Application Note : 1208

Solid-Phase Extraction of Phencyclidine (PCP) in Urine and analysis by LDTD–MS/MS

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Introduction

Analysis of the Phencyclidine (PCP), a recreational dissociative drug, in urine can require a sample clean-up step to reduce the interference effect from the matrix. To obtain an optimal sample clean-up, the Silia*PrepX*™ HLB SPE cartridges are used in the extraction procedure prior to ultra-fast analysis by Laser Diode Thermal Desorption (LDTD).

The LDTD Ion Source uses an infrared laser diode to desorb samples that have been previously dried onto a 96-well LazWell™ plate after sample preparation extraction. 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.

Solid Phase Cartridge

The Silia*PrepX* HLB cartridge is used for the sample extraction procedure.

Figure 1: Silia*PrepX* HLB Polymeric SPE cartridge

Silia*PrepX* HLB Formats		
Formats	**Qty / Pk**	**Product number**
1 mL / 30 mg	100	SPE-P0002-01AA
3 mL / 60 mg	50	SPE-P0002-03BB
6 mL / 100 mg	30	SPE-P0002-06C
6 mL / 200 mg	30	SPE-P0002-06G
6 mL / 500 mg	30	SPE-P0002-06P
2 mL / 10 mg	1	96W-P0002-1A
2 mL / 30 mg	1	96W-P0002-AA

Table 1: Silia*PrepX* HLB product numbers

LDTD-MS/MS System

Figure 2: LDTD system on Thermo Vantage Mass Spectrometer.

Sample Method

Extraction Procedure

Cartridge:	Silia*PrepX* HLB (1 mL / 30 mg)
Activation:	1 mL MeOH
	1 mL NH_4OH (2% v/v) in Water
Load:	200 µL sample
	40 µL IS (PCP-d5 at 200 ng/mL in MeOH)
	200 µL NH_4OH (4% v/v) in Water
Wash 1:	1 mL Water/MeOH (50/50) + NH_4OH (2% v/v)
Wash 2 :	1 mL MeOH/Water (80/20) + NH_4OH (2% v/v)
Elution:	1 mL MeOH/Water (80/20) + 0.02N HCl
	Spot: 8 µL in LazWell plate

*Organic phase can be evaporated and reconstituted to further concentrate the sample

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 (10 to 1,000 ng/mL).

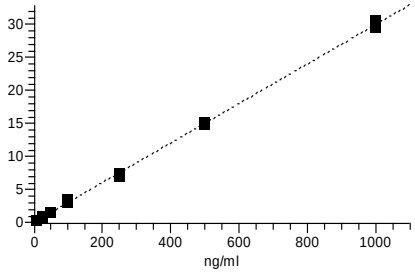

Figure 3: PCP standard curve

	r^2	Slope (ratio area / concentration)	y-Intercept
Run 1	0.9988	0.0298	0.0832
Run 2	0.9951	0.0319	0.0598
Run 3	0.9988	0.0307	0.0781

Table 2: Calibration Curve Parameters

Accuracy and Precision

As shown on **Table 2 and 3,** the inter-run and intra-run accuracy and the precision are between 96.1 to 104.3% and 1.4 to 10.1% respectively.

	QC-Low	QC-Med	QC-High
Conc. (ng/mL)	25	100	500
N	12	12	12
Mean (ng/mL)	24.89	102.04	503.64
%RSD	5.7	5.3	3.9
%Nom	99.6	102.0	100.7

Table 3: Inter-run precision and accuracy for PCP

	LLOQ	QC-Low	QC-Med	QC-High	ULOQ
Conc. (ng/mL)	10	25	100	500	1000
N	4	4	4	4	4
Mean (ng/mL)	9.61	24.89	104.34	504.51	1002.86
%RSD	10.1	6.0	6.6	1.4	1.5
%Nom	96.1	99.6	104.3	100.9	100.3

Table 4: Intra-run precision and accuracy for PCP

Recovery

The drug was recovered at 101% at 1,000 ng/mL of concentration (N = 4).

Stability Verification

Following the SPE extraction process, all samples were stored at 4°C to evaluate the wet stability of the drug. After stability time, all samples were re-spotted and analyzed. Linearity, precision and accuracy were evaluated to determine the stability. **Table 5** shows that a wet stability of 43h 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 and 4°C for stability. Then, standards and QCs were analyzed and the linearity, precision and accuracy are verified. **Table 5** shows that the dry stability of 42h is obtained with good precision and accuracy of LOQ standard.

	Wet Stability	Dry in LazWell (RT)
Time (h)	43h	42h*
Temp. (°C)	4°C	RT + 4°C
Conc. (ng/mL)	10	10
N	4	4
Mean (ng/mL)	10.49	9.82
%RSD	14.17	7.38
%Nom	104.9	98.2

Table 5: Stability Results for PCP

*Stability time :24h(RT) +18h(4°C)

Conclusions

The sample solid extraction using **Silia*PrepX* HLB** polymeric SPE cartridges ensures accurate and precise results with a linear standard curve ($r^2 > 0.99$).

A fast analysis can be achieved using LDTD-MS/MS system. This system allows a total sample-to-sample analysis time of **8 seconds**.

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