Quantitative Headspace Analysis by Cryoadsorption: Applications in Detection of Explosive Materials, Fire Retardants, and Food Spoilage

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The use of purge and trap methods for sampling volatile organic compounds prior to chromatographic analysis is a mature technology. Application to low volatility compounds has been far less facile and sensitive. Especially problematic has been applications that require precise quantitative analysis and analyses as a function of sample temperature. In this talk we discuss the application of short lengths of alumina coated PLOT columns as purge traps, and operate the traps at low temperature during the collection cycles to improve efficiency, in a method called cryoadsorption to analyze the headspace of explosive materials, car parts and rotten meat. Detection using headspace analysis is complicated by mixture effects. For example, explosive materials are often mixed with taggents (materials added intentionally for identification), plasticizers, motor oil and binders (materials added to make all of the components cohere). Additionally, the major component in the mixture is not necessarily the easiest component to detect; however, this fact is not important as long as several of the components can be detected. We have applied this method as a function of temperature to a medium volatility solid, coumarin, as a demonstration. Further application of this method was to the pure explosive compound 2,4,6-trinitrotoluene (TNT), the practical military explosives C-4, Semtex-A and Semtex-H (plastic explosives made from RDX and PETN (trinitro-triazacyclohexane and pentaerythritol trinitrate, respectively), and various detonator cord (lead azide) and detonator sheet. The temperature dependent measurements, presented in terms of the van’t Hoff equation, give a predictive capability that is critical to standardization and calibration of detection devices used in the field. We also applied this method to the detection of flame retardant compounds in the headspace of car parts, including sections from the dashboard, seat upholstery and ceiling materials. We have identified tris(1,3-dichloroisopropyl) phoshate (TDCPP) a flame retardant used as an alternative to the phased-out polybrominated diphenyl ether flame retardants. Last, we applied this method to the identification of compounds in the headspace of rotten meat with the goal of developing a procedure for detecting food spoilage during food processing and packaging.