The Determination of Ketamine Levels in Hair Samples Using EI-GCMS



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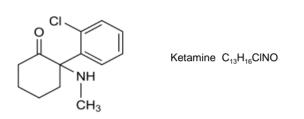


Accredited to ISO/IEC 17025

Introduction

Ketamine was first synthesised in the early 1960s. It is a powerful anaesthetic drug used in both human and veterinary treatment. Its perception altering properties has made ketamine a popular drug to be misused. Ketamine comes in liquid form but can also be obtained as a powder or tablet and so can be administered orally, nasally insufflated or injected.

An established benefit of using hair samples in drug testing when compared to urine, oral fluid and blood samples is the longer window of detection available. The aim of this poster is to provide information on the ketamine levels detected and the prevalence of the drug in hair samples collected in the UK.

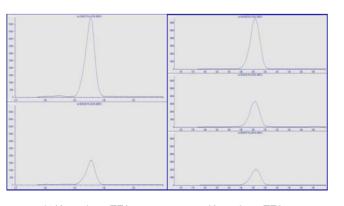


Methods

Since 2002, TrichoTech has received 2115 samples of hair requiring screening for ketamine by enzyme linked immunosorbent assay. 193 of these samples were analysed by electron impact - gas chromatography mass spectrometry (EI-GCMS).

The hair sections analysed were weighed and then washed with a solvent. The hair samples were submitted to alkaline digestion and then liquid:liquid extraction with a mixture of chloroform:iso-propanol. After solvent evaporation the residues were reconstituted in phosphate buffer pH 7.2. For the confirmation analysis by EI-GCMS, the samples were extracted using OASIS MCX solid phase extraction cartridges (Waters, Elstree, UK) or HCX Solid Phase Extraction cartridges (IST, Hengoed, UK). Prior to 2006 samples were analysed underivatised; since 2006 samples have been derivatised with trifluoroacetic acid anhydride (TFAA) (Sigma, Poole, UK) to produce the trifluoroacetyl (TFA) derivative. The samples were analysed by EI-GCMS using HP5973 (Agilent, Wokingham, UK) using single ion monitoring (Figure 1).

The cut-off for the assay was 0.2 ng/mg hair. Ketamine and d4-Ketamine reference standards were acquired from Cerilliant (LGC Promochem, Teddington, UK).



d4 Ketamine - TFA Ketamine - TFA Figure 1. Ion Chromatograms of the TFA derivative of d4 Ketamine & Ketamine The uncertainty of measurement (UM) is the associated variability of the analytical tests and was calculated by multiplying the relative standard deviation by 2 to provide a level of confidence of approximately 95%.

<u>Results</u>

The calibration curve for ketamine was measured over the range 3.6 to 120 ng/ml. The uncertainty of measurement for ketamine was 6% calculated from intra-and inter-day quality controls.

Ketamine was detected in 48 hair samples. The results are grouped by levels detected and are displayed in Figure 2.

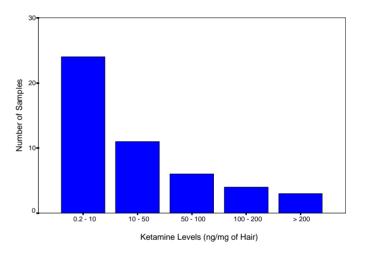


Figure 2. Ketamine Levels in Hair - Grouped by Levels Detected

Conclusions

- The levels detected in the 48 samples confirmed by EI-GCMS ranged from 0.2 to 750.8 ng/mg of hair. The highest frequency of results were in the 0.2 to 10 ng/mg hair range. The median was 10.1 ng/mg hair.
- The percentage of hair samples submitted to the laboratory for ketamine analysis with the presence of ketamine detected by EI-GCMS was 2.3%.
- The present limit of quantification for ketamine is 0.2 ng/mg hair (assuming a 10 mg sample). The validated method is both sensitive and reproducible for the quantification of ketamine in hair samples.
- 4. The uncertainty of measurement of ketamine was determined by the EI-GCMS method and was found to be 6%. The detection of drugs in hair has an additional variable which is the extraction efficiency of the drug from within the hair matrix. The uncertainty associated with the extraction efficiency is not possible to determine, because of the drug's intrinsic nature within the hair matrix and as such its contribution to the overall analytical uncertainty is not evaluated. This is one reason why it is not possible to correlate the levels detected in hair with the amount of drug used by the individual. The levels of drugs detected in hair are best used as a guide to changes in use in the individual when sectional analysis is performed or when two different periods are compared in the same individual.