

Ultrasonic, Non-Invasive Classification/Discrimination of Liquid Explosives (LEs) and Threat Liquids from Non-Threat Liquids in Sealed Containers

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Abstract—This paper focuses on a set of laboratory measurements acquired with a Container Screening Device (CSD) that has been applied to several types of liquids in small commercially available plastic containers. The objective of this study was to further understand the critical acoustic measurement discrimination/sensitivity issues associated with quantifying the effectiveness of the approach for classifying and discriminating liquid threats from benign and commercially available liquids. A list of pertinent threat liquids was generated and measurements were conducted to demonstrate the CSD's ability to acquire accurate and repeatable acoustic information for effectively classifying and discriminating these liquids from non-threat liquids such as wine, whiskey, water, shampoo, etc. The device was used to measure the acoustic velocity, (the primary discriminator) and relative attenuation of 180 liquids at room temperature, while recording the key parameters that play a role in the accuracy and precision (repeatability) of the measurement. Density data were manually generated and evaluated to determine the effects of an additional acoustic signature on the discrimination algorithm. Results showed that by using a 3-parameter measurement approach (velocity, attenuation, and density), the methodology can be effectively employed for the rapid and accurate classification/discrimination of threat liquids versus non-threat liquids in small, carry-on, standard "stream-of-commerce" containers.

1. INTRODUCTION

Government agencies and homeland security organizations are searching for more effective approaches for dealing with the increasing demand for inspections involving potential threat liquids and hazardous chemicals, including liquid explosives (LEs). The quantity and variability of hand-held and cargo-sized containers being shipped worldwide drives the need for rapid and effective ways for conducting non-intrusive inspections of liquid-filled containers of a diverse range of types, shapes and sizes. Such inspections need to quickly classify/discriminate between liquids within containers and also ascertain the presence of unexpected objects within a container. The science base, methodology and prototype device for classification/discrimination between classes of liquids has been developed.

A methodology that enables rapid and reliable inspections of liquid-filled containers in a nondestructive and non-invasive fashion would be of significant benefit to Customs and Border Protection (CBP) and Transportation Security Administration (TSA) personnel. Further, if this methodology provided a means to effectively classify and discriminate threat liquids from non-threat liquids, this capability would address a set of critical tasks typically encountered by military, law enforcement, and intelligence community personnel charged with enforcing government policies, maintaining public safety, and ensuring national security. The most common approaches today include banning liquid substances altogether, severely limiting the quantities of liquids through a port-of-entry, or physically sampling the contents of a container and performing a field or laboratory analysis. Prohibition or strict limitations of quantities are extreme measures, while field or laboratory analysis of liquid contents may be very time-consuming, costly, or both. Opening sealed containers of unknown origin is dangerous and associated with a host of potential hazards. Often these types of inspection activities require using physical protection as well as other costly precautions while potentially endangering field personnel. X-ray technologies are often quite costly, bulky, inadequate (in their ability to penetrate or characterize liquid contents), and impractical for real-time or immediate response scenarios. Commercially available technologies that claim to provide these types of capabilities do not have the appropriate measurement sensitivity, are not field-hardened, have insufficient reliability (high false alarms), require a high level of expertise for operation, and are typically not well-suited for the wide variety of containers and liquids that are routinely encountered in the field.

The Pacific Northwest National Laboratory (PNNL) has developed a prototype container screening device (CSD) using pulse-compression acoustics, where swept frequency ultrasonic energy is applied into the container and physical properties of the liquid contents are measured. The return echoes from these pulses are analyzed in terms of time-of-flight, amplitude decay, and frequency content (all as a function of temperature), to extract the speed of sound, relative attenuation, and density of the liquid contents. These properties are then evaluated using an advanced statistical analysis method in conjunction with a database of

both threat and non-threat liquid physical properties to provide classification and discrimination results. This paper focuses on a set of laboratory measurements acquired with the CSD as applied to several types of liquids in small, commercially available plastic containers.

2. PRINCIPLES OF OPERATION

The Container Screening Device (CSD) employs frequency-modulated (FM) chirp excitation and pulse-compression signal processing techniques to measure ultrasonic velocity and a relative attenuation value for liquids within a container, and is capable of determining other acoustic properties from through-transmission, contact measurements over a wide frequency range. Recent algorithm developments are beginning to address the issues of container wall variations and thickness. A description of the basic science, measurement approach and sources of variability in the measurement will be presented and laboratory measurements acquired from a suite of commercial products and precursor liquids used in the manufacturing of Homemade Explosives (HMEs) will be given.

The CSD uses two 5-MHz ultrasonic transducers mounted on extendible arms that can be placed on opposite outside walls of a container to actively transmit and receive an acoustic pulse through the liquid contents. The instrument simultaneously records the acoustic echo, the distance between the transducers, and the external temperature of the container under examination. An attached tablet PC is loaded with custom software that provides a graphical user interface (GUI). The patented device [1] processes the acoustic pulses and accurately measures the time-of-flight and acoustic energy to compute the temperature-corrected acoustic velocity (speed of sound) and relative acoustic attenuation of the liquid for characterization and classification purposes. These pulses are also used to determine if there are hidden compartments, contraband, or other anomalous items hidden inside a container or bulk solid item. Additional acoustic properties (such as density, acoustic absorption spectra, etc.) may also be obtained and are currently under study at PNNL.

The prototype device was designed to focus on small containers, specifically over a size range of a 1-in. diameter test tube to that of a 10-in. diameter container. The current measurement platform was conceptualized and developed using automated distance and temperature measurement protocols, state-of-the-art broadband, piezo-composite ultrasonic transducers, advanced electronics circuit design, and advanced signal processing algorithms and software that directly address pertinent inspection/examination issues for small containers.

In order to obtain high-accuracy time-of-flight (TOF) measurements, traditional ultrasonic methods resort to the use of higher frequency transducers. However, many containers (plastic, glass) and/or fluids (castor oil, honey)

exhibit high attenuation properties, which do not allow higher frequencies to penetrate effectively. This requires additional energy at the transducer and leads to longer duration “ringing” of the excitation pulse, effectively masking return signals that may arrive from the far wall of smaller diameter containers, degrading the ability to detect and resolve signals of interest. The reduction in allowable frequencies also reduces the TOF resolution/accuracy, which precludes the use of typical, commercially available ultrasonic technologies. Therefore, research was directed toward employing an advanced pulse compression technique, whereby large amounts of ultrasonic energy are transmitted into the medium, resulting in higher signal-to-noise ratios (SNRs) and more accurate TOF measurements.

Pulse compression is a technique that has been employed in both RADAR [2-3] and medical ultrasound [4-5]. It is used to transmit large amounts of energy over a long period of time without sacrificing temporal resolution. A wide bandwidth, long duration frequency chirp is commonly used to excite the source (transmitting transducer). This pulse is received by one or more receiving transducers. Cross-correlation between the transmitted pulse and the received pulses results in a waveform containing the same time, amplitude and spectral information as the received pulse. Pulse compression has recently been used with broadband air-coupled transducers, where energy transmission, signal-to-noise ratio (SNR), and TOF accuracy are relatively low compared with conventional direct coupled ultrasound [6-9]. Gan et al. [7] found that pulse compression provided the air-coupled system with the ability to detect received pulses even when they were well below the noise floor due to the frequency-encoded transmitted pulse. In addition, they were able to resolve closely spaced return echoes from various reflection sources with high accuracy, which was not possible with typical ultrasonic tone-burst or square-wave excitation technologies. The pulse compression technique has also been used in conjunction with air-coupled ultrasound to interrogate food containers [10] and detect foreign objects within food materials [11]. More recent work at PNNL has expanded to the use of pulse compression methods in slurries and in air-coupled applications for both material property measurements and ultrasonic imaging and flaw detection applications.

Poor SNR is very common in air-coupled ultrasonic testing due to impedance mismatches between air and most other materials. Traditional ultrasound may improve the SNR by simply using high-power pulse transmission, commonly using tone-burst excitation techniques. A long-duration tone burst can efficiently transmit large amounts of energy into air or any other medium. However, tone-burst excitation generally results in poor TOF accuracy and provides a narrow-banded response in the frequency domain. A long-duration frequency sweep (chirp) can also efficiently transmit energy into a medium; however, signal processing techniques can be used to convert a long-duration chirp into a compressed broadband pulse for extremely accurate TOF

measurements and a correspondingly broad-banded response in the frequency domain.

Pulse compression is a signal processing technique carried out by cross-correlating a transmitted chirp with a received signal. The cross-correlation function effectively locates the specific frequency pattern within the received waveform and outputs a compressed waveform containing information associated with the frequency-dependent amplitude, and transit time of the transmitted pulse. This procedure is extremely useful when trying to locate echoes within a signal whose amplitude is well below that of the noise floor. Gan et al. [7] demonstrated an increased SNR using the pulse compression technique to locate an echo within a noisy return signal. The energy associated with the compressed cross-correlation signal is directly related to the duration of the transmitted chirp pulse. Therefore, to achieve a higher SNR, a longer duration pulse is employed. As stated earlier, the pulse compression technique results in accurate TOF measurements. This is directly related to the frequency bandwidth of the transmitted and received pulses, where a larger bandwidth results in higher TOF resolution. Effectively, the cross-correlation output will appear as a broadband pulse with a width inversely proportional to the bandwidth of the transmitted chirp. This phenomenon leads to another advantage of the pulse compression technique also known as deconvolution. For a system containing multiple echoes, a traditional ultrasonic tone-burst configuration would not be able to discriminate between closely spaced echoes. However, a long-duration, broadband transmitted chirp results in a compressed cross-correlation function having multiple narrow-width pulses, which allows multiple echoes to be easily resolved. Details of the measurement methodology and algorithm development have been reported by Tucker et al. and Diaz et al. [12-13].

3. MEASUREMENT METHODOLOGY

The study focused on measuring and analyzing room temperature ultrasonic data with the CSD prototype on a set of 180 liquids that included both commercially available (benign) liquids, and a subset of hazardous, flammable and explosive compounds dissolved in solvents including 2,4,6-Trinitrotoluene in acetonitrile, pentaerythritol tetranitrate (PETN) in acetonitrile, nitroglycerin in acetonitrile, RDX in acetonitrile/DMSO (90:10 ratio), and HMX in acetonitrile/DMSO (90:10 ratio) at multiple concentrations. The benign products spanned a range of common liquid commodities that included cosmetics to personal hygiene items, cleaning liquids to car products, beverage items to liquid medication and so on, in order to incorporate a wide array of liquids representing a wide variety of potential acoustic property ranges. Table 1 provides a high-level summary of the liquid categories gathered for this study and also provides example liquids for each category.

The prototype CSD platform was used to measure the acoustic velocity and relative attenuation of 180 liquids at room temperature (approximately 70°F) while recording the

Table 1 – General Liquid Categories Studied with CSD (24 Threat Liquids, 156 Non-Threat (Benign) Liquids)

Liquid Category	Total Number of Liquids in Category
Hair Care Products	14
Automotive Products	18
Liquors	7
Toiletries	8
Cleaners	22
Medicines	12
Consumable Beverages	57
Skin Care Products	18
Threat Liquids and Flammables	24
Total Number of Liquids	180

key parameters that play a role in the accuracy and precision (repeatability) of the measurement. The CSD algorithms were designed to detect and measure discrete echoes in the container walls to compute wall thickness prior to calculating the acoustic velocity. In many cases, this algorithm works well; however, more work is needed to enable a consistent and accurate wall-thickness determination under all conditions. To reduce the effects of container variability (wall thickness, wall material type, container shape, contour, and curvature), these measurements were conducted using the same container type (polyethylene terephthalate - PETE 120 ml containers) with the exception of nitric acid, which required a poly-coated glass bottle for containment. Key measurement parameters such as container diameter, container-wall temperature, acoustic velocity, and relative attenuation values were recorded for each of these samples. A series of multiple trials were conducted using a set of 20 identical PETE containers, and de-ionized, degassed water was used as the baseline liquid medium for measurement of acoustic properties. Tests were also conducted with different operators performing measurements using the device on a subset of the initial water measurements to quantify operator variability. Finally, a set of 10 measurements were conducted on each liquid in the 180 liquid test-set.

After physically measuring the acoustic properties with the CSD, density measurements were manually obtained for every product being analyzed. The density of the liquid can be obtained using ultrasonic energy from the outside wall of the container, but the CSD is currently not configured to make this measurement. Using the acoustic reverberations in the wall of the container, a logarithmic amplitude plot can be created and used to determine the acoustic impedance from the slope. An important feature of this method is a patented self-calibrating technique [14]. If, for example, the voltage to the transducer decreases by 1%, each echo changes by the *same* amount, but the slope on a logarithmic plot is *not* changed. Thus, this method does not rely on maintaining a specified voltage over a long period of time. As discussed previously, multiple reflections within the container wall can be obtained using the pulse compression technique as observed in all data acquired with the CSD,

and these signals can be analyzed to determine the acoustic impedance using the same method. From the acquisition of acoustic impedance and the measurement of acoustic velocity, the density of the liquid can be accurately and repeatably obtained.

To evaluate the effectiveness of the density parameter, a density data set was fabricated using manually measured density values. Ten values were randomly generated using a realistic standard deviation between that obtained for measured values of acoustic velocity and relative attenuation, for each liquid. The density of each liquid was measured gravimetrically using a calibrated Toledo™, four decimal, mass balance. After taking a vial and placing it inside the balance and setting it to zero, a syringe was used to extract 1 mL of the product and dispense it inside the zeroed vial. The weight of the 1 mL of liquid was recorded in grams (g). This volumetric method provided a density value in units of g/mL. Multiple measurements were made for each liquid to ensure statistical validity.

4. DATA

Table 2 is an example compilation of the data collected and recorded from the CSD along with the generated density metric for a single liquid. The measurement columns ‘Temperature’ and ‘Distance’ are two parameters that are measured non-ultrasonically via the CSD. These measurements represent the temperature of the container wall (under the assumption that the liquid is in thermal equilibrium with the wall) and the path distance that the ultrasonic waves travel through the liquid and container walls. The ‘Velocity’ and ‘Attenuation’ columns are the output result from the CSD when the first two parameters are merged into the ultrasonic data collected by the CSD. Finally, the ‘Density’ column is a generated list of values based on the average of the physical density measurement and generated randomly with a standard deviation between the standard deviation values calculated from actual measured values of velocity and attenuation.

Table 2 – Example of Data Collected

Test No.	Bottle ID	Liquid	Temperature (deg F)	Distance (in)	Velocity (m/s)	Attenuation	Density (g/mL)	
1	PETE	Hair Product	70.9	1.90	978.8	1.316	0.950	
2	PETE	Hair Product	70.9	1.90	979.0	1.395	0.952	
3	PETE	Hair Product	70.5	1.90	978.6	0.894	0.946	
4	PETE	Hair Product	70.7	1.90	978.9	1.120	0.949	
5	PETE	Hair Product	70.7	1.90	978.0	1.259	0.946	
6	PETE	Hair Product	70.9	1.91	978.8	1.280	0.956	
7	PETE	Hair Product	70.9	1.90	977.7	1.275	0.946	
8	PETE	Hair Product	70.7	1.90	978.8	1.689	0.955	
9	PETE	Hair Product	70.0	1.90	978.5	1.001	0.946	
10	PETE	Hair Product	70.0	1.90	978.9	1.189	0.952	
			Ave:	70.6	1.90	978.6	1.250	*
			Std Dev:	0.321	0.0012	0.4228	0.215	*

This study collected data from a number of different explosives in a variety of concentrated solutions. Figure 1 shows the results from a preliminary evaluation and correlation between the acoustically measured properties of velocity and attenuation and the percent weight concentration of HMX in solution. Similar trends were observed for the other explosives in solution. The density metric also exhibited trends when concentrations were increased.

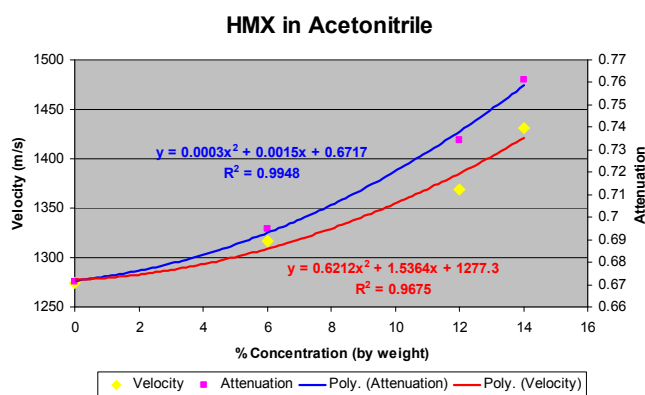


Figure 1 – Example of Acoustic Properties as a Function of Concentration

Table 4 – Variants of Confusion Matrices

Confusion Matrix	Velocity	Velocity and Attenuation	Velocity and Density	Velocity, Attenuation, and Density	Velocity, Density, and Attenuation
Total Number of Confusions	12	20	7	1	1

Table 4 illustrates that the addition of a third comparative acoustic signature allows the statistical analysis algorithm to more effectively classify and discriminate threat liquids from non-threat liquids. The ‘1’ in the last two columns of Table 4 show that for all data in the 180 liquid data set, only one scenario was presented where a confusion or incorrect classification was observed. This was the nitroglycerin (6%) and the CKBe cologne scenario.

7. CONCLUSIONS

From the work conducted to date at PNNL, tests have demonstrated the ability of the CSD technology to rapidly and effectively classify and discriminate several types of liquids (including threat liquids and precursor chemicals) in PETE containers with the necessary accuracy and reliability, and with the demonstrated ability to consistently “group” and separate many consumable/benign commercial products from LEs and precursor chemicals. The only liquid that was not considered a threat-liquid but was misclassified acoustically as a threat liquid was the CKBe cologne sample evaluated here. This liquid was misclassified 25% of the time (25 permutations out of 100 total permutations) with 6% nitroglycerin in acetonitrile solution. It should be noted that NG 6% concentration is generally too low to be considered a threat. Under the experimental constraints of this study, it was determined that this multi-metric signature approach was highly effective at correct and repeatable discrimination of threat and non-threat liquids.

As the set of measured liquids grows, the capability for acoustic classification and discrimination of liquids will be better quantified. Also, as the current signatures are more effectively resolved and as additional measurement properties (signatures) are obtained, such as absorption spectra, the ability to employ more advanced mathematical methods for discrimination will be studied. More work is needed to optimize the CSD’s algorithms to better address the effects of container variations on the acoustic property measurements.

Fluid characterization testing with the CSD also reinforced what the primary factors are that influence the accuracy and repeatability of attenuation measurements. Factors include transducer coupling to the container, beam divergence, container material, container geometry, distance measurement, temperature measurement, transducer alignment, and frequency bandwidth of the transducers. The most crucial component for accurate attenuation measurements is consistent coupling. To efficiently transmit ultrasonic energy into a container, adequate, repeatable and consistent coupling are required. While traditionally an

ultrasonic gel couplant is used to achieve coupling, tests at PNNL have demonstrated the viability of using a custom-designed dry couplant membrane that eliminates the use of gel or other coupling agents. Container wall material plays a significant role, in that different materials will absorb varying amounts of ultrasonic energy. In addition, container material and geometry variances lead to variations in reflection coefficients, thereby changing the amount of energy transmitted and reflected at each interface. Container walls can also be flexible, leading to incorrect distance measurements. This is overcome by the design of the CSD container fixture, which is specifically designed to accurately measure the distance between transducer faces in real time. Transducer alignment is critical for the accurate measure of attenuation. The transducer faces must be directly opposite and aligned parallel to each other for accurate transmission and reception of ultrasonic waves. The CSD fixture has been machined with strict tolerances to allow for precise transducer alignment, but future work will focus on alternate and more sophisticated approaches to the distance measurement process. As with velocity, distance and temperature measurements are critical for accurate attenuations measurements. Divergence is a physical phenomenon associated with ultrasonic wave propagation whereby the energy begins to “spread out” as a function of distance from the source, similar to a flashlight. The divergence error varies depending on the ultrasonic wave speed in the fluid and the area of the transducer face that is effectively coupled to the container wall. This divergence of the beam spreads the energy over a larger area, which decreases the energy along the straight-line path between the transducers. Because divergence is dependent upon the fluid velocity and container geometry, further refinement of the CSD’s analysis and processing algorithms is planned to compensate for these effects. Finally, transducer bandwidth plays a role in accurate attenuation measurements. With a larger frequency range over which to calculate the attenuation spectra, a more consistent estimate of the relative attenuation can be obtained.

With regard to acoustic velocity, more work is slated for addressing real-time wall thickness compensation from the detection and evaluation of wall echoes in the processed data. Also, consistent detection of the appropriate echoes in the received wave “packets” from the processed waveforms will be addressed in future work.

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