD-MS/MS System

Figure 2: LDTD system on Thermo Vantage Mass Spectrometer.

Sample Method

Extraction Procedure

100 µL Oral Fluid Calibration Buffer
20 µL IS (PCP-d5 at 250 ng/mL in MeOH)
100 µL NaOH (0.1N in Water)
- Mix

600 µL Ethyl Acetate
- Mix and centrifuge (2 min. / 14000 rpm)

Transfer 400 µL organic phase
Add 20 µL HCl (0.02N) in MeOH
- Evaporate to dryness

Reconstitute with 40 µL MeOH/Water (75/25)
Spot 2 µL of organic phase in LazWell plate
- Evaporate to dryness

LDTD-MS/MS Parameters

LDTD

Gas Flow:	3 L/min	
Laser pattern:	Time (s)	Power (%)
	0	0
	2	0
	5	45
	7	45
	7.1	0
	8	0

MS/MS Method

	Transition	CE	S-Lens
PCP	244->159	15	50
PCP-d5	249->164	15	50
Mode:	Positive		

Results and Discussion

Linearity Results

As shown in **Figure 3,** excellent linearity ($r^2 > 0.99$) with no signs of carryover effect is achieved in the quantification range (1 to 100 ng/mL).

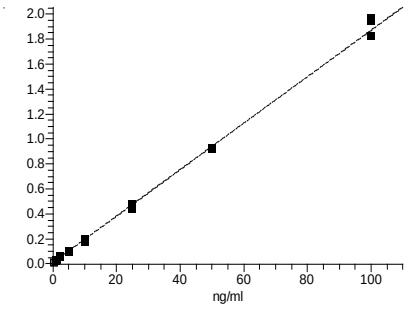

Figure 3: PCP standard curve

	r^2	Slope (ratio area / concentration)	y-Intercept
Run 1	0.9980	0.0862	0.0091
Run 2	0.9987	0.0222	0.0096
Run 3	0.9992	0.0220	0.0052

Table 1: Calibration Curve Parameters

Accuracy and Precision

As shown on **Table 2 and 3,** the inter-run and intra-run accuracy and precision are between 94.8 to 106.9% and 0.3 to 13.6%, respectively.

	QC-Low	QC-Med	QC-High
Conc. (ng/ml)	2.5	10	50
N	8	9	9
Mean (ng/ml)	2.67	9.73	48.78
%RSD	4.4	6.2	0.7
%Nom	106.9	97.3	97.6

Table 2: Inter-run precision and accuracy for PCP

	LLOQ	QC-Low	QC-Med	QC-High	ULOQ
Conc. (ng/mL)	1	2.5	10	50	100
N	3	3	3	3	3
Mean (ng/mL)	1.05	2.65	9.48	48.90	102.26
%RSD	13.6	5.6	7.3	0.3	4.0
%Nom	104.8	105.9	94.8	97.5	102.3

Table 3: Intra-run precision and accuracy for PCP

Detection limit (LOD)

A detection limit of 0.5 ng/mL can be reached with a blank interference of 23.1% at this concentration.

Stability Verification

Following the extraction process, all samples were stored at 4°C to evaluate the wet stability of the drug. After 66h, all samples were re-spotted and analyzed. Linearity, precision and accuracy were evaluated to determine the stability. **Table 4** shows that a wet stability of 66h is obtained with good precision and accuracy of LOQ standard.

The stability of dry samples in LazWell plate was also determined. All standards and QCs are spotted, dried and kept at room temperature for 6h and 4°C for 67h. Then, standards and QCs were analyzed and the linearity, precision and accuracy are verified. **Table 4** shows the dry stability results and the storage conditions of the LazWell.

	Wet Stability	Dry in LazWell (RT+ 4°C)
Time (h)	66	6h(RT) + 67h(4°C)
Temp. (°C)	4°C	RT+4°C
Conc. (ng/mL)	1	1
N	3	3
Mean (ng/mL)	1.06	1.04
%RSD	1.7	2.7
%Nom	107.9	103.5

Table 4: Stability Results for PCP

Correction Factor

Values reported represent diluted oral fluid. To convert to whole saliva, you must multiply by a factor of 3X.

Conclusions

The ease of use of the Intercept® oral fluid sampling device from OraSure provides an accurate and fast sampling method for many drugs of abuse. The combination of the oral fluid extraction procedure with the analysis speed of the LDTD-MS/MS is an ideal solution in high-throughput drug analysis.

A fast, sensitive and reproducible method for the analysis of PCP in oral fluid matrix is achieved using a simple buffer extraction method combined to the speed of analysis of the LDTD-MS/MS with a total sample-to-sample analysis time of **8 seconds**.

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