K6 Determination of 2-Chloracetophenone in Air by SPME-GC/MS

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After attending this presentation, attendees will understand the analysis of 2-Chloracetophenone in air by SPME extraction and GC/MS analysis.

This presentation will impact the forensic community and/or humanity by demonstrating a robust, sensitive and simple analytical method for the determination and measurement of 2-Chloracetophenone (CN) in air. Sampling by SPME requires no pumps, and no polluting organic solvents, thus reducing the sampling cost.

Laboratory and field evaluations were performed to validate the solidphase microextraction (SPME) technique for the determination of CN in air. This is a new, rapid air sampling/sample preparation methodology suitable for use in the working environment and in forensic applications. The Threshold Limit Value (TLV)-Time Weighted Average (TWA) for CN of 0.32 mg/m^3 is recommended by the American Conference of Governmental Industrial Hygienists (ACGIH).

CN is widely used as tear gas by law enforcement agents and also by civilians for the purpose of personal protection. Recently, there has been an increase in crimes involving robbery and rape using tear gas sprays (Kataoka M. et al., J Forensic Sci 2002; 47(1): 44-51). Exposure to this lachrymator produces an intense sensory irritation of the eyes, contact dermatitis, and respiratory distress.

SPME, introduced by Pawliszyn et al. in recent years, is a solvent-free technique that combines sampling and sample preparation in a single step. The SPME sampler is a 1 cm long fused-silica fiber core coated with a polymeric phase. The coated fiber can be moved into and out of a stainless steel needle (area of needle opening, 0.00086 cm^2). By retracting the coated fiber into its needle (Z, from 1 to 35 mm) during sampling, SPME can be used as a TWA diffusive sampler.

In the present work, a method involving gas chromatography/mass spectrometry (GC/MS) and SPME was developed for quantitative analysis of CN in air. The TWA concentration of CN was analysed in a military storage facility, containing tear gas canisters, for a period from 240 to 480 min to evaluate the risk.

For laboratory validation and field TWA sampling of CN with the SPME technique, a 65 µm fiber in PDMS/DVB was used. The sampling was performed adopting a Z value of 3 mm and exposing to the air for periods of time from 60 a 480 min. After the sampling, the fiber was analysed with GC/MS.

Vapors of CN (0.032-3.2 mg/m^3) were generated by a syringe pump in a dynamic system with monitored temperature (20 and 35°C), relative humidity (10 and 80%) and air velocities (0.2 and 83 cm/s). Every thirty minutes, 200 µl of CN generated vapors were injected into the GC/MS system to monitor the dynamic system CN concentrations.

The theoretical sample rate (SR, ml/min) of CN was estimated by the Fuller-Schettler-Giddings diffusion coefficient. The experimental SR was obtained by comparing GC/MS standard solutions of CN with the amount of CN adsorbed into the fiber allowed in the sampling chamber at known concentrations.

Statistical analysis of laboratory validations demonstrated that temperature, relative humidity and air velocity did not affect the absorption efficiencies ($p<0.05$). The theoretical and experimental SR values (0.01086 and 0.00891 ml/min, respectively, at 25°C for Z=3 mm), were in good agreement. The method's precision (n=5) was established to be 10% relative standard deviation for 0.032 mg/m^3 and 8% RSD for 3.2 mg/m^3 (for 240 min sampling and Z=3 mm). The total on-column limit of quantification (LOQ) was 5 pg (0.460mg/m^3/min), and the linearity of the method ranged from 5 to 5000 pg (105 m/z).

The results obtained from the field study for the determination of the TWA concentration of airborne CN showed values ranging from 0.049 to 0.206 mg/m^3.

2-Chloracetophenone , SPME GC/MS Detection, Chemical Weapons

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