Application Note: 51995

Confirmation and Quantitation of Phencyclidine in Urine Using the ISQ Single Quadrupole GC-MS

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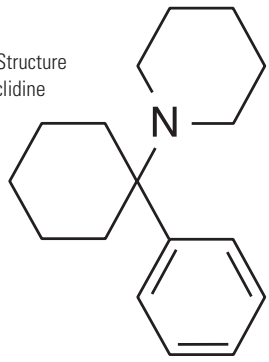
Key Words

- ISQ Single Quadrupole GC-MS
- Forensic Toxicology
- PCP
- SAMHSA Workplace Drug Testing

Overview

Phencyclidine (PCP, 1-phenylcyclohexylpiperidine, angel dust) is a recreational drug exhibiting hallucinogenic and neurotoxic effects, as well as being a legitimate veterinary tranquilizer. PCP is self-administered by either smoking, nasal insufflation and intravenous injection, or by oral ingestion.[1]

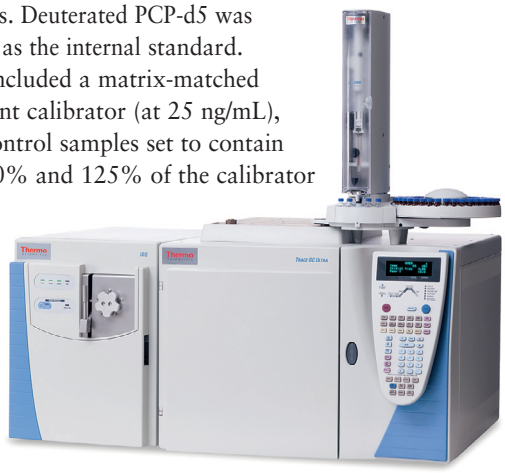
Chemical Structure of Phencyclidine



A forensic toxicology method for the confirmation and quantitation of phencyclidine in human urine was developed using the Thermo Scientific ISQ Single Quadrupole GC-MS system. This method adheres to guidelines published by the United States Substance Abuse and Mental Health Services Administration (SAMHSA),[2] the College of American Pathologists (CAP), the Society of Forensic Toxicologists (SOFT) and the European Workplace Drug Testing Society (EWDTS).

Methods

All validation samples were prepared as batches using a 2 mL sample size. Standard materials were obtained for calibration, and separate sources of parent PCP were used as controls. Deuterated PCP-d5 was employed as the internal standard. Batches included a matrix-matched single point calibrator (at 25 ng/mL), quality control samples set to contain PCP at 40% and 125% of the calibrator (6 ng/mL and 31.25 ng/mL respectively), and a negative control, which was blank urine with internal standard only. Thermo Scientific HyperSep Verify-CX solid phase extraction columns (200 mg, 10 mL, P/N: 60108-742) were used for sample extraction.



The ISQ™ mass spectrometer system was operated in selected ion monitoring mode (SIM), collecting 3 ions for the target compound, and 2 ions for the deuterated internal standard. A Thermo Scientific AS3000 II autosampler, and a Thermo Scientific TRACE GC Ultra gas chromatograph, equipped with a split/splitless injection port, provided sample introduction and separation. A 15 m × 0.25 mm ID × 0.25 µm film thickness Thermo Scientific TraceGOLD TG-5MS (P/N: 26098-1300) analytical column was used to enhance separation of the target compound from matrix components (Figures 1 and 2). Thermo Scientific ToxLab Forms software automated the acquisition and processing of all data, including quantitation and ion ratio confirmation calculations.

Batches were reviewed for conformance to quality control criteria regarding both quantitative and qualitative performance, based on accrediting agency guidelines. All quality controls within a batch demonstrated quantitative results within ± 20% of their expected (theoretical) concentration. Additionally, ion ratio ranges for qualifier ions for target compounds were established using ± 20% of the ratios calculated for the 25 ng/mL calibration standard. These ranges were used to assess ion ratio performance. ToxLab™ Forms performed ion ratio confirmations, retention time checking, and quality control conformance automatically as a part of batch acquisition and processing. For precision analyses, a coefficient of variation (CV) of <10% of the average calculated quality control amounts was required, and inter-day percent differences of calculated amounts also had to be less than 10%.

Results

- Assay linearity ranged from 2.5 ng/mL to 5000 ng/mL for PCP
- Limits of detection and quantitation of 2.5 ng/mL using a 2 mL sample size
- Intra- and inter-day precision of < 10% CV at the quality control levels of 6 ng/mL and 31.25 ng/mL
- Correlation coefficient (R^2) of 0.9990 for PCP (Figure 3)

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Chromatography

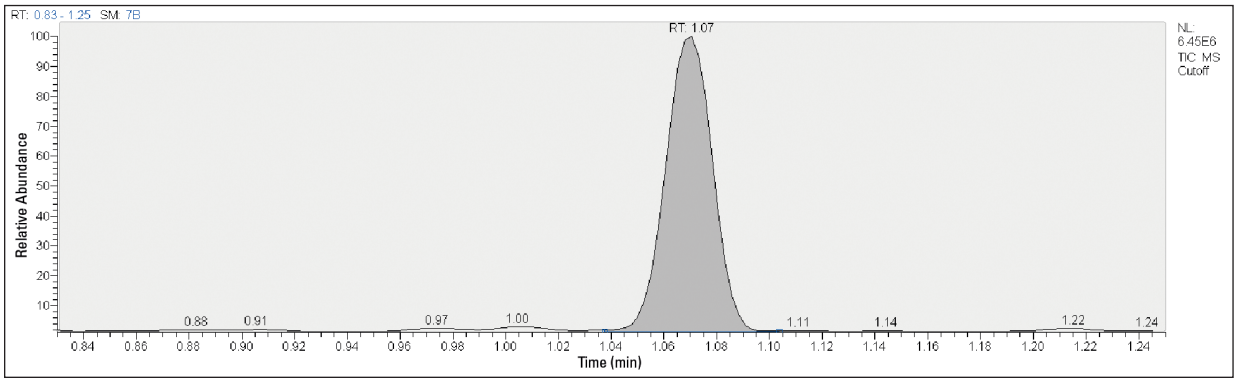

Figure 1: Total ion chromatogram of PCP from an extracted urine sample at the cutoff (25 ng/mL)

Extracted Ions

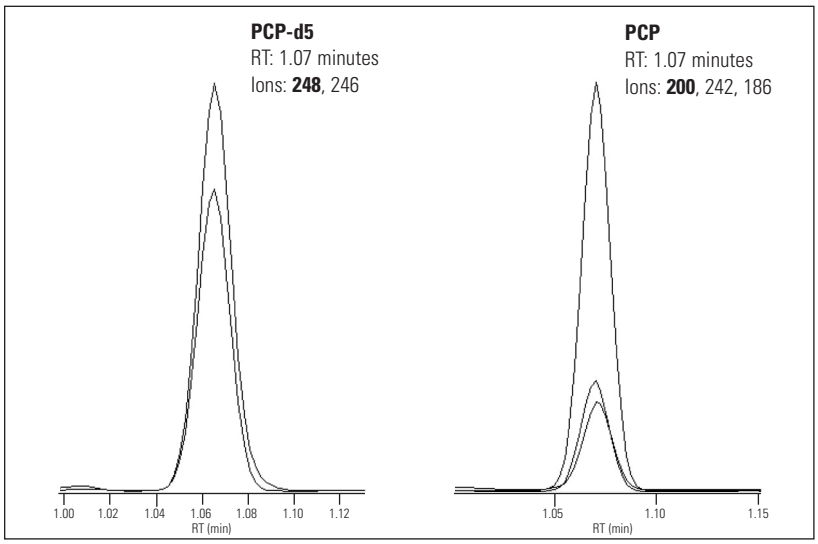

Figure 2: Extracted ion overlays of the PCP-d5 and PCP at the cutoff (25 ng/mL). Note that no interference is seen from coeluting matrix ions.

Linearity Study

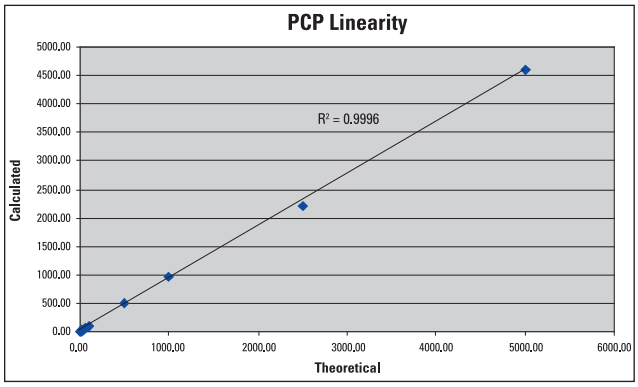

Figure 3: Linearity study results for PCP comparing calculated concentrations to the expected amounts at each level

Conclusions

A method was developed to demonstrate the performance of the ISQ GC-MS system for the confirmation and quantitation of phencyclidine in a urine matrix. The assay described offers a broad linearity (2.5 – 5000 ng/mL) to cover a wide range of analyte concentrations, thus reducing the need for dilutions or repeat extractions. Excellent precision was also demonstrated around the 25 ng/mL cutoff, with CV measurements of 10% or less over the study. Limits of detection and quantitation at 2.5 ng/mL ensure sensitive performance for retest and directed assay samples. The methodology described offers a means for a forensic toxicology laboratory to confirm and quantitate phencyclidine in human urine.

References

1. Disposition of Toxic Drugs and Chemicals in Man, Eighth Edition. Randall C. Baselt, Biomedical Publications, 2008.
2. Mandatory Guidelines for Federal Workplace Drug Testing Programs, Revised Mandatory Guidelines, Fed. Reg. 71857-71907 (Nov. 25, 2008)

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AN51995_E 08/10M