



Technical note

Technical note: Headspace analysis of explosive compounds using a novel sampling chamber



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ABSTRACT

The development of instruments and methods for explosive vapor detection is a continually evolving field of interest. A thorough understanding of the characteristic vapor signatures of explosive material is imperative for the development and testing of new and current detectors. In this research a headspace sampling chamber was designed to contain explosive materials for the controlled, reproducible sampling and characterization of vapors associated with these materials. In a detonation test, the chamber was shown to contain an explosion equivalent to three grams of trinitrotoluene (TNT) without damage to the chamber. The efficacy of the chamber in controlled headspace sampling was evaluated in laboratory tests with bulk explosive materials. Small quantities of TNT, triacetone triperoxide (TATP) and hexamethylene triperoxide diamine (HMTD) were separately placed in the sampling chamber, and the headspace of each material was analyzed by gas chromatography/mass spectrometry (GC/MS) with online cryogenic trapping to yield characteristic vapor signatures for each explosive compound. Chamber sampling conditions, temperature and sampling time, were varied to demonstrate suitability for precise headspace analysis.

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1. Introduction

Vapor sampling is a common approach to the detection of trace explosive materials carried out for security and safety purposes, at home and abroad. Vapor detection of explosives is often preferred over particle detection as it allows for non-contact detection of items such as buried landmines, baggage, or moving people. Vapor detection methods are complicated by the large variation in the vapor pressures of explosives compounds, leading to large variations in expected vapor concentration at a sampling location [1]. For example, nitroglycerine or nitrocellulose-based explosives such as smokeless powders and dynamite, as well as the peroxide explosive triacetone triperoxide (TATP), have considerable vapor pressures and thus can be detected more readily in the vapor phase. However, many other explosive compounds are significantly less volatile, presenting concentrations that are several

orders of magnitude lower. Fortunately, these less volatile explosive compounds are often associated with non-explosive compounds that are more volatile and present at more favorable vapor concentrations. As an example 2,4-dinitrotoluene (2,4-DNT) is reliably detected in the headspace of trinitrotoluene (TNT), which have estimated vapor pressures of 4.11×10^{-7} and 9.15×10^{-9} atm, respectively [2]. Additives such as taggants, solvents, oxidizers, and plasticizers are also often more volatile than their parent compounds and can be more easily detected in the vapor phase [1]. Additionally, materials, such as hexamethylene triperoxidediamine (HMTD), have volatile degradation products detectable in its characteristic vapor signature [3].

The development and testing of sensors and other instrumental and biological detection methods for explosives is continuously evolving, requiring a keen understanding of explosive vapor signatures. It is also crucial that this fundamental research be carried out in a controlled and reproducible fashion. In this research, a headspace sampling chamber was designed to contain small quantities of explosive materials for the analysis of the vapor signatures in a temperature and humidity-controlled environment.

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2. Chamber design

A custom headspace sampling chamber was designed with emphasis placed on experimental flexibility and reliability of vapor transfer. A number of practical considerations were made in the design of the chamber, including sample isolation from laboratory air contaminants, varying sample dimensions, controlled sample environment (i.e. temperature and humidity), surface inertness, and a flexible sampling interface.

The chamber and lid, fabricated from 304 stainless steel, are shown in Fig. 1. The lid is held in place by four 0.25–20 × 2.75 in stainless steel screws to compress Viton o-rings around the chamber openings to ensure that the chamber is air and water tight. The internal volume of the chamber was pressure tested with nitrogen at 40 psig (276 kPa) for 8 h with no leaks detected. The internal surface of both the chamber and lid were coated with Sulfinert 2000 (SilcoTek, Bellefonte, PA) to render the surface chemically inert. This inert coating significantly improves sample recovery and reduces carry over. To avoid static charge on the chamber, a 4–40 hole was drilled and tapped on the chamber and attached via a grounding strap to Earth ground during all experiments.

A stream of purified air was flowed into the inlet at the lower back of the chamber, and released through an outlet at the top of the chamber lid. The air was supplied from a source of clean air that was temperature and humidity controlled (Miller–Nelson Test Atmosphere Generator, Model HCS-501, Assay Technology, Inc., Livermore, CA). The outlet was attached to a heated transfer line via a one-eighth inch national pipe thread (NPT) fitting threaded into the chamber lid. Swagelok fittings of various sizes can be adapted to the NPT fitting for the attachment of other instruments and transfer lines.

An insert was also fabricated (Fig. 2) to fit into the center of the chamber with a centered pocket 2.5 cm in diameter and 1.9 cm deep. The sample pocket was surrounded by 115 channels, each of which was 1.9 mm in diameter. The arrangement of channels was designed to ensure laminar flow over a range of desired sampling conditions and ensure that the air would have adequate residence time to reach thermal equilibrium within the chamber. Avoiding turbulent flow and associated eddy currents minimizes the chance of lofting small particles into the air stream or undesirable sampling anomalies. A total air flow rate below 100 mL min⁻¹ was calculated to result in a Reynolds number under 2300, thus laminar airflow was maintained. Multiple inserts with varying dimensions were fabricated, allowing for varying sample dimensions and amounts of explosive materials.

Channels along the vertical corners of the chamber and horizontally across the lid and the base of the chamber provide a serpentine path for recirculated water from a temperature-regulated water bath (Isotemp Refrigerated/Heated Bath Circulator 4100 R28, Fisher Scientific), that pass through the chamber walls to provide a uniform, controlled temperature around the sample. As an example, the chamber was heated from 8 °C to 50 °C in 48 min (Fig. 3). The time necessary to reach this uniform temperature is due both to the mass of the chamber as well as the time necessary to heat the 4.5 L of water in the recirculating water bath. For typical experiments, the chamber temperature was allowed to reach equilibrium prior to introducing the sample. With this arrangement, the operational temperature of the chamber was from below –10 °C to above 110 °C through the use of mixed solvents in the recirculating bath. Through the use of infrared thermal imagery (IR Flexcam Thermal Imager, Model TI55FT-20, Fluke, Everett, WA), the heat radiating from the water channels can be clearly visualized in both the lid and body of the chamber (Fig. 3), indicating a continuous flow of water through

the channel path and, in the end, a uniform and stable temperature distribution.

3. Experimental methods

3.1. Durability testing

The durability of the chamber was tested by placing a known quantity of high-explosive material into the chamber, attaching the chamber lid as normal, and then detonating the explosive. This procedure was performed by the Federal Bureau of Investigation, Explosive Unit (FBI-EU) scientists at an approved explosive detonation area. Prior to detonation, the sample insert was removed from the chamber. A single blasting cap with 1 g of RDX and a 2.5 cm × 1.7 cm piece of Composition 2 (C2) were placed into the chamber (total detonation equivalent to approximately 3 g of TNT) [4,5]. The chamber was closed with the stainless steel screws, and detonated remotely.

3.2. Headspace sampling

Proof-of-concept experiments were carried out with three types of explosives representing varying functionalities and volatilities: TNT, TATP and HMTD. Purified, bulk TATP and HMTD were obtained from FBI-EU scientists. Solid TNT was obtained by evaporating off the solvent of a 10 mg mL⁻¹ TNT solution in methanol/acetonitrile (1:1) obtained from AccuStandard (AccuStandard, Inc., New Haven, CT). The explosive materials were allowed to equilibrate in plastic, antistatic vials (ESD Plastic Containers, Yorba Linda, CA), in the headspace chamber for 30 min prior to sample collection. Sampling and analysis conditions were optimized for each analyte(s) comprising the vapor signature of the explosive material (Table 1).

An Agilent gas chromatograph/mass spectrometer (GC/MS), model 7890/5977 (Agilent Technologies, Santa Clara, CA) configured with a 30 m × 0.32 mm i.d., Rtx-Volatile Amine column (Restek, Bellefonte, PA) was used for all analyses. The headspace of each analyte was transferred from the sample chamber to the GC/MS via a SilcoTek-coated stainless steel transfer line heated to 85 °C and attached by Swagelok fittings. The headspace was collected and concentrated using an online cooled injection system (CIS) (CIS-G4, Gerstel Inc., Linthicum, MD). After a pre-determined volume of sample air passed through the CIS sorbent, sample analytes were desorbed from the CIS onto the head of the GC column.

Sampling parameters were varied to demonstrate the versatility of the chamber. While in this work chamber temperature and sampling time were altered, other variables could also be changed, including flow rate and humidity. For this set of experiments, a fresh 10 mg sample of solid TNT from the evaporated solution was used in each test. TNT headspace was collected from the chamber at 100 mL min⁻¹ for 15 min (1.5 L), 30 min (3 L), and 60 min (6 L) at 25 °C. Additionally, 3 L of headspace of similar samples was collected with the chamber at 25 °C, 35 °C, and 50 °C. All other parameters remained the same.

4. Results and discussion

4.1. Durability testing

The headspace sampling chamber maintained structural integrity following a high-explosive material detonation equivalent to three grams of TNT. Fig. 4 depicts the container immediately following the detonation. No observable structural damage was done to the chamber, though a black carbon residue covered the inside surface. During the blast, pressure was released through vent holes where the air flow enters and exits during normal

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