

# An improved method for cyanide determination in blood using solid-phase microextraction and gas chromatography/mass spectrometry

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GIAMPIETRO FRISON, FLAVIO ZANCANARO, DONATA FAVRETTO and  
SANTO DAVIDE FERRARA

Forensic Toxicology and Antidoping, University Hospital of Padova, Via Falloppio 50, I-35121 Padova, Italy

**AIMS:** A new method is described for the qualitative and quantitative analysis of cyanide (CN), a very short-acting and powerful toxic agent, in human whole blood. It involves the conversion of CN into hydrogen cyanide (HCN) and its subsequent headspace solid-phase microextraction (SPME) and detection by gas chromatography/mass spectrometry (GC/MS) in selected ion monitoring (SIM) conditions.

**METHODS:** Method development consisted of: comparative studies of adsorption efficiency of five different SPME fibers; optimisation of SPME conditions: salt effect, adsorption and desorption times, adsorption temperature, carryover effect; choice of internal standard; optimisation of GC/MS conditions: type of column, injection conditions, temperature programme, single ion monitoring parameters.

**RESULTS:** Optimising the conditions for GC/MS and SPME led to the choice of a 75- $\mu$ m carboxen/polydimethylsiloxane SPME fiber which, under the analytical conditions used (adsorption and desorption times of 10 and 3 min, adsorption temperature of 30°C), showed no carryover effects; D<sub>3</sub>-acetonitrile as internal standard; a capillary GC column with a polar stationary phase. Method validation was carried out in terms of linearity, precision and trueness in both water solutions and blood. The limit of detection (LOD) and limit of quantitation (LOQ) were only determined in water solutions. The assay is linear over three orders of magnitude (water 0.01-10, blood 0.05-10 mcg/mL); and the LOD and LOQ in water were determined to be 0.006 and 0.01 mcg/ml, respectively. A good intra- and inter-assay precision was obtained, which was always < 8%.

**CONCLUSIONS:** The described method proved suitable for the determination of CN in blood. It benefits from the choice of: HS-SPME sampling, which avoids the necessity of cryofocusing; a CBX/PDMS fiber, used here for the first time to determine CN in blood; a capillary column of the FFAP type, which is easy to use in conjunction with MS detectors and suitable for a variety of volatile analytes; a deuterated internal standard. The method is simple, fast and sensitive enough to be applied for the rapid diagnosis of cyanide intoxication in clinical and forensic toxicology.

**KEYWORDS:** *Cyanide, SPME, Mass Spectrometry*

**Corresponding author:** [giampietro.frison@sanita.padova.it](mailto:giampietro.frison@sanita.padova.it)