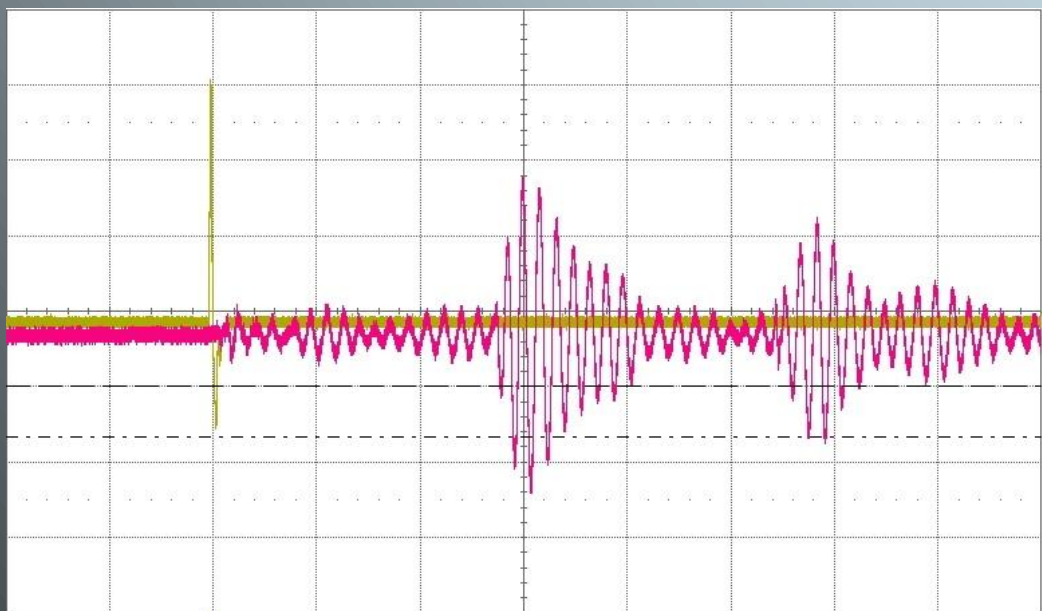




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# The Ultrasonic Densitometer Time Domain Response: Final Report

W. Rowell, Z. Dzbikowicz, G. Janssens-Maenhout, J. Howell



Institute for the Protection and the Security of the Citizen

2009

EUR 22740 EN

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JRC36916  
EUR 22740 EN  
ISBN 978-92-79-19318-7  
ISSN 1018-5593  
doi:10.2788/50704

Luxembourg: Office for Official Publications of the European Communities

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**EUROPEAN COMMISSION**  
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# **Ultrasonic densitometer for non-invasive in-field detection of illicit liquids in suspect containers**

## **FINAL REPORT**

**W. Rowell, Z. Dzbikowicz, G. Janssens-Maenhout, J. Howell**

**September 2009**



**Abstract**

Experiments were undertaken to investigate the feasibility of using propagating ultrasonic waves to find the speed of sound and density of solutions contained in opaque, sealed containers. A portable design is proposed which consists of 3 ultrasonic transducers aligned on a single plane along the surface of a tank. The content is then examined by measuring the time it takes for a signal to reflect off the back wall of the tank and return to another transducer. This time domain response approach delivered a very accurate analysis, with a low spread of results. This report demonstrates that by using this technique, very small changes in density can be observed. The final error in the density has been found to be less than 2%, which is adequate to reliably tell the difference between salt and fresh water.

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## 1. Project Synopsis

**Project title:** Ultrasonic densitometer for non-invasive in-field detection of illicit liquids in suspect containers.

**Project number:**

**Country:** Italy and the United Kingdom.

**Beneficiary:** Joint Research Centre for European Commission and the University of Glasgow.

**Project objective:** To produce a concept which will enable an easy, fast and accurate determination of an unknown tank's content, hence reducing the number of false alarms and non-detections of sensitive materials.

**Preface:** This project was undertaken as part of a final year industrial placement for a Masters in Mechanical Engineering degree. It was carried out in conjunction with the Mechanical Engineering Department at the University of Glasgow and supported by the Non-Proliferation and Nuclear Safeguards Unit at the Joint Research Centre (JRC) for European Commission, Ispra, Italy. The Non-Proliferation and Nuclear Safeguards Unit is part of the Institute for Protection and Security of the Citizen (IPSC). The project duration was from 1<sup>st</sup> February 2006 until 29<sup>th</sup> September 2006.

## **2. Introduction**

### 2.1 Background

Examining the content of a sealed container can be a sensitive and potentially dangerous problem. If its contents are toxic or the tank is someone else's property then there is a great advantage of using non-intrusive, non-destructive testing and by examining the contents from the outside. The following report highlights an investigation into a possible method of determining the contents of the tank. This is done by examining the data provided by pulse echo analysis of a propagating ultrasonic wave transmitted via externally mounted ultrasonic transducers. The proposed design will enable an easy, fast and accurate determination of an unknown tank's content, hence reducing the number of false alarms and non-detections of sensitive materials.

Using the medium's time domain response (TDR) or acoustic resonance spectrum (ARS) it is hoped to be able to measure the propagation time of a transmitted ultrasonic wave, from which the speed of sound and density can be calculated. Using these two physical properties it is conceived that the tank content can then be found. However, full classification of a liquid is much more difficult than it may first appear as two different liquids can share some of the same physical properties. Therefore the experimental challenge is to analyse a number of different factors raised by the characteristics of ultrasonic wave propagation, with the theoretical challenge being to provide accurate, insightful guidelines into the limiting factors affecting these techniques.

At the Non-Proliferation and Nuclear Safeguards Unit, Institute for the Protection and Security of the Citizen, Joint Research Centre, European Commission, research into the design of an ultrasonic densitometer has been active since 2002. This report advances the work of G. Janssens-Maenhout and L. Dechamp (2002) and S. Fowler (2005) and aims to provide a clearer understanding of issues affecting the ultrasonic densitometer. This report also offers an alternative view in the field of acoustic resonance spectroscopy than has been previously reported and attempts to draw more accurate conclusions from previous results.

### 2.2 Objectives

The specific aim of this project is to perform a study of the feasibility of an externally mounted ultrasonic densitometer using non-intrusive and reflected propagating waves on tanks of various dimensions. In the investigation performed by S. Fowler, the transducers were mounted on either side of the tank. This creates problems with proper alignment of the transducers as it is difficult to ensure that the transducers are at exactly the same height and angular position. Experimental analysis leads up to the creation of a guide rail, creating a much more accurate method of movement and helps overcome this problem. Furthermore when access is limited or perhaps only one side of the tank is available, S. Fowler's technique cannot be used. Therefore this report proposes a new method where the ultrasonic wave is reflected off the far wall back to receiving transducers. It is perceived that this concept will allow usage even when access is extremely limited.

The envisaged model must be able to operate on tanks with limited access, containing various different types of oils, milk, alcohols and sugar solutions. In particular the design must be robust enough to allow its application to the detection of sensitive materials used in the nuclear fuel cycle. The density measurement device must also be able to cope with tanks of unknown physical parameters. As the aim of this investigation has a specific emphasis on application to monitoring illicit trafficking in the nuclear industry, then a product must be developed which reflects these needs, enabling the tests to be undertaken "in-field" and not just within the laboratory.

Key areas that need to be investigated include:

- the **accuracy** of the device: the estimates of the liquid density must be sufficiently close to the real value,
- the **precision** of the device: the spread of readings from the device must be within a small range,
- the **adaptability** and **robustness** of the device: the device must cope with environments out with the laboratory,
- the **portability** and **usability** of the device: the device must be easily transportable, easy to use, work quickly and give a cost effective analysis.

### 2.3 Density Measurements Techniques

There are number of different methods available to measure the density of a material, which range from Archimedes' principle to the attenuation of gamma rays. All these various methods have advantages and disadvantages, which include; accuracy, sensitivity to environmental factors (such as temperature, pressure, vibration and corrosion), cost (due to installation, purchase and maintenance) and degree of exposure to the test substance.

There are a number of current non-invasive techniques available: Table 2.1 reviews a few of the various methods.

Medium	Range	Commercial Device
Radio waves	1 GHz – 1000 GHz	Radar probe
Light waves/ Coherent light	5 $\mu\text{m}$ – 10 $\mu\text{m}$ (FIR) 430 nm – 950 nm	Infrared camera Laser mapping / Lidar
Radioactive source	$^{137}\text{Cs}$ (661.64 keV)	$\Gamma$ - Tomography
Sound waves	20 kHz – 10 MHz	Sonic/Ultrasonic sensor

Table 2.1, Overview of current non-invasive density measurement techniques.

It is up to the designer to choose the best method of investigation to satisfy each individual specification. The device described herewith utilises ultrasonic wave propagation via the use of transducers, which analyse the reflected signal from the specimen. Ultrasonic devices are a good way of analysing density for this application as they are non-intrusive, insensitive to vibrations, relatively inexpensive, stay calibrated for long periods of time and are free from radioactive radiation.

### 2.4 Classifying a Liquid

Classifying a liquid is much more difficult than it may first appear as two different liquids can very easily share some of the same physical properties. If an accurate classification of a liquid is required then a combination of a number of physical properties is needed. Fortunately three certain physical parameters are enough to characterise a liquid to an acceptable degree of accuracy, these being liquid density, speed of sound and acoustic attenuation. Many liquids may have similar physical properties but the combination of these three unique properties allows accurate differentiation between comparable liquids to be made. Table 2.2 highlights the different speed of sound,  $c$ , and densities,  $\rho$ , for various liquids. However, for acoustic attenuation no definitive study has yet been performed to create an empirical database for all these liquids in atmospheric temperature ranges.

Liquid	Speed of sound, m.s <sup>-1</sup>	Density, kg.m <sup>-3</sup>
Acetone	1170	790
Liquid Argon (87 K)	840	1430
Methanol	1100	790
Gallium (30 K)	2870	610
Glycerine	1920	1260
Liquid He <sup>4</sup> (2 K)	228	145
Mercury	1450	13530
Liquid Nitrogen (77 K)	860	850
Silicone oil	1350	1100
Seawater	1530	1020
Water (20 °C)	1480	1000

Table 2.2, Acoustic properties of representative liquids.

### 2.5 Overview of Relevant Patents

Research into the technique of assessing the density of a sample with propagating ultrasonic waves has increased greatly over the past two decades. Since the publication of the first patent in July 1987, there have been an additional 28 applicable patents raised. Funding from the US Government to US facilities, such as Los Alamos National Laboratory, has ensured innovative advances in this highly specialised corner of research though the late twentieth and into the twenty-first century. An overview of the current Patents has been performed by S. Fowler (2005) in his report “*Operational proof of the Ultrasonic densitometer for non-invasive detection of toxic liquids in metal tanks*”.

Since the completion of S. Fowler’s report there have been further developments in the ultrasonic wave propagation sector of research. The following Patents represent innovative advances in this sector:

FY<sup>1</sup> 2005 Patent Recipients and License Income Recipients from Dr Dipen N. Sinha<sup>2</sup>, (MST-11<sup>3</sup>):

- Apparatus and Method for Comparing Corresponding Acoustic Resonances in Liquids.
- Apparatus and Method for Remote, Non-invasive Characterisation of Structures and Fluids inside Containers.
- Non-invasive Identification of Fluids by Swept-Frequency Acoustic Interferometry.
- Non-invasive Method for Determining the Liquid Level and Density Inside of a Container.

The technique described in this report does not overlap with any of the previous work performed by D. N. Sinha, nor is it covered by any of the described patents.

### 2.6 The Need for Inspections in the Nuclear Industry

Following past failures and recent developments, the nuclear industry continues to be under extremely heavy scrutiny. After accidents like those at Three Mile Island and Chernobyl the world is becoming increasingly sceptical about the nuclear industry. It has also been reported through trial testimony of known terrorists that Osama bin Ladens al Qaeda are seeking nuclear explosive materials (plutonium or highly enriched uranium) and the technical expertise for building atomic bombs, together with other dangerous nuclear materials for use in "dirty bombs" that spread radioactive contamination with conventional high explosives. It is therefore extremely important to restrict access to nuclear materials and produce safeguards to act as deterrents for any country considering supplying terrorists with any nuclear materials or undertaking a clandestine nuclear weapons program.

<sup>1</sup> Patent and Licensing Body code for, “For Year of”.

<sup>2</sup> Dr D. N. Sinha is the world leader in Acoustic Resonance Spectroscopy techniques and in general purpose non-invasive diagnostics tools. He has published 60 papers in these areas and has been awarded the “Distinguished Performance Award” by the Los Alamos National Laboratory.

<sup>3</sup> Personal reference number for Dr D. N. Sinha.

### 2.7 Nuclear Materials Management and International Safeguards

Nuclear materials are extremely hazardous and access to these materials should be restricted to avoid any danger to the public or the environment. There are three traditional methods of ensuring a nuclear non-proliferation regime; they are export controls, physical protection and safeguards.

Export controls are related to the direct trading of sensitive items across international borders. These items may be key equipment, dangerous nuclear or other associated materials required by the nuclear industry. Alternatively a major commodity sought after by countries furthering nuclear research is detailed technology and personal expertise as this can be more valuable than anything bought or manufactured.

Physical protection is aimed at prevention rather than detection and is linked to avoiding any theft of dangerous materials or sabotage by restricting access to controlled areas. Access control encapsulates a large and diverse spectrum of management. This can be as basic as the use of fencing, gates or secure entry procedures. Surveillance techniques are also used from the use of guards and dogs to the utilisation of CCTV and satellite tracking methods. Containment in a number of forms, such as the use of seals, relieves some pressure on surveillance as it allows areas to be sealed with certainty that no access can be granted without detection. Also the formation of task forces or response teams is used to investigate sabotage threats and produce risk assessments. Finally, the vetting and careful control of company employees attempts to remove any potential internal problems.

Safeguards, along with physical protection, act as a deterrent and try to ensure the non-proliferation of nuclear materials. All civil nuclear materials, in Non-Proliferation Treaty (NPT) signatory countries, are subject to safeguards control, these are: uranium in depleted, natural or enriched forms; all forms of plutonium, irrespective of type or composition and thorium. The objectives of safeguards are to detect:

- Inconsistencies in the accounting system
- Inconsistencies in the nuclear related building designs within 3 months
- Diversion of a goal<sup>4</sup> quantity of Plutonium, Uranium or Thorium
- Inconsistencies in the measurement systems
- Diversion of 1 container/item from a secure store

The minimum safeguards requirements state that any facility must be able to locate and account all items on an inventory to a high accuracy. They must be able to provide operating records for all items on stock from which material balance accounts can be constructed. All Special Nuclear Materials (SNM) must be able to be accounted for to a known certainty with these items being made available to be checked annually at the personal inventory verification. Furthermore sufficient access must be granted to permit inspectors to investigate that safeguard standards continue to be maintained on site.

### 2.8 Safeguard Agency Recommendations

There are two agencies that are responsible for nuclear safeguarding: The International Atomic Energy Authority (IAEA) and European Atomic Energy Community (EURATOM). Each provides slightly different assessments for the quantity needed and conversion time required for standard reactor grade nuclear material to be turned into weapons-grade fissile material. Most commercial pressurised water and boiling water reactors utilise the Uranium isotope U-235 at a level of enrichment of around 4%. Weapons-grade nuclear fissile material contains U-235 at an enrichment level of 90% and above and must therefore undergo a further enrichment process following the extraction from the nuclear fuel cycle. The

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<sup>4</sup>A goal quantity is the amount of specific nuclear material required to create a nuclear device, see Tables 1.3 and 1.4 for goal quantities.

“conversion” time it takes to do this change is critical as it presents the inspectors with an opportunity to detect that nuclear material has been removed so they can advise the IAEA accordingly.

### International Atomic Energy Authority (IAEA)

Nuclear Material	Low Enriched Uranium (LEU)	High Enriched Uranium (HEU)	Fresh Plutonium (Pu)	Plutonium Isotope (Pu-238)	Thorium (Th)	Uranium Isotope (U-233)
Goal quantity required	75 kg	25 kg	8 kg	<80%	20 ton	8 kg
Conversion time to Fissile material	1 year	1 month	1 month	3 months	1 year	1 month
Probability	For false alarm < 5%					

Table 2.3, IAEA statistics on quantity of material, time required and probability of detection.

### European Atomic Energy Community (EURATOM)

Nuclear Material	Low Enriched Uranium (LEU)	High Enriched Uranium (HEU)	Fresh Plutonium (Pu)	Irradiated Plutonium	Thorium (Th)	Thorium Isotope (Th-233)
Goal quantity required	75 kg	25 kg	8 kg	1 F.A.	20 ton	8 kg
Conversion time to Fissile material	1 year	4 weeks	4 weeks	3 months	1 year	4 weeks
Probability	For false alarm < 5% and for non-detection < 10%					

Table 2.4, EURATOM statistics on quantity of material, time required and probability of detection.

## 2.9 Other Sectors of Application

Density measurement of a medium utilising the proposed ultrasonic densitometer has a number of advantages not only for nuclear safeguards, but also for other activities where on-site, non-invasive inspection is beneficial. Other fields to benefit from the design include:

### 2.9.1 National Security

- Aiding the detection of prohibited chemical weapons within sealed tanks and artillery shells.
- Counter-drug, customs and drug verification analysis.

### 2.9.2 Industrial

- Usage in the petrochemical industry to perform level measurements and fractional distillation guarantees.
- Process control and characterisation of chemicals and pharmaceuticals.



2.9.3 Quality Control of Foods and Drinks

- Determining whether any EU stocks of edible oils stored on farms have been defrauded by the addition of water to the storage silos.
- Determining whether olive oil has undergone the special extraction process involved in the creation of extra virgin olive oil or whether a marketed extra virgin olive oil is actually normal olive oil.
- Determining the alcohol content and quality of beverages sold by the drinks industry, to make sure they are correctly represented on the labels of the products.
- Ensuring that abnormal milk in the farming industry does not enter the raw milk supply to be provided for public consumption.

2.9.4 Environmental Sensors

- Aiding the monitoring of water and air quality.

### 3. Time Domain Response (TDR)

#### 3.1 Principles of Operation

One method of identifying and estimating the density of a medium is by analysing the speed of sound by means of time domain response. The basic principle involves sending a very short pulsed signal (see Figure 3.1) of appropriate ultrasonic frequency through a medium and analysing the time it takes for the echo to return. The liquid/solid interfaces at the nearside wall of the tank allows sound to propagate through whereas the interface at the far wall creates a reflective surface which causes the ultrasonic wave to bounce back to the transducer. The data is then analysed on an oscilloscope (see Figure 3.2) and the time taken for the signal to return is measured.

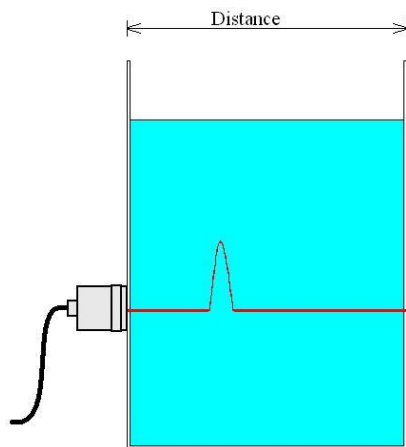


Figure 3.1, Pulsed wave travelling through tank.

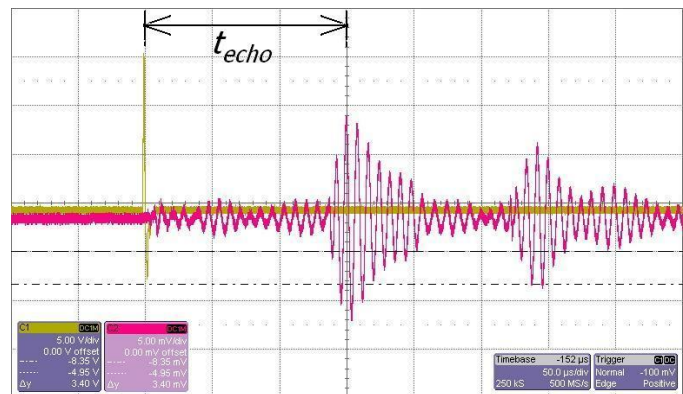


Figure 3.2, Transmitted and received signal response.

Once the time,  $t_{echo}$ , had been obtained a simple calculation was done to work out the speed of sound,  $C_{speedofsound}$ , of the medium:

$$C_{speedofsound} = \frac{2 \times \text{distance}}{t_{echo}}$$

The calculated speed of sound was then cross referenced against a database (see Table 3.1), from which the bulk modulus<sup>5</sup>,  $\beta$ , was found.

Solution	Acoustic Velocity (m.s <sup>-1</sup> )	Bulk Modulus, $\beta$ , (Pa, N.m <sup>-2</sup> ) $\times 10^9$
Carbon Tetrachloride	No data	1.31
Ethyl Alcohol	No data	1.06
Gasoline	No data	1.3
Glycerine	1920	4.52
Mercury	1450	2.85
SAE 30 Oil	1350	1.5
Seawater	1530	2.35
Water	1480	2.15

Table 3.1, Bulk modulus for a selection of common liquids.

<sup>5</sup> The bulk modulus, measured in N.m<sup>-2</sup>, is the inverse of the compressibility of a liquid. It measures the response in pressure due to a change in relative volume, essentially measuring the substance's resistance to uniform compression.

The density,  $\rho$ , of the medium was then calculated with the speed of sound calculated and the data found for the bulk modulus via the following relationship:

$$\rho = \frac{c_{\text{speed of sound}}^2}{\beta}$$

The hypothesis can then be accepted if this density agrees with what is expected.

### 3.2 Applying the Principle to the Specification

Unfortunately the simple application of this technique would not work without the inspector physically measuring the tank with a tape measure, wasting valuable time and incurring another measurement inaccuracy. Furthermore, one of the main specifications in this investigation was to create a system which did not require any prior knowledge of the dimensions of the tank to perform an analysis. Therefore a different method was proposed.

### 3.3 The Proposed Concept

To overcome this it was proposed to add more transducers and so form a different geometric relationship from the reflected signals. Whereas in the first example only one transducer was needed, here the design had to deal with using a total of three transducers. All the transducers were aligned in a single plane to allow the usage of the device even when only one side of the tank is accessible.

In this set up, one of the transducers acts as the transmitter and the other two as receivers (see Figure 3.3). The first receiving transducer is moved as close as possible to the transmitting transducer and acts as a reference. The other receiving transducer is moved as far away as possible from the transmitting transducer, but within any physical constraints obstructing access to the tank and the maximum transducer separation<sup>6</sup> of the transducer. The propagation times acquired via the externally mounted transducers is then drawn through an algorithm to provide the speed of sound and density.

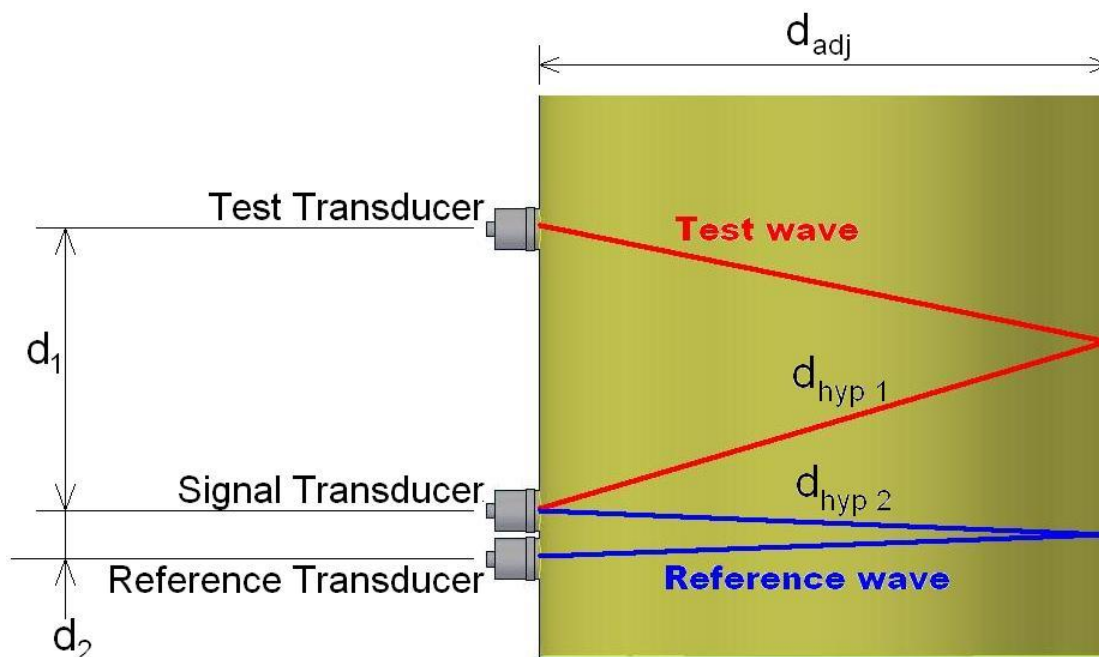


Figure 3.3, TDR experimental set up showing distance variables.

<sup>6</sup> The maximum transducer separation is defined in the next chapter, titled Divergence.

The time taken for the test,  $t_1$ , and reference,  $t_2$ , waves to propagate was obtained via the oscilloscope:

$$t_1 = \frac{2 \times d_{hyp1}}{c} \qquad t_2 = \frac{2 \times d_{hyp2}}{c}$$

applying Pythagoras's theorem then creates the following relationships,

$$t_1 = \sqrt{\frac{d_1^2}{c^2} + \frac{d_{adj}^2}{c^2}} \qquad t_2 = \sqrt{\frac{d_2^2}{c^2} + \frac{d_{adj}^2}{c^2}}$$

rearranging,

$$t_1^2 = \frac{d_1^2 + d_{adj}^2}{c^2} \qquad t_2^2 = \frac{d_2^2 + d_{adj}^2}{c^2}$$

and,

$$\frac{d_{adj}^2}{c^2} = t_1^2 - \frac{d_1^2}{c^2} \qquad \frac{d_{adj}^2}{c^2} = t_2^2 - \frac{d_2^2}{c^2}$$

combining the equations,

$$t_1^2 - \frac{d_1^2}{c^2} = t_2^2 - \frac{d_2^2}{c^2}$$

$$0 = t_2^2 - t_1^2 - \frac{d_2^2}{c^2} + \frac{d_1^2}{c^2}$$

$$c^2 = \frac{d_1^2 - d_2^2}{t_1^2 - t_2^2}$$

which when rearranged becomes:

$$c = \sqrt{\frac{d_1^2 - d_2^2}{t_1^2 - t_2^2}}$$

Once the speed of sound of the medium has been derived then the density,  $\rho$ , can be calculated using the following relationship:

$$\rho = \frac{\beta}{c^2}$$

Where bulk modulus,  $\beta$ , is once again found by cross referencing the calculated speed of sound against data supplied.

### 3.4 Propagation Time Capture Procedure

The aim was to determine the time it takes for a signal to propagate from the signal transducer, through the tank to the receiving transducers. Therefore an understanding about the steps that the system performs must have been achieved.

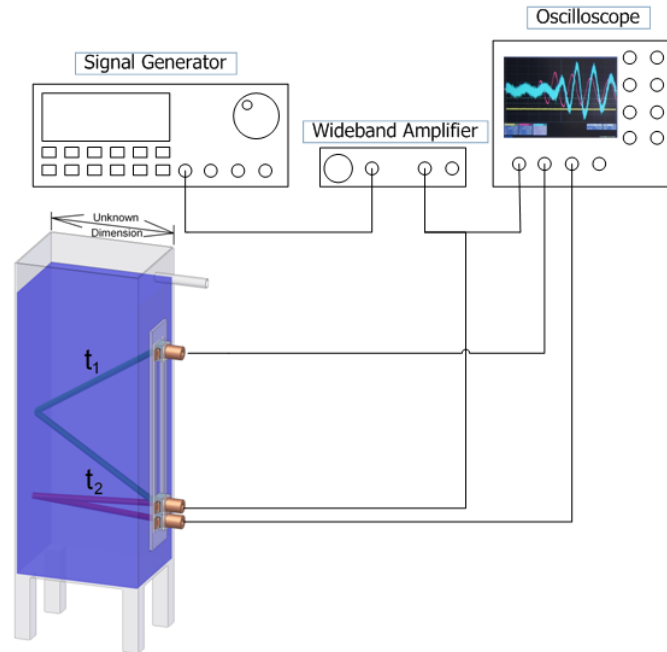


Figure 3.4, TDR experimental set up for capturing time for wave propagation.

1. Signal of appropriate frequency created and transmitted by the signal generator.
2. Signal amplified to an appropriate level via the wideband amplifier.
3. Electrical signal transformed into mechanical movement via the piezoelectric elements within the transducer.
4. Ultrasonic wave propagated through the tank by the signal transducer.
5. Reflected wave picked up by the receiving transducers.
6. Transformed back to electrical waves in receiving transducers and the signal sent to the oscilloscope.
7. The oscilloscope displays a visual image of the wave.

Fine adjustments of the oscilloscope's controls were then required to manoeuvre the peaks and gain an accurate propagation time.

### 3.5 Limitations of the Application

There are associated limitations with this type of experimental procedure. It has been hypothesised that the best results will be received when the test transducer is as far away as possible from the signal transducer. To receive an adequate understanding of the limitations involved with moving the transducer a long distance apart, we must consider wave divergence.

## 4. Divergence

### 4.1 Limiting Factor

When a wave propagates through a medium it tends to spread from the focus of the beam. The magnitude of this spread is referred to as divergence and is an important concept when selecting the type of transducer for the investigation.

An example is that of a radio in a car which still picks up radio when the car antenna is not in direct line of sight of the emitting antenna. This occurs because the electromagnetic radio waves manage to bend around obstructions and allow a signal to be received. However, when the same car enters a tunnel, though the signal may be held for a period of time, it will eventually be lost after a certain distance as the radio waves cannot bend any further. Therefore it can be seen that waves have a finite divergence property.

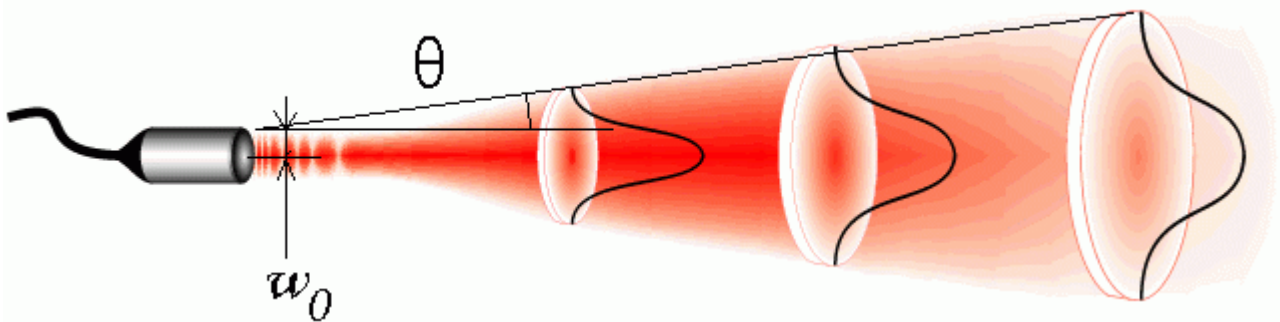


Figure 4.1, Transducer divergence.

Pressure waves exhibit the same divergence properties as electromagnetic waves but to different extents depending on a number of signal parameters. As ultrasonic waves only travel at the speed of sound, unlike electromagnetic waves which travel at the speed of light, then a large divergence is experienced. Furthermore, it is known that waves with a long wavelength and hence short frequency, diverge more than that of shorter wavelengths and higher frequencies.

The first step in measuring the magnitude of the divergence comes from calculating the wavelength,  $\lambda$ , of the signal by obtaining the speed of sound,  $c$ , from empirical data and the frequency,  $\nu$ , from the signal generator, utilising the following relationship:

$$\lambda = \frac{c}{\nu}$$

Once the wavelength of the signal has been found, the divergence,  $\theta$ , can be obtained via the following relationship, where  $\omega_0$  represents the beam waist, or in the context of this analysis, the active transducer diameter:

$$\theta = \frac{\lambda}{\pi\omega_0}$$

Whilst interesting on its own, the divergence becomes more important when analysing its relevance to limiting factors of the Ultrasonic densitometer. To cut out as much noise (Rayleigh waves<sup>7</sup>) as possible from propagating out of the signal transducer, through the wall of the tank, to the receiving transducer, the receiving transducer must be positioned as far away as the divergence will allow from the signal transducer. This maximum permissible distance,  $d_{max}$ , is a property of the tank as well as the wave:

<sup>7</sup> Rayleigh waves, also known as Rayleigh-Lamb waves, are waves which travel along incident to the surface of a system.

$$d_{\max} = 2d_{\text{adj}} \tan \theta$$

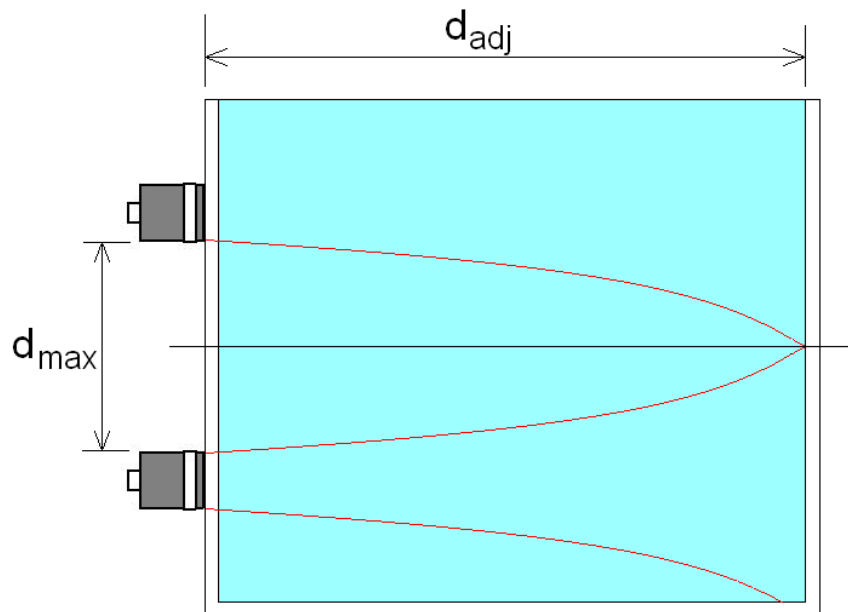


Figure 4.2, Maximum permissible transducer separation diagram.

For the various transducers available the maximum transducer separation,  $d_{\max}$ , was calculated.

Transducer	1	2	3	4
Frequency range	500 kHz	1 MHz	2.25 MHz	0.86 – 2.1 MHz
Active Diameter, mm	25	19	19	20
Wavelength, m	$2.996 \times 10^{-3}$	$1.498 \times 10^{-3}$	$0.6658 \times 10^{-3}$	$(0.713 - 1.742) \times 10^{-3}$
Divergence	$4.37^0$	$2.88^0$	$1.28^0$	$1.30^0 - 3.18^0$
Maximum transducer separation, m	$0.153 \times d_{\text{adj}}$	$0.100 \times d_{\text{adj}}$	$0.045 \times d_{\text{adj}}$	$0.045 \times d_{\text{adj}} - 0.111 \times d_{\text{adj}}$

Table 4.1, Theoretical divergence of available transducers.

## 5. Wedge Configuration

### 5.1 Combating Divergence

In response to the problem of divergence affecting the design of the anticipated ultrasonic densitometer, a wedge configuration was tested. The wedge holds the transducers at an angle and therefore enables the signal and test transducers to be placed further apart. This means that the wave will travel further through the test liquid, increasing the propagation time and hence reducing the error on this reading.

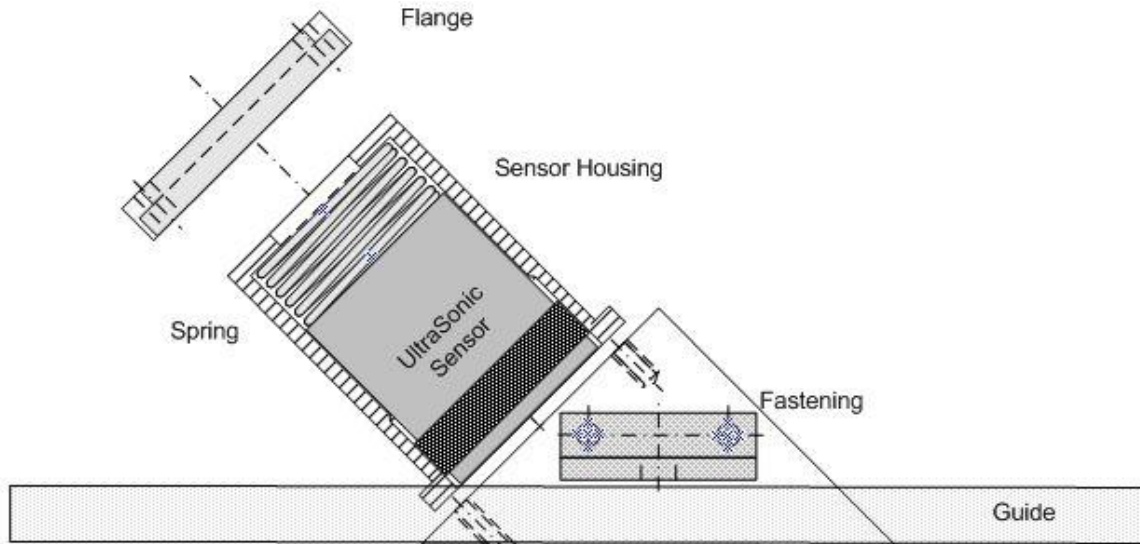


Figure 5.1, Schematic arrangement of ultrasonic transducer and wedge.

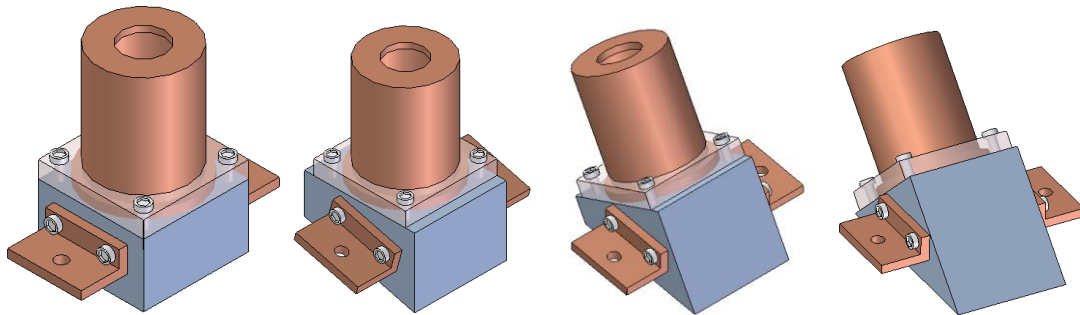


Figure 5.2, various angles used in the analysis, [0°, 15°, 30° and 45°].

Utilising the proposed wedges will allow a greater separation between the transducers to occur. The maximum permissible distance,  $d_{max}$ , can now be calculated, again considering the divergence,  $\theta$ , and the angle at which the transducer is held,  $\phi$ :

$$d_{max} = 2d_{adj} \tan(\theta + \phi)$$



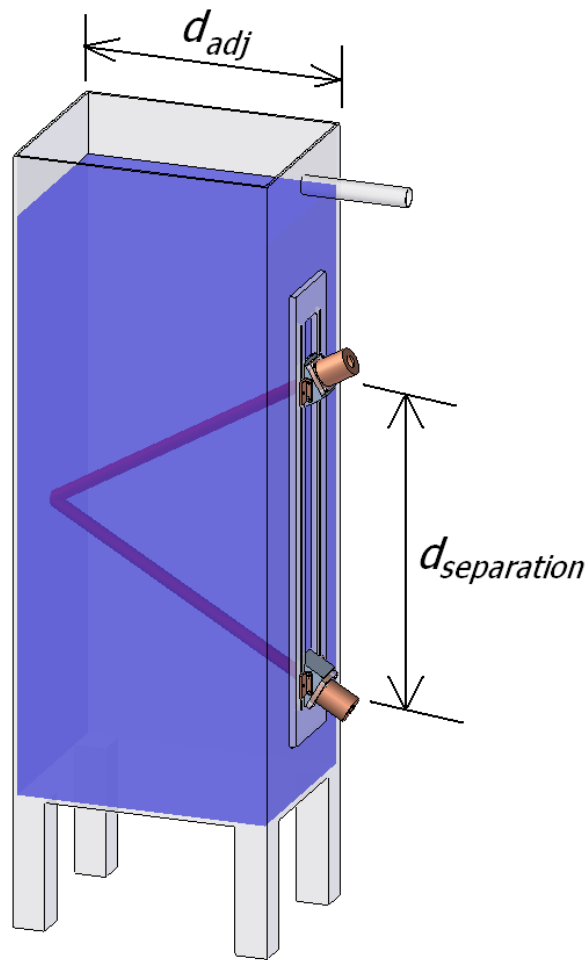


Figure 5.3, Tank arrangement highlighting the new separation of the transducers.

Wedge Angle	0°	15°	30°	45°
Maximum transducer separation without wedge, m	0.153× $d_{adj}$	0.153× $d_{adj}$	0.153× $d_{adj}$	0.153× $d_{adj}$
Maximum transducer separation with wedge, m	0.153× $d_{adj}$	0.703× $d_{adj}$	1.368× $d_{adj}$	2.331× $d_{adj}$
Minimum transducer separation with wedge, m	0.05 <sup>8</sup>	0.375× $d_{adj}$	0.960× $d_{adj}$	1.716× $d_{adj}$
Optimal transducer separation, m	0.153× $d_{adj}$	0.536× $d_{adj}$	1.155× $d_{adj}$	2× $d_{adj}$

Table 5.1, Transducer separations for provided 500 kHz transducers

To gain a further insight into the possible problems associated with this new geometric arrangement we must now consider the effects of refraction.

<sup>8</sup> Due to dimensions of wedge.

5.2 Refraction

When a propagating ultrasonic wave passes from one medium into another with a different refractive index than the first and at an oblique angle, refraction occurs. Refraction takes place at the interface between the two medium due to the dissimilar velocities of the acoustic waves within the two materials. The waves, produced by the transducer, travel faster in a material with a greater acoustic velocity and as the angle of incidence is oblique then one part of the pressure wave will reach the interface first, either slowly or hastening this part of the signal and therefore altering its trajectory. The angular change is quantified by Snell's Law, which equates the ratio of each materials acoustic velocity,  $v_1$  and  $v_2$ , to the ratio of the sines of incident angle,  $\theta_1$ , and the refraction angle,  $\theta_2$ :

$$\frac{\sin \theta_1}{v_1} = \frac{\sin \theta_2}{v_2}$$

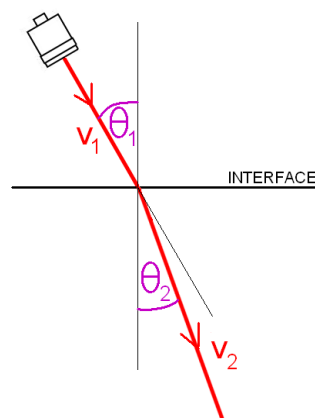


Figure 5.4, Example of refraction.

The effect of refraction causes a further complication to the design of the ultrasonic densitometer. With the proposed design, shown in Figure 5.3, refraction occurs at 4 interfaces:

- Between the Aluminium block and the Steel tank wall,
- Between the Steel tank wall and the fluid,
- Between the fluid and the Steel tank wall,
- Between the Steel tank wall and the Aluminium block.

This increased number of refraction interfaces causes the wave to travel further through the steel tank wall and less through the fluid under test. This will add an extra error into the analysis of density, speed of sound and attenuation and if the tank was made from a different material the error would alter again. Therefore, careful analysis into the effect of refraction must be undertaken to reduce this error as far as possible to achieve an accurate result. However this is not the only factor which can affect the accuracy of the results. To gain a fuller understanding of the error incurred, the environment which surrounds the tank must be considered.

## 6. Environmental Influences

The physical properties of a system are not always constant and can change with certain atmospheric fluctuations. As the proposed design utilises an analytical examination to measure the density of a system, then an understanding must be obtained into the reasons why these changes take place.

### 6.1 Temperature

Physically, temperature is a measure related to the average kinetic energy of the particles within a substance. The kinetic energy possessed by a system is related to the mass and velocity of the particles of that system and has a large effect on the speed of sound, density and a number of other properties of a medium. It is therefore important to understand how temperature affects a system as not including this aspect into the design of a densitometer would cause large inaccuracies. Clearly exhibited in Figure 6.1 it can be seen that even small changes in ambient temperature can dramatically affect the speed of sound of water.

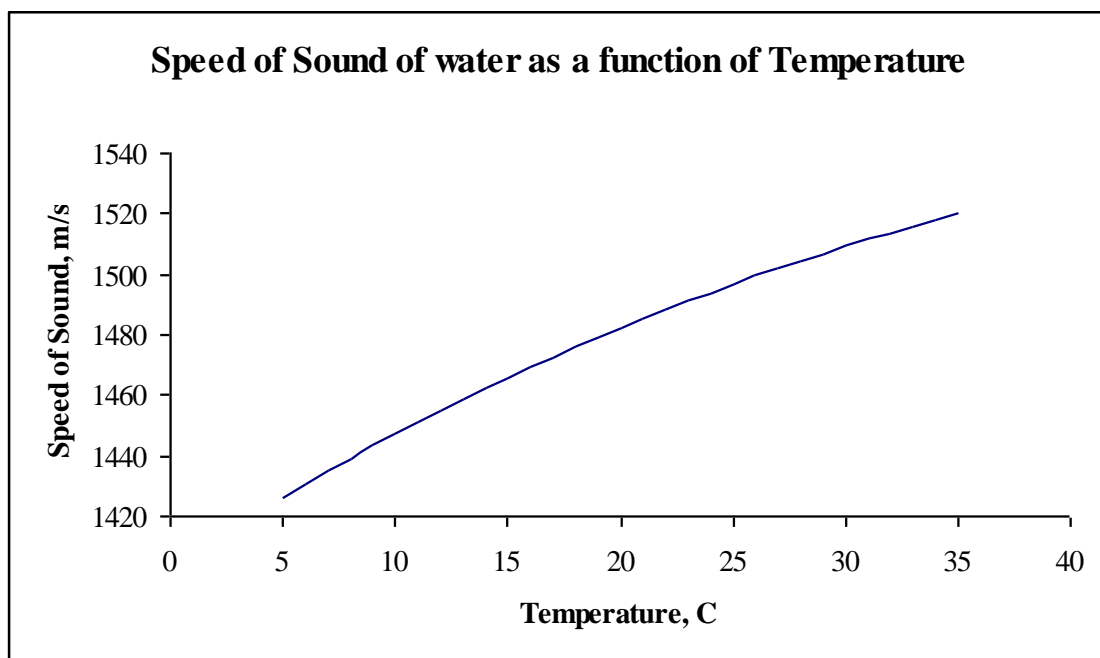


Figure 6.1, Speed of sound of water as a function of temperature.

Numerous models have been produced to try and obtain the most accurate reading for the effect of temperature. The values obtained from these models vary slightly over the same temperature range so the most accurate model would include a combination of them all. The following theoretical analysis aided the creation of an algorithm, which was used to determine the accuracy of the experiments. During analysis, a digital thermometer was inserted into the tank and the exact temperature was found. From this the theoretical speed of sound of the water could be found and cross referenced across the experimental result, highlighting the degree of accuracy.

#### 6.1.1 Bilaniuk and Wong

Bilaniuk and Wong (1993, 1996) converted Del Grosso's and Mader's 1972 data to the 1990 International Temperature Scale and then produced three sets of coefficients depending on the number of temperature points which were converted and taken into account in their data fitting routines. Validity range: 0-100°C at atmospheric pressure.

a) 112 point equation

$$c = 1402.38742 + 5.03821344T - 5.80539349 \times 10^{-2}T^2 + 3.32000870 \times 10^{-4}T^3 - 1.44537900 \times 10^{-6}T^4 + 2.99402365 \times 10^{-9}T^5$$

b) 36 point equation

$$c = 1402.38677 + 5.03798765T - 5.80980033 \times 10^{-2}T^2 + 3.34296650 \times 10^{-4}T^3 - 1.47936902 \times 10^{-6}T^4 + 3.14893508 \times 10^{-9}T^5$$

c) 148 point equation

$$c = 1402.38744 + 5.0383617T - 5.81172916 \times 10^{-2}T^2 + 3.34638117 \times 10^{-4}T^3 - 1.48259672 \times 10^{-6}T^4 + 3.16585020 \times 10^{-9}T^5$$

### 6.1.2 Marczak

Marczak (1997) combined three sets of experimental measurements, Del Grosso and Mader (1972), Kroebel and Mahrt (1976) and Fujii and Masui (1993), and produced a fifth order polynomial based on the 1990 International Temperature Scale. Validity range: 0-95 °C at atmospheric pressure.

$$c = 1402.385 + 5.038813T - 5.799136 \times 10^{-2}T^2 + 3.287156 \times 10^{-4}T^3 - 1.398845 \times 10^{-6}T^4 + 2.787860 \times 10^{-9}T^5$$

### 6.1.3 Lubbers and Graaff

Lubbers and Graaff (1998) produced simple equations with a restricted temperature range for medical ultrasound applications, including tissue mimicking materials and test objects. Within the quoted temperature ranges they claim that the maximum error is approximately 0.18 ms<sup>-1</sup> in comparisons with experimental data and more detailed equations such as Bilaniuk and Wong (1993, 1996).

a) A simple equation for use in the temperature interval 5-35°C

$$c = 1404.3 + 4.7T - 0.04T^2$$

b) A simple equation for use in the temperature interval 5-40°C

$$c = 1405.03 + 4.624T - 3.83 \times 10^{-2}T^2$$

As all equations yield different values for the speed of sound an average value combining all of them has been used for my calculations. Using this analysis the calculated speed of sound in pure water at 20°C is:

1482.340131 ms <sup>-1</sup>
------------------------------

## 6.2 Pressure

Sound waves propagate through a medium as waves of alternating pressure, using local regions of compression and rarefaction<sup>9</sup>. Sound propagates more easily through areas of high pressure as the molecules are closer together and hence transmission of these pressure vibrations becomes more efficient.

As the ambient pressure of air rises, from weather patterns or other effects, so the pressure on the test system increases, this in turn increases the pressure within the medium. As this internal pressure within the medium increases the sound waves find it easier to propagate and so the speed of sound and density of

<sup>9</sup> Rarefaction is the reduction of a medium's density, or the opposite of compression.

the medium increase. Fortunately speed of sound and density changes with atmospheric pressure changes are small and for the means of this examination can be neglected.

## 7. Error Analysis

Prior to experimental procedure an error analysis was carried out to locate and understand the sources of error. Reducing the error in this technique is paramount to its success and the only way to ensure usage of this is to make sure the error is as small as possible.

### 7.1 Assumptions

Firstly, the parameters of the test must be set by analysing the experimental assumptions, listed in decreasing criticality:

- The selection of a reflection peak on the Oscilloscope is always accurate,
- The wave propagates exactly as expected across the tank,
- The wall thickness of the tank is constant and uniform,
- The tank walls are perfectly parallel
- There is no build up of rust or other contaminant on the inner or outer walls of the tank,
- The grease lubricant does not effect the analysis,
- The effect of medium disturbance is negligible,
- The transducers are all attached to the tank by the same pressure,
- The accuracy of the oscilloscope is infinitely precise.

### 7.2 Equipment Inaccuracies

The errors within the equipment used must also be considered, these are as follows:

- Steel ruler  $\pm$  0.5 mm [at 20<sup>0</sup>C]
- Accurate slide ruler  $\pm$  0.05 mm [at 20<sup>0</sup>C]

### 7.3 Error Quantification

Adapted from <http://www.lhup.edu/~dismanek/scenario/errorman/calculus.htm>

The error,  $E$ , of a result of a squared function,  $R=R(x^2)$ , can be described as follows:

$$R = x^2$$

Hence;

$$\frac{\Delta R}{\Delta x} = 2x$$

Therefore:

$$E = \frac{\Delta R}{R} = 2 \frac{\Delta x}{x}$$

Applying this concept to determine total error,  $E_{total}$ , in the speed of sound calculations, the overall error can be quantified as:

$$E_{total} = \frac{\sqrt{\left( \left( \frac{d_1^2 - d_2^2}{d_1 + d_2} \right)^2 + 2\varepsilon_d \left( \frac{d_1 + d_2}{d_1 + d_2} \right) \right)}}{\sqrt{\left( \left( \frac{t_1^2 - t_2^2}{t_1 + t_2} \right)^2 + 2\varepsilon_t \left( \frac{t_1 + t_2}{t_1 + t_2} \right) \right)}}$$

Where  $\varepsilon_d$  and  $\varepsilon_t$  represent the error in the reading in the transducer separation and propagation time measurements respectively.

Estimating the error on the probe separation to be 0.2mm and the error on the oscilloscope reading to be 1  $\mu$ s, the expected error in the speed of sound for various tank diameters is displayed as follows:

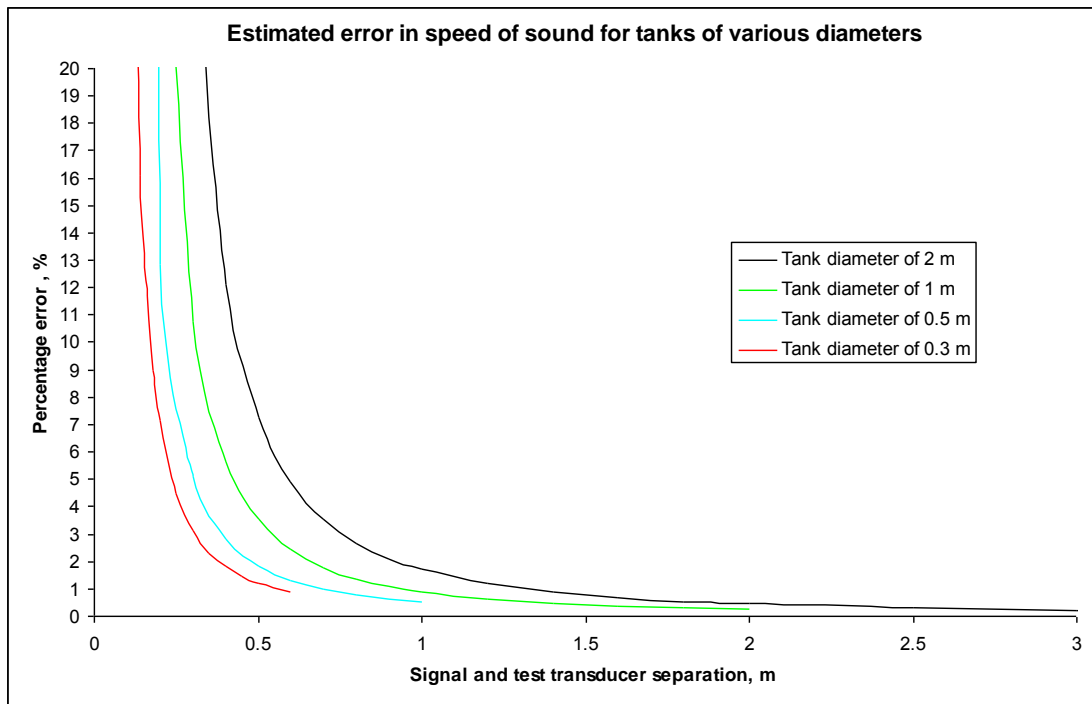


Figure 7.1 Error analyses of various tank diameters.

This shows that all propagation time readings produced by the proposed ultrasonic densitometer are subject to a large error when the transducer separation is low and especially on tanks with large diameters. The following experiments quantify this error to a sufficient level such that confident characterisation of the tanks content can be achieved.

## **8. Results**



## 8.1. Transducer analysis

### Aim

- To perform an assessment of the various responses of the transducers provided and to determine which were best to perform the following analysis.
- To and validate the time domain response (TDR) techniques.

### Experimental Setup

- The transducers were set up as in Figure 8.1.1.
- The two transducers were arranged at the same height and the propagation time and signal quality analysed.
- The frequency of the signal from the signal generator was changed according to the centre frequency of the transducer.
- The temperature of the water was constantly monitored via a digital thermometer.
- The tank used was tank number 1<sup>10</sup> depicted in Figure 8.1.2.

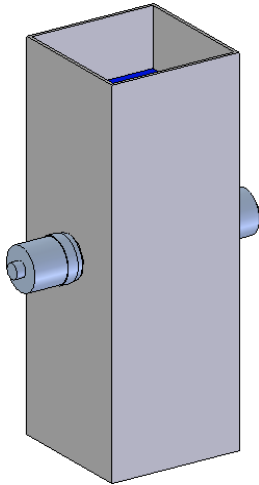


Figure 8.1.1, Experimental setup of transducers and tank.

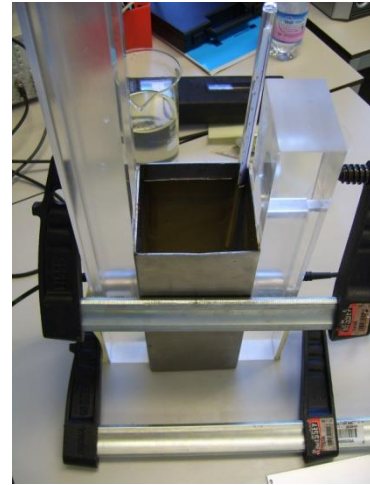


Figure 8.1.2, Actual experimental setup.

### Instrument Setup

- Wave Function = Truncated Sine wave
- Amplitude = 20 V
- Phase = 270<sup>0</sup>
- Burst Count = 1
- Frequency = 500 kHz, 860 kHz, 1 MHz, 2.1 MHz, 2.25MHz
- Period = 310 ms
- Amplifier rating = Maximum permissible with output clip, raising output amplitude to 30 V

<sup>10</sup> See Appendix C for tank dimensions.

**Transducers undergoing test**Transducer 1

- Brand = Imasonic, IM-0.5-25-P (Immersion probe)
- Centre Frequency = 500 kHz
- Active diameter = 25 mm
- Housing length = 38.9 mm
- Housing diameter = 35 mm

Transducer 2

- Brand = Imasonic, IM-1.0-19-P (Immersion probe)
- Centre Frequency = 1 MHz
- Active diameter = 19 mm
- Housing length = 32 mm
- Housing diameter = 27 mm

Transducer 3

- Brand = Imasonic, IM-2.25-19-P (Immersion probe)
- Centre Frequency = 2.25 MHz
- Active diameter = 19 mm
- Housing length = 32 mm
- Housing diameter = 27 mm

Transducer 4

- Brand = Imasonic, IM-0.5-25-P (Immersion probe)
- Centre Frequency = 0.86 & 2.1 MHz
- Active diameter = 20 mm
- Housing length = 49 mm
- Housing diameter = 24 mm

Temperature of water = 11.6<sup>0</sup>C

**Theoretical Propagation time**

$$t_{propagation} = \left( \frac{d_{waterbody}}{c_{H_2O}} \right) + \left( \frac{2 \times d_{wallthickness}}{c_{steel}} \right) = \left( \frac{0.10455}{1453.50272} \right) + \left( \frac{2 \times 0.00225}{4507.04} \right)$$

$$t_{propagation} = 0.00007293 \text{ sec}$$

**Results**

Transducer	1	2	3	4 (at 0.86 MHz)	4 (at 2.1 MHz)
Time for signal to propagate to the receiving transducer, $\mu$ s	73.24	73.62	73.75	73.38	73.81
Percentage error, %	0.425	0.946	1.124	0.617	1.207

Table 8.1.2, TDR propagation times and percentage errors in readings.

**Discussion**

It can be seen from Table 8.1.2 that transducer 1 (500 kHz) provides the most accurate reading for the propagation time of the wave. This however is not the most critical aspect as calibration can remove any inaccuracies caused by time delay in the equipment. The size of the housing for these transducers was larger than for the other transducers and this allowed them to be held more precisely than the others. Furthermore, the signal produced the least noise compared to other transducers. Therefore all further analysis proceeded using only the 500 kHz transducers. Finally, it has been shown theoretically in Appendix B.4 that a propagating wave decays at a much faster rate with increasing frequency, so therefore a low frequency is required to try to maintain as much of the original signal as possible.

## 8.2. Speed of sound verification

### Aim

- To provide some background into measurement and movement techniques.
- To aid selection of the correct oscilloscope peak and to help understand the wave interactions within the test specimen.
- To prove that as the distance between the transducers increases, so does the propagation time.
- To validate provided speed of sound data.
- To test effect of using a reflected signal.

### Experimental Setup

- Two transducers were mounted on the outside wall of the tank.
- The signal transducer omitted a pulsed signal and the receiving transducer received the signal, with the time taken for this process to occur being recorded.
- The receiving transducer was moved further away from the signal transducer along the tank wall.
- The temperature of the water was constantly monitored via a digital thermometer.
- The tank used was tank number 1, depicted in Figure 8.1.1.

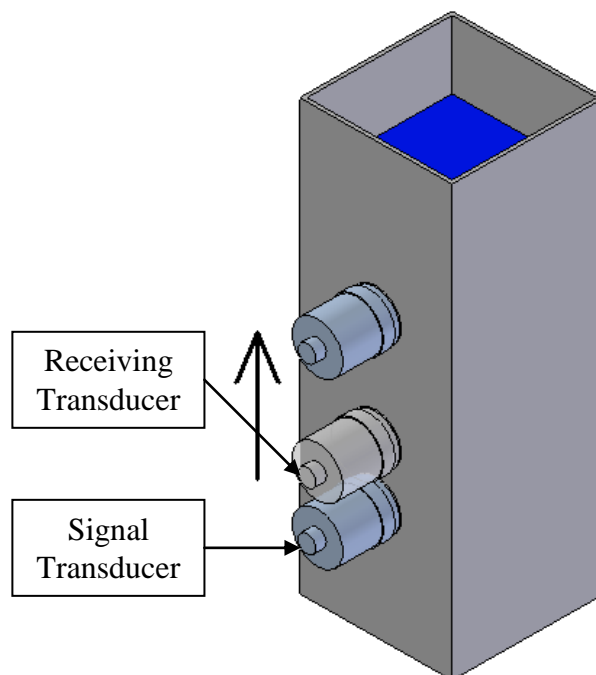


Figure 8.2.1, Setup of experiment depicting the movement of the transducers.

### Instrument Setup

- Wave Function = Truncated Sine wave
- Amplitude = 20 V
- Phase =  $270^{\circ}$
- Burst Count = 1
- Frequency = 500 kHz
- Period = 310 ms
- Amplifier rating = Maximum permissible without incurring an output clip.  
Raising output amplitude to 30 V

**Transducer undergoing test**Transducer 1

- Brand = Imasonic, IM-0.5-25-P (Immersion probe)
- Centre Frequency = 500 kHz
- Active diameter = 25 mm
- Housing length = 38.9 mm
- Housing diameter = 35 mm

Temperature of water = 12.5<sup>0</sup>C

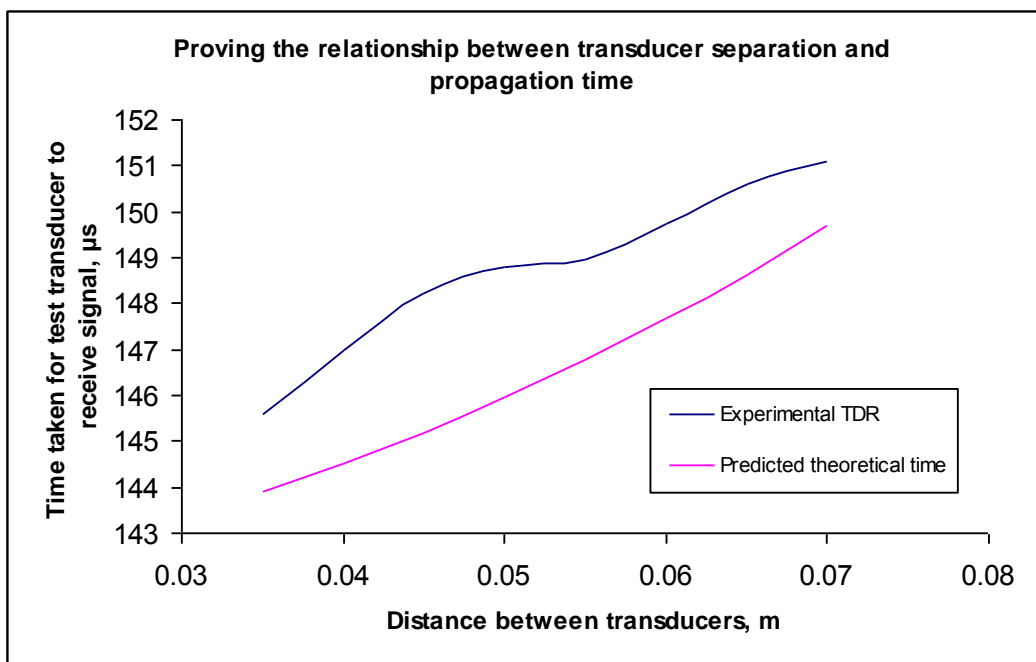
**Results**

Figure 8.2.2, Proving the relationship between transducer separation and propagation time.

Using the time taken for the wave to propagate from the signal to the test transducer via the time domain response technique, the speed of sound was then calculated from the following relationship:

$$c_{\text{speed of sound}} = \frac{2 \times \sqrt{(d_{\text{liquid body}} + d_{\text{tank wall thickness}})^2 + (d_{\text{transducer separation}})^2}}{t_{\text{propagation}}}$$

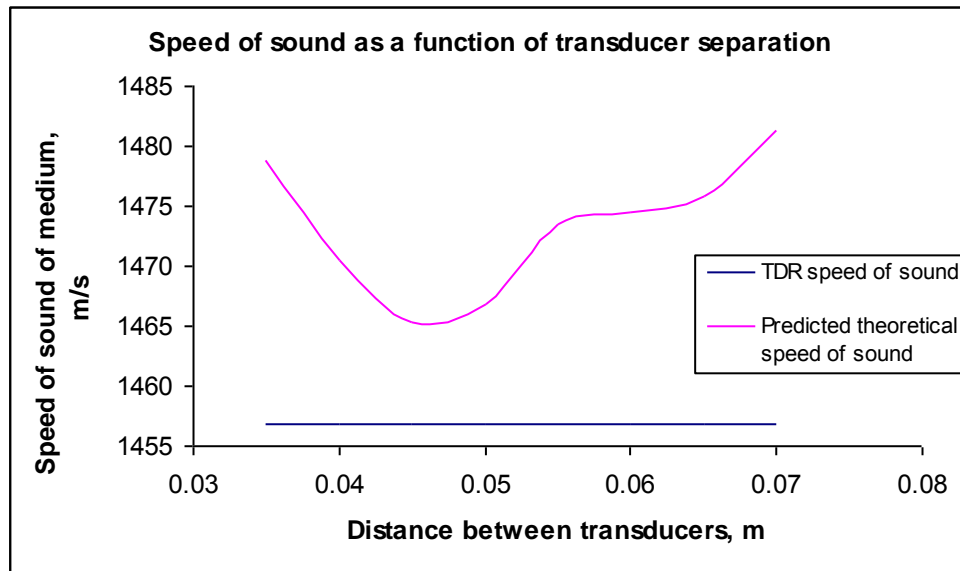


Figure 8.2.3, Speed of sound as a function of transducer separation.

## Discussion

It can be seen from Figure 8.2.2 that the propagation time for an ultrasonic wave is a function of transducer separation. Therefore, as transducer separation increases the time taken for the test transducer to receive the signal from the signal transducer increases. The shape of the graph matched the expected results well but not exactly, this shows that error has an effect on the propagation time.

Movement of the transducers proved more difficult than was expected. Keeping the tension on the G-clamps so they didn't fall off and moving the transducers at the same time was a delicate operation. Unfortunately this problem had further implications than just being an annoying inconvenience. To make sure the same peak on the oscilloscope was selected each time, visual contact had to be maintained with the oscilloscope screen whilst undertaking this process. Regularly the tension would be lost, the clamp would fall and test would have to be stopped and restarted. Also when moving the transducers the wave form on the oscilloscope would change, two peaks would merge into one and the original would be lost. Therefore extreme care and patience had to be shown when moving the transducers.

The selection of the correct oscilloscope peak is paramount to the success of the proposed ultrasonic densitometer. In a perfect situation the returned signal would be infinitely precise with no noise and contain only one returned signal peak. Unfortunately the real scenario is much different. When a wave reflects back from the interface between the liquid under test and the tank wall, complex interactions take place. The reflected signal interacts with echoes cause by the tank wall vibrating and by other propagating waves, beyond the desired longitudinal waves. This therefore makes it very hard for the correct signal peak to be picked out on the oscilloscope.

From Figure 8.2.3 it can be seen that theoretically the speed of sound does not change as a function of transducer distance. Although, the experimental data seems to imply that there may be some sort of relationship combining these two. This phenomenon can be explained and proved by an analysis of the error.

The Figure allows us to visualise the error in the signal, which is around  $1\%$  or  $20\text{ms}^{-1}$ . When considering the usage of the metal ruler with an accuracy of only  $0.5\text{mm}$ , the slide rule with an accuracy of  $0.005\text{mm}$  and the error in the oscilloscope, then the total error in the speed of sound for this experiment is:

$$E_{total} = \frac{2 \times \sqrt{\left( \left( \frac{0.00005}{0.1023} + \frac{0.00005}{0.00225} \right)^2 + \left( \frac{0.0005}{0.035} \right)^2 \right)}{\left( \frac{0.000001}{0.00000548} \right)^2} = 22.97$$

This is within the maximum error of  $22\text{ms}^{-1}$  and therefore any observed relationship in between speed of sound and transducer separation is down to systematic error only.

### 8.3. Geometric relationship proof

#### Aim

- To test the geometrical proof derived previously.
- To see what affect the use of the ultrasonic pulse delay time evaluation unit has on the accuracy of the results.
- To see if this greater level of automation is beneficial.

#### Experimental Setup

- The set up shown in Figure 8.3.1 was used.
- The receiving transducer was moved further away from the signal transducer along the tank wall, with the reference transducer staying stationary and as close to the signal transducer as possible.
- The temperature of the water was constantly monitored via a digital thermometer.
- The tank used was tank number 2, depicted in Figure 8.3.2.

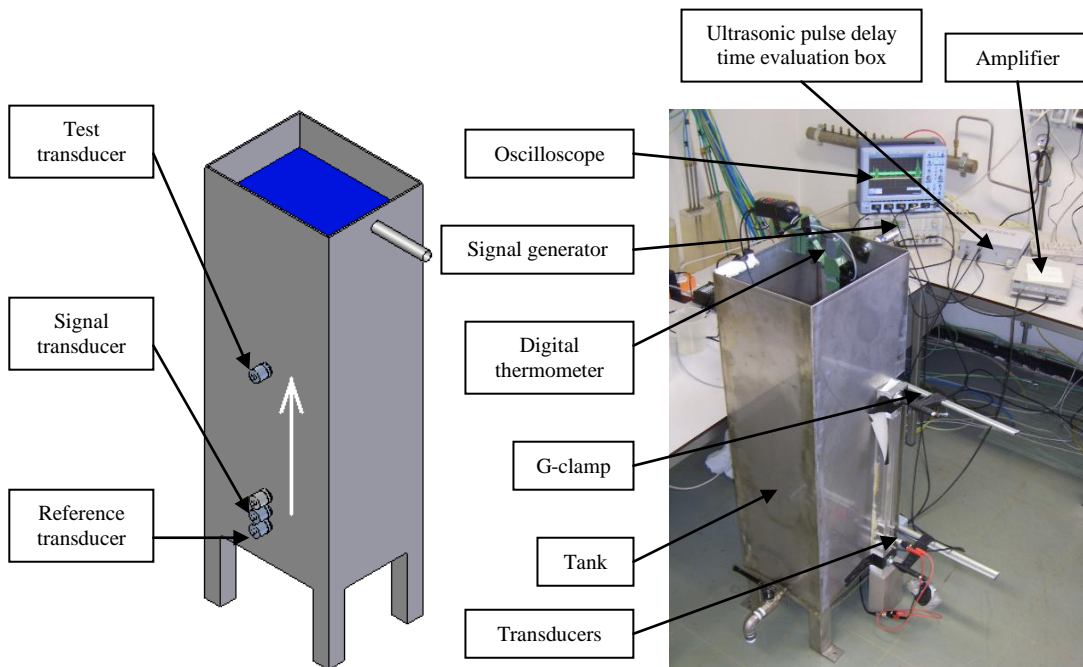


Figure 8.3.1, Movement of transducer.

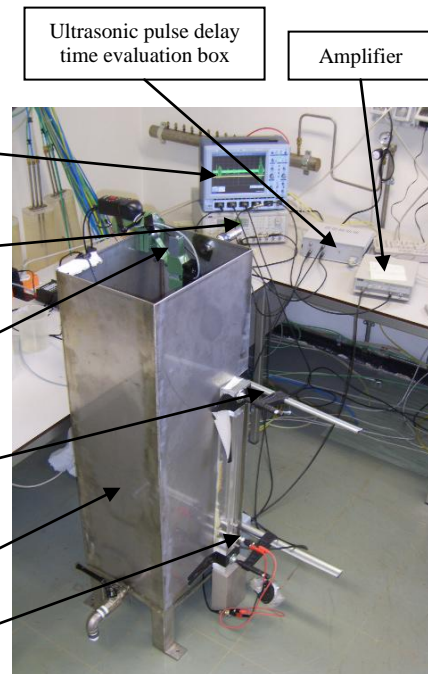


Figure 8.3.2, Actual experimental setup.

#### Instrument Setup

- Wave Function = Truncated Sine wave
- Amplitude = 20 V
- Phase =  $270^{\circ}$
- Burst Count = 1
- Frequency = 500 kHz
- Period = 310 ms
- Amplifier rating = Maximum permissible without incurring an output clip.  
Raising output amplitude to 30 V



**Transducer undergoing test**

Transducer 1

- Brand = Imasonic, IM-0.5-25-P (Immersion probe)
- Centre Frequency = 500 kHz
- Active diameter = 25 mm
- Housing length = 38.9 mm
- Housing diameter = 35 mm

Temperature of water = 20.3°C

**Results**

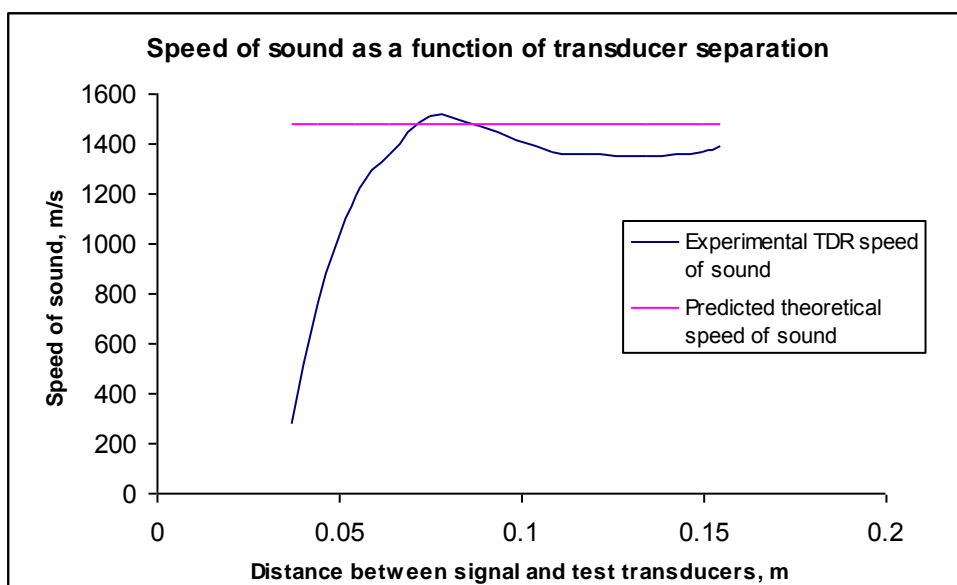


Figure 8.3.3, Speed of sound as a function of transducer separation.

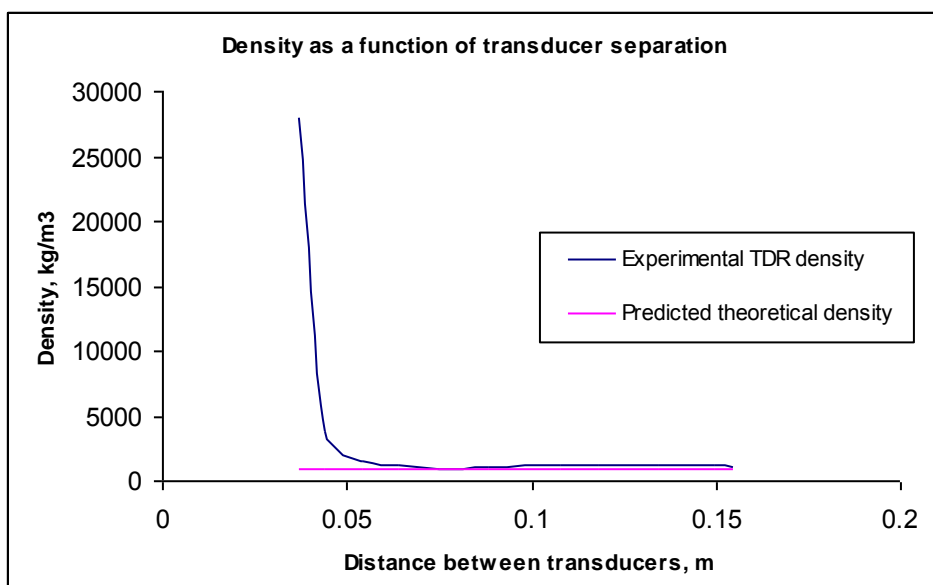


Figure 8.3.4, Density as a function of transducer separation.

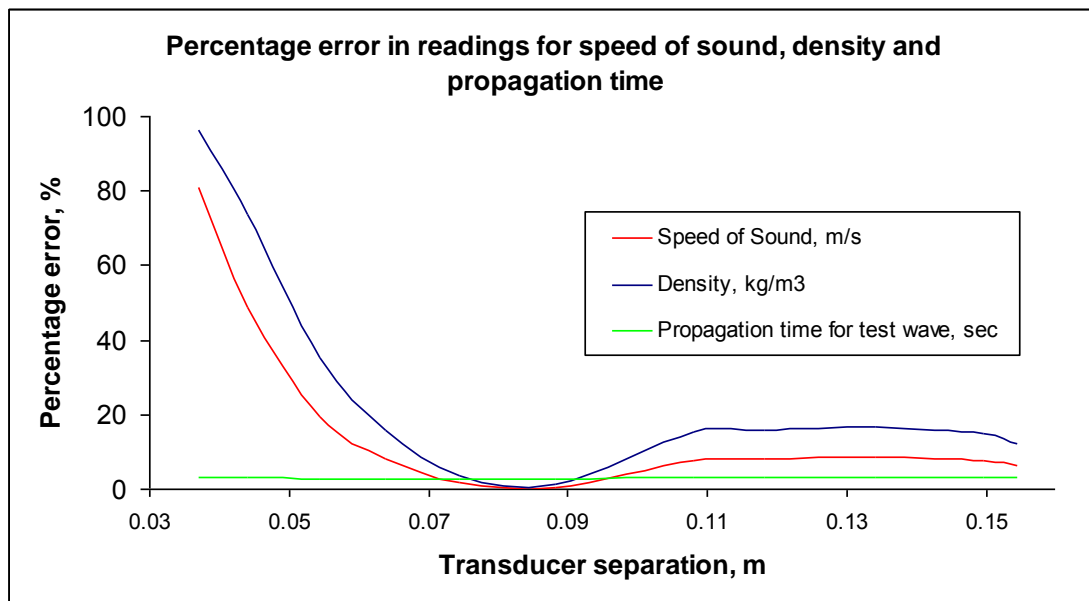
**Error**

Figure 8.3.5. Percentage error in readings for speed of sound, density and propagation times in both receiving transducers.

**Discussion**

Again it can be seen from Figure 8.3.5, that the error decreases exponentially as the distance between the transducers increases linearly. This is in stark contrast to the error in the propagation times, which is constant at around 3%. This suggests that the main factor controlling the accuracy of the density and speed of sound is the transducer separation.

The ultrasonic pulse delay time evaluation unit added yet another complicated electrical device into the circuit. This is good for automation as the final goal would benefit from a high degree of automation. However, as the box was not set with a delay, it often gave out readings for the initial echo from the wave propagating within the tank wall. The test ran over a 100 second period with the box taking one recording every second and the raw data was displayed in an Excel file. This had to be painstakingly scrutinised to remove any rogue results affecting the average value and therefore any benefit provided by the further degree of automation was cancelled out. If this process was not performed the accuracy of the experiment would have suffered considerably.

## 8.4. Curved surface attachment

### Aim

- To examine what effect a tank with a much greater diameter would have on the error.
- To investigate the problems associated with operating the proposed system on a curved surface.

### Experimental Setup

- The set up shown in Figure 8.4.1 was used.
- The receiving transducer was moved further away from the signal transducer along the tank wall, with the reference transducer staying stationary and as close to the signal transducer as possible.
- The temperature of the water was constantly monitored via a digital thermometer.
- Transducers fixed to tank with straps rather than clamps.
- The tank used was tank number 3, depicted in Figure 8.4.2.

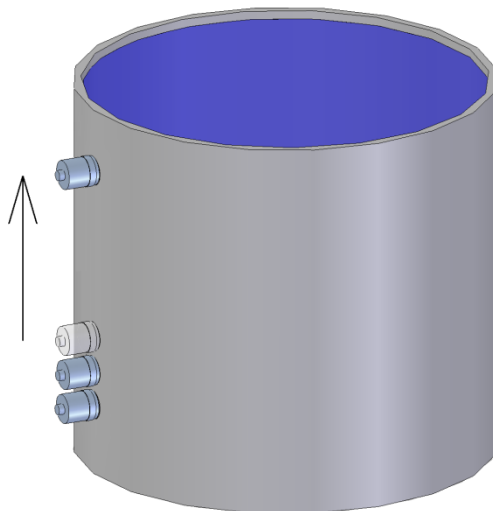


Figure 8.4.1, Movement of transducer.



Figure 8.4.2, Actual experimental set up.

### Instrument Setup

- Wave Function = Truncated Sine wave
- Amplitude = 20 V
- Phase =  $270^0$
- Burst Count = 1
- Frequency = 500 kHz
- Period = 310 ms
- Amplifier rating = Maximum permissible with output clip, raising output amplitude to 30 V

### Transducer undergoing test

#### Transducer 1

- Brand = Imasonic, IM-0.5-25-P (Immersion probe)
- Centre Frequency = 500 kHz
- Active diameter = 25 mm

- Housing length = 38.9 mm
- Housing diameter = 35 mm

Temperature of water = 17.4<sup>0</sup>C

## Results

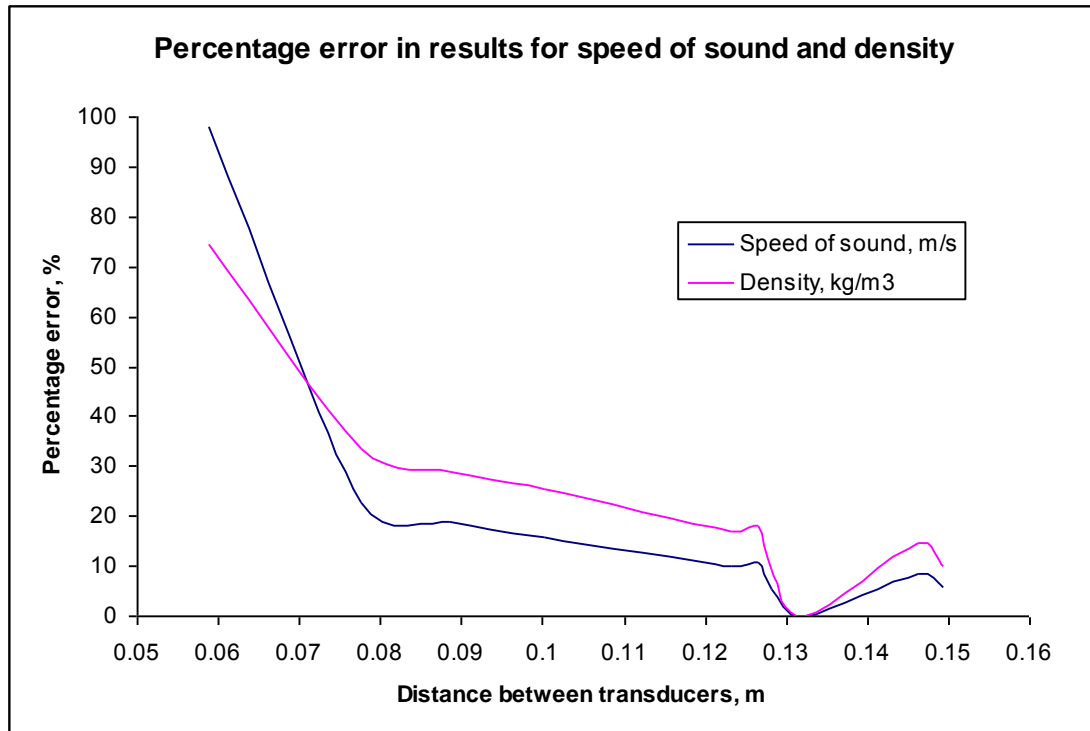


Figure 8.4.3, Percentage error in readings for speed of sound and density.

## Discussion

The main advantage of using a tank with a large diameter is that divergence becomes less of an issue enabling the test transducer to be moved further away from the signal transducer. Increasing the transducer separation increases the accuracy of the test and therefore this technique presents a more accurate result than the previous examples. A final accuracy of 10.9% is achieved on the density reading. This would have decreased further, however beyond a transducer separation of 0.15m the slide ruler could no longer measure and the investigation was halted.

The increased accuracy in the density and speed of sound just after the transducer separation reaches 0.13m is due to the peak examined at the start of the test interacting with another wave and disappearing forcing an estimation of its location to be undertaken.

As the radius of curvature was relatively large, the problems of attaching the flat faced transducers to the tank wall were minimised and this did not affect the results. Furthermore, the usage of straps to attach the transducers to the outside of the tank wall actually provided some unexpected benefits. As the tension was kept constant no slackening and retightening processes needed to be undertaken. This meant that when the transducer was being moved, the oscilloscope screen could easily be watched and the correct peak selected.

## 8.5. Signal transducer movement

### Aim

- To see what effect moving the signal transducer has on the accuracy of the results.

### Experimental Setup

- The set up shown in Figure 8.5.1 was used.
- The receiving and reference transducers stayed stationary whilst the signal transducer was moved progressively closer to it along the tank wall.
- The temperature of the water was constantly monitored via a digital thermometer.
- The tank used was tank number 3, depicted in Figure 8.5.2.

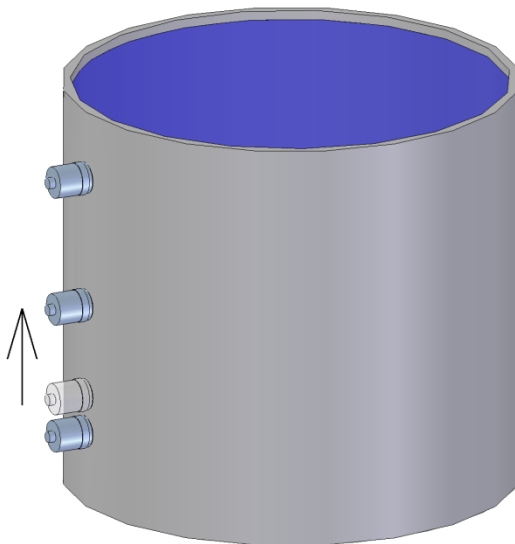


Figure 8.5.1, Movement of transducer.



Figure 8.5.2, Actual experimental setup.

### Instrument Setup

- Wave Function = Truncated Sine wave
- Amplitude = 20 V
- Phase =  $270^{\circ}$
- Burst Count = 1
- Frequency = 500 kHz
- Period = 310 ms
- Amplifier rating = Maximum permissible with output clip, raising output amplitude to 30 V

### Transducer undergoing test

#### Transducer 1

- Brand = Imasonic, IM-0.5-25-P (Immersion probe)
- Centre Frequency = 500 kHz
- Active diameter = 25 mm
- Housing length = 38.9 mm

- Housing diameter = 35 mm

Temperature of water = 17.3<sup>0</sup>C

**Results**

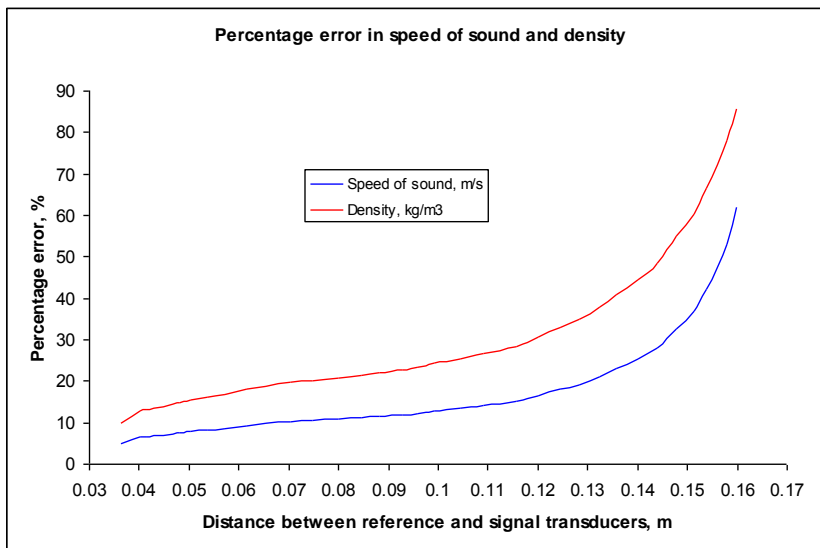


Figure 85.3, Percentage error in speed of sound and density.

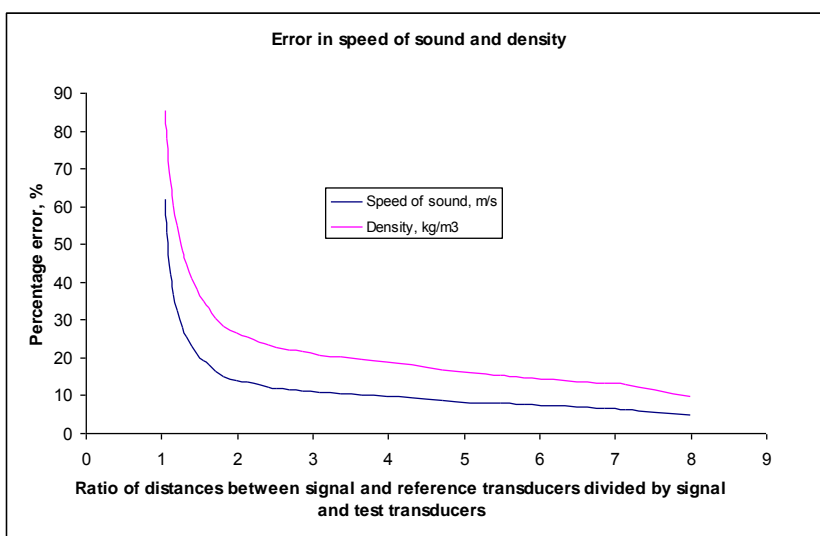


Figure 85.4, Error in speed of sound and density.

**Discussion**

From Figure 85.3 it can be seen that the most accurate results are obtained when the signal transducer is as close as possible to the reference transducer. From Figure 8.5.4 it can be seen that as the ratio of the distances between the signal and reference transducers divided by the signal and test transducers increases, so the accuracy of the analysis increases. Therefore the most accurate results are obtained when the reference transducer is as close to the signal transducer as possible and the test transducer is as far away as possible from the signal transducer.

## 8.6. Repeatability analysis

### Aim

- To investigate the repeatability of the time domain response test.

### Experimental Setup

- The set up shown in Figure 8.6.1 was used.
- The receiving transducer was moved further away from the signal transducer along the tank wall, with the reference transducer staying stationary and as close to the signal transducer as possible.
- The temperature of the water was constantly monitored via a digital thermometer.
- The tank used was tank number 3, depicted in Figure 8.6.2.

### Instrument Setup

- Wave Function = Truncated Sine wave
- Amplitude = 20 V
- Phase =  $270^{\circ}$
- Burst Count = 1
- Frequency = 500 kHz
- Period = 310 ms
- Amplifier rating = Maximum permissible with output clip, raising output amplitude to 30 V

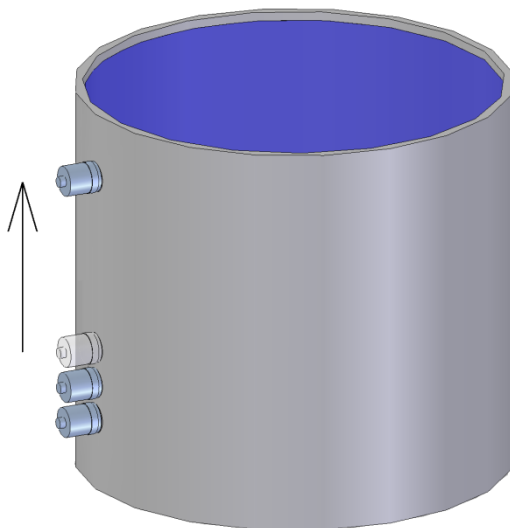


Figure 8.6.1, Movement of transducer.



Figure 8.6.2, Large storage tank.

**Transducer undergoing test**Transducer 1

- Brand = Imasonic, IM-0.5-25-P (Immersion probe)
- Centre Frequency = 500 kHz
- Active diameter = 25 mm
- Housing length = 38.9 mm
- Housing diameter = 35 mm

Temperature of water = 18.4<sup>0</sup>C

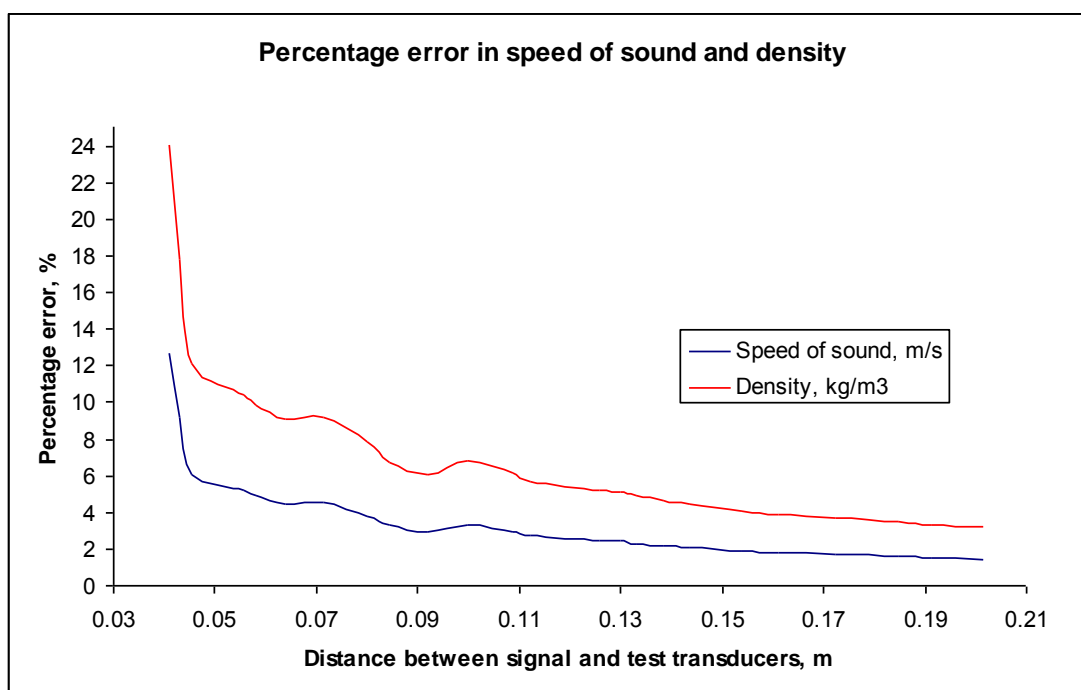
**Results**

Figure 8.6.3, Percentage error in speed of sound and density.

**Discussion**

These results show that the test can be repeated and similar readings can be produced. However the result of this investigation is an increased accuracy, with the percentage error in the density being within 3.5% and speed of sound within 1.5%. Although this is a good result it does emphasise the frailties of the experiment. It highlights what difference can be made from selecting an incorrect peak on the oscilloscope.



## 8.7. The wedge design

### Aim

- To investigate what affect the wedge design has on the errors incurred in the experiment.
- To see what effect the various angles have on signal clarity and accuracy.

### Experimental Setup

- The set up shown in Figure 8.7.1 was used.
- The receiving transducer was moved further away from the signal transducer along the tank wall within the guide at small increments.
- The various angles available were utilised.
- Calibration within the aluminium wedges was performed prior to the experiment to remove the delay caused by these from affecting the speed of sound and density analysis.
- The temperature of the water was constantly monitored via a digital thermometer.
- The tank used was tank number 2, depicted in Figure 8.7.2.

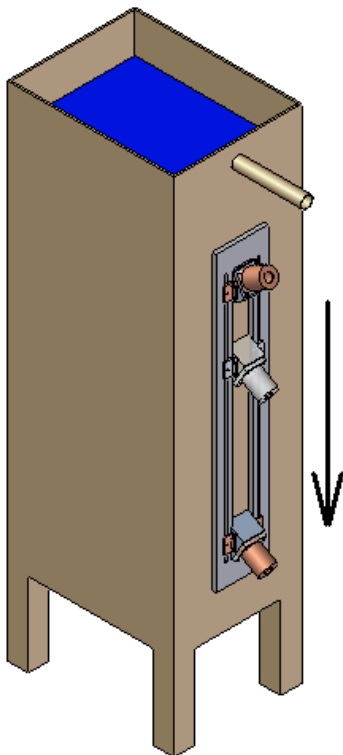


Figure 8.7.1. Experimental setup depicting transducer movement.

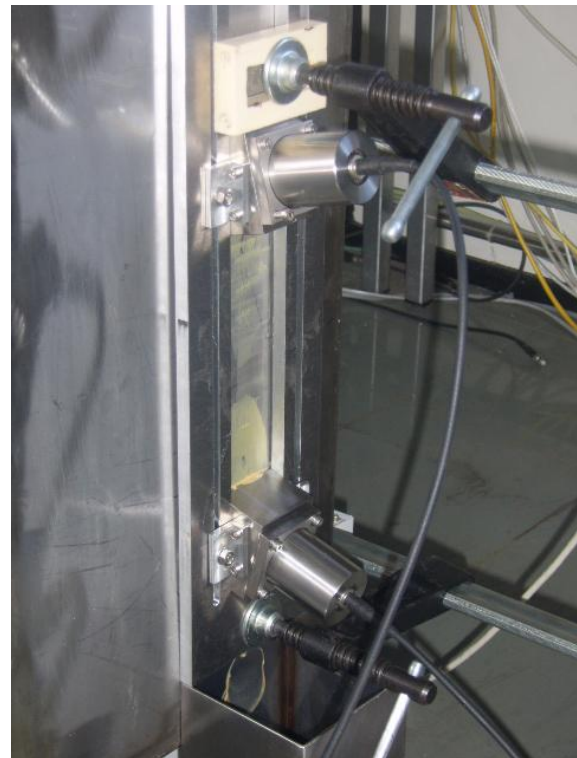


Figure 8.7.2. Actual experimental setup.

### Instrument Setup

- |                 |   |                     |
|-----------------|---|---------------------|
| • Wave Function | = | Truncated Sine wave |
| • Amplitude     | = | 20 V                |
| • Phase         | = | 270 <sup>0</sup>    |
| • Burst Count   | = | 1                   |
| • Frequency     | = | 500 kHz             |
| • Period        | = | 310 ms              |

- Amplifier rating = Maximum permissible with output clip, raising output amplitude to 30 V

**Transducer undergoing test**

Transducer 1

- Brand = Imasonic, IM-0.5-25-P (Immersion probe)
- Centre Frequency = 500 kHz
- Active diameter = 25 mm
- Housing length = 38.9 mm
- Housing diameter = 35 mm

Temperature of water = 20.7°C

**Calibration**

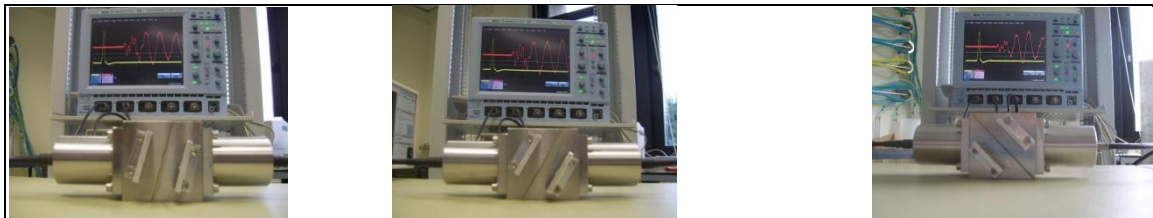


Figure 8.7.3, Delay in aluminium blocks represented on the oscilloscope, [15°, 30°, 45°].

**Results**

15° wedge – Time delay in wedges, cables and transducers = 12.746 μs

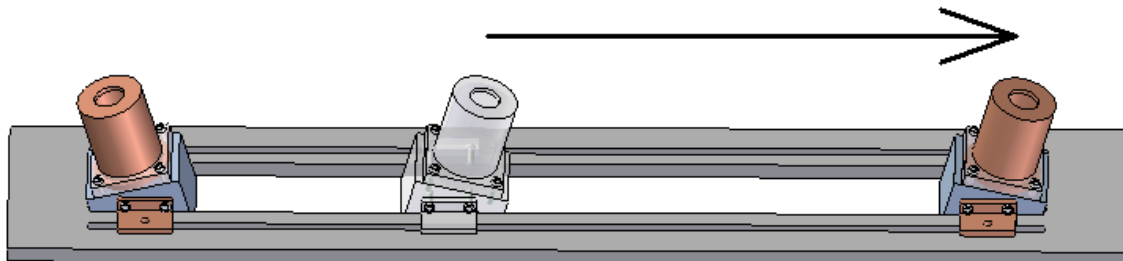


Figure 8.7.4, 15° wedge design setup, depicting movement of test transducer.

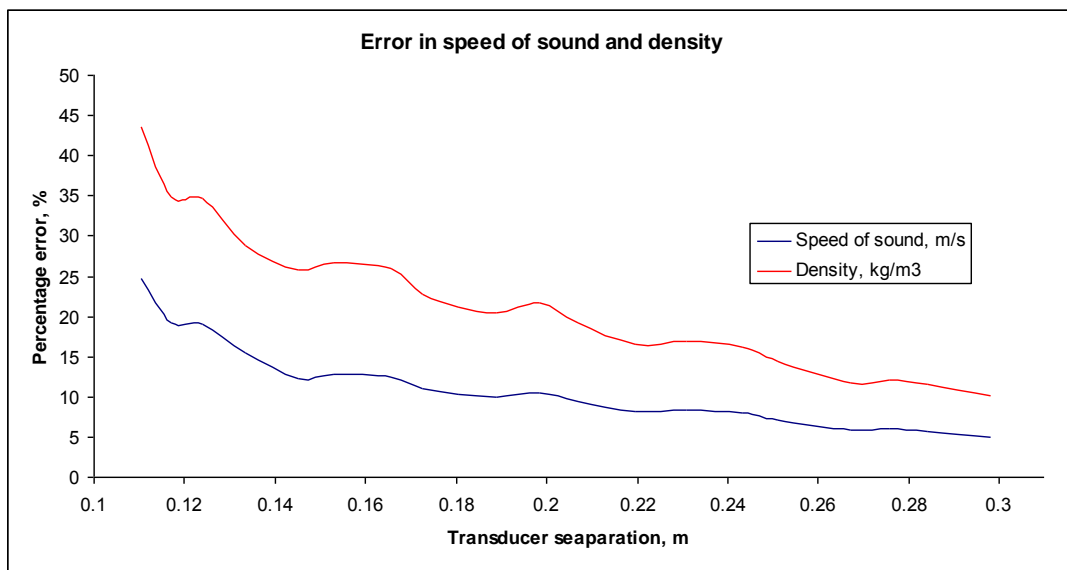


Figure 8.7.5, Error in speed of sound and density for 30° wedges.

30° wedge – Time delay in wedges, cables and transducers = 12.363 μs

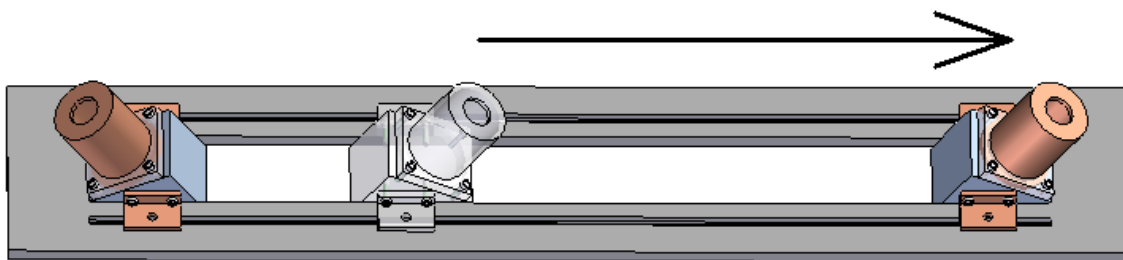


Figure 8.7.6, 30° wedge design se up, depicting movement of test transducer.

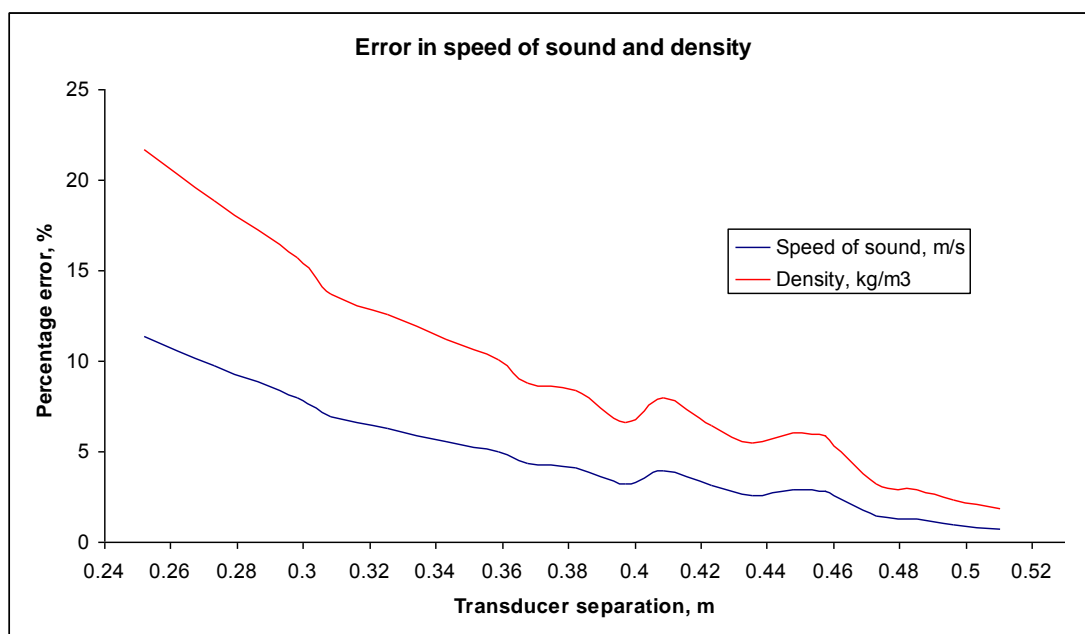


Figure 8.7.7, Error in speed of sound and density for 15° wedges.

45° wedge – Time delay in wedges, cables and transducers = 12.774 μs

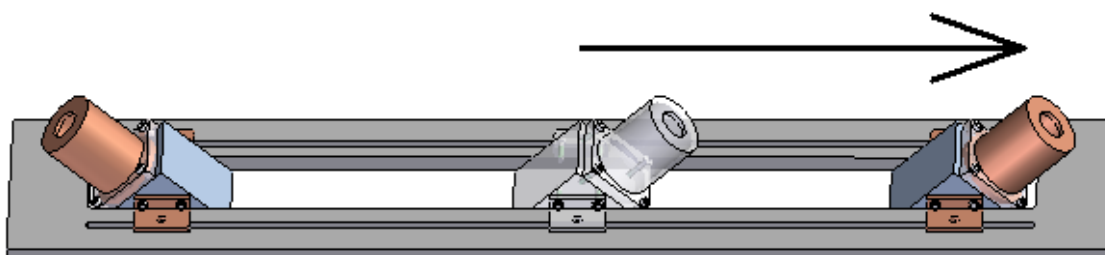


Figure 8.7.8, 45° wedge design setup, depicting movement of test transducer.

The graph for the 45° angled wedges has not been included due to the fact that no peaks could be picked out with any certainty from the background noise.

## Discussion

Firstly it can be seen that the inclusion of the wedge set up gives a marked improvement on the accuracy of the readings. The speed of sound is within 0.8% and the density within 2% of the actual values. These final readings are extremely promising and show that this technique can be used to determine changes in speed of sound to within  $12 \text{ ms}^{-1}$  and the changes in density to within  $20 \text{ kg.m}^{-3}$ . Referring back to Table 2.2, Acoustic Properties of Representative Liquids, the proposed design could therefore determine between sea water and fresh water (a change in speed of sound of as little as  $50 \text{ ms}^{-1}$ ) with a precise accuracy. Beyond this, most of the other solutions could be precisely characterised by this technique. However there are a few problems associated with this design which need to be discussed.

Firstly the selection of a peak on the oscilloscope is an extremely sensitive issue. The increased attenuation from the aluminium blocks create further problems when trying to select peaks and the increased number of interfaces the wave must pass through distorts the signal again. Furthermore, the fact that the transducers are held at an angle means that refraction processes occur. This causes a lot of problems creating more Rayleigh (surface) waves, disturbing the receiving transducers and removing a portion of the longitudinal signal. All this encapsulated means that the signal is extremely noisy and precise peaks are extremely difficult to pick out. When the  $45^{\circ}$  angle was used no signal could be detected at all beyond the back ground noise.

As the transducers are strapped to aluminium blocks as well this causes problems for the transducers both emitting and receiving. The increased surface area in contact with the wall of the tank reduces the signal amplitude and causes unusual attenuation of the signal. It also meant that a larger area of the tank had to be flat and therefore the device could not be secured satisfactorily, disabling it from working on the large storage tank (tank number 3).

## 8.8. The Field Test

### Aim

- To perform a test on a different liquid to investigate whether an accurate characterisation of the liquid can be achieved using just the time domain response.

### Experimental Setup

- The set up shown in Figure 8.8.1 was used.
- All transducers were left stationary and a single test was done.
- The test was performed on a large glass wine bottle containing wine at an alcohol content of 10 %.

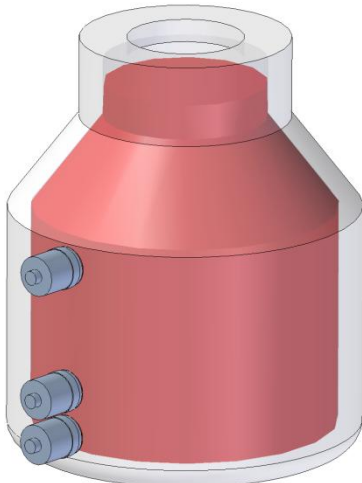


Figure 8.8.1. Experimental setup.

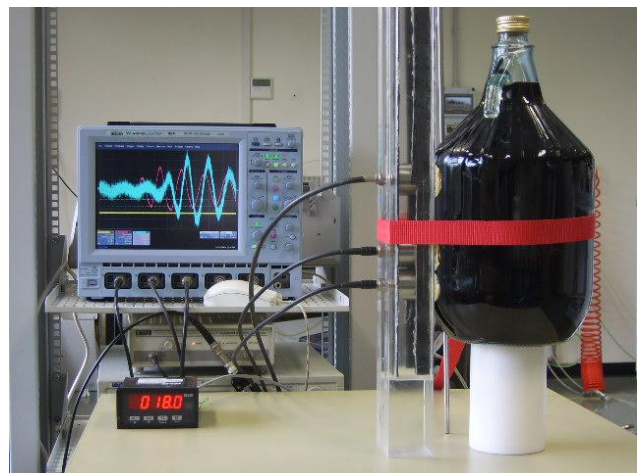


Figure 8.8.2. Actual setup depicting returned echoes.

### Instrument Setup

- Wave Function = Truncated Sine wave
- Amplitude = 20 V
- Phase =  $270^{\circ}$
- Burst Count = 1
- Frequency = 500 kHz
- Period = 310 ms
- Amplifier rating = Maximum permissible with output clip, raising output amplitude to 30 V

### Transducer undergoing test

#### Transducer 1

- Brand = Imasonic, IM-0.5-25-P (Immersion probe)
- Centre Frequency = 500 kHz
- Active diameter = 25 mm
- Housing length = 38.9 mm
- Housing diameter = 35 mm

Temperature of atmosphere around tank =  $18.0^{\circ}\text{C}$

## Results

Separation between signal and reference transducers	=	36.65 mm
Separation between signal and test transducers	=	114.15 mm
Propagation time for reference wave	=	226.696 $\mu$ s
Propagation time for test wave	=	236.076 $\mu$ s
<u>Experimental speed of sound</u>	=	<u>1640.84 m.s<sup>-1</sup></u>
Bulk modulus of sea water	=	2.35 G.Pa
<u>Experimental density</u>	=	<u>873 kg.m<sup>-3</sup></u>
Density measured using “Density Meter – DMA 35”	=	993 kg.m <sup>-3</sup>
Speed of sound of similar wine	=	1540 m.s <sup>-1</sup>

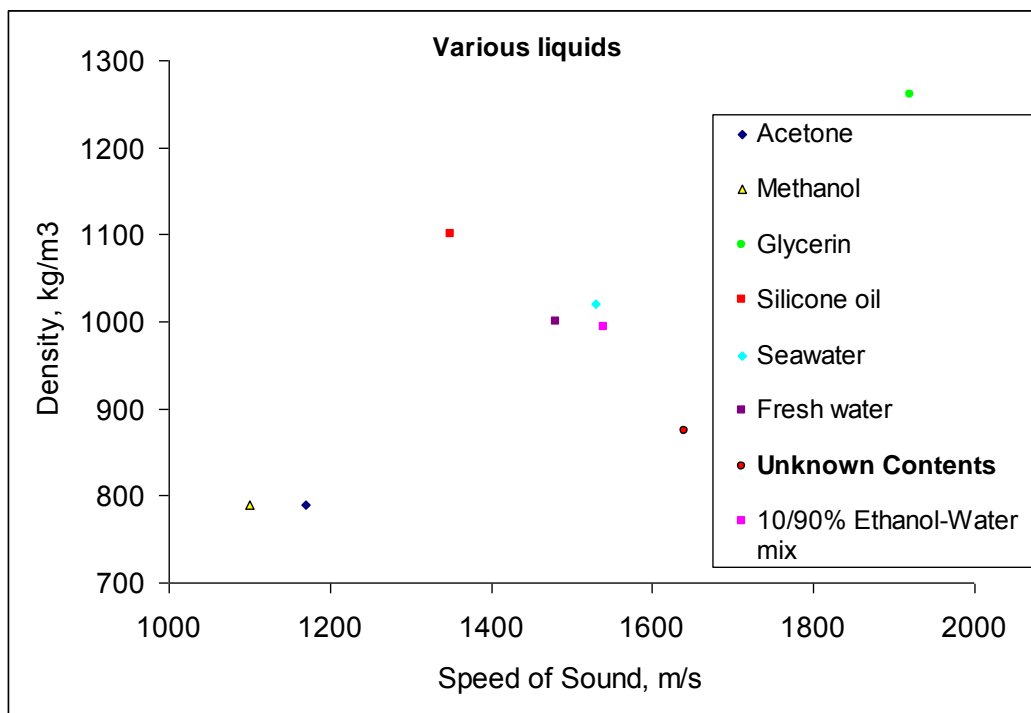


Figure 8.8.3, Graph depicting speed of sound versus density for various liquids.

## Discussion

Figure 8.8.3, it can be seen that the most accurate model of the contents of the tank is that of the 10/90% Ethanol-Water mix. The content of the container was 10% alcohol table wine and so this technique has accurately managed to decipher the correct solution. The liquids plotted in Figure 8.8.3 were those from Table 2.2 that were stable at the 18<sup>0</sup>C. The 10/90% Ethanol-Water mix was included after the content of the barrel was known.

Perhaps the number of liquids plotted in Figure 8.8.3 is not comprehensive enough and this has allowed the system to correctly identify the solution. The error in the signal is large and other liquids could easily be chosen other than the 10/90% Ethanol-Water mix. The method of calculating the density involved the use of the bulk modulus for sea water taken from Table 3.1, as this was the closest value given for the calculated speed of sound, which inherently incurs an error. However, if just the speed of sound is

analysed then the closest model to the predicted model is again the 10/90% Ethanol-Water mix. This experiment therefore shows extremely encouraging results for managing to characterise a solution.

## 9. Conclusion

Considering the results of the eight experiments undertaken into determining the feasibility of an ultrasonic densitometer for non-invasive in-field detection of illicit liquids in suspect containers, there are a number of conclusions which can be drawn. All the experiments concur that as the transducer separation increases so the propagation time increases. The geometric relationship combining these propagation times and the transducer separation accurately models the speed of sound and hence density of the solution. Referring to the introduction, the following paragraphs confirm that in view of the initial outcomes, the proposed design has satisfactorily achieved merit in most criteria; with the specific design conclusions being discussed later.

### 9.1 Outcome Assessment

**Accuracy:** The time domain response consistently returned reliable speed of sound and density analysis. The errors in the results have been reduced to such a level that accurate detection of a solutions speed of sound can be obtained. The proposed design can determine the speed of sound of a tanks contents to within 0.8% and the density to within 2%. This enables the user to be able to determine between very small differences in acoustic velocity such as that of fresh and salt water.

**Precision:** Unfortunately the precision of the device is not as good as the accuracy. Varied results have been obtained for the same tests on the same tanks. Spreads of over 7% for the density results and 5% for the speed of sound results have been found for repeated tests. This is due to the selection of incorrect peaks on the oscilloscope. When the wave propagates through a system, especially at large angles of incidence and high transducer separations, the wave interferes heavily with other waves in the tank. These added signals mean that the selection of the correct reflection peak from the background noise can be extremely difficult. The selection of an incorrect peak can significantly affect the experimental speed of sound and density, hampering the chances of modelling the contents of the tank with any confidence. The reasons behind the inaccuracies experienced cascade down to the fact that the wrong transducers were used for the experiments. Contact transducers should have been used for the analysis but only immersion transducers were available. Immersion transducers are made to interface with a liquid and not a solid.

**Adaptability and Robustness:** As the test has been shown to display a constant error across a variety of operating temperatures, it can be concluded that the device may be used out with the laboratory, where temperature ranges can fluctuate depending on a number of variables. As the propagation times alter with changing temperature then it is difficult to conclusively determine the contents of the tank using the speed of sound and density alone. A demonstration of the effect of temperature on water has been performed but for other solutions, not such a complete data analysis has yet been produced. Furthermore, as the digital thermometer was placed outside the tank, then this does not deliver the actual temperature of the solution within the tank. Hence, if the solution within the tank is not in an isothermal state or is heated via a localised source, then this could not be compensated for with the proposed design.

**Portability and Usability:** The wedge design provides an extremely accurate method of determining the separation of the transducers. As the transducers are attached to a guide rail then the system also becomes highly portable and simple to attach securely and quickly to a tank wall. However, the system still requires a high level of human interaction. The use of the ultrasonic pulse delay evaluation unit, described in experiment 3, created a higher degree of automation however a delay must be built into this device before any reliable results are achieved.

### 9.2 Specific Design of a Reflected Beam Ultrasonic Densitometer

A system measuring the acoustic resonance spectrum of a tank is theoretically the best approach, as the data returned would provide a method of detection which is insensitive to mechanical vibrations.



However, experimentally the results produced in the short period of time that the author had access to frequency domain capture equipment highlighted that the clarity of the signal was a major stumbling block in the success of this technique. This method would also require further study into how the waves propagate within the tank. The authors proposed concept, discussed in the Appendix A, may not be ultimately accurate, however it is hard to establish why an increase in signal amplitude is experienced without constructive interference being present. Additionally the practicality of this technique is compromised due to the extended algorithms used during Fast Fourier Transform requiring large, expensive memory banks to perform the operations. If this technique was to be deployed for in-field testing a cheaper instrument would have to be produced.

It can be seen, both experimentally and theoretically, that the most accurate analysis of the speed of sound and density comes when the reference transducer is as close as possible to the signal transducer and the test transducer is as far away as possible. Therefore the test would benefit from the usage of transducers with smaller active diameters and smaller casings. Furthermore if these smaller transducers had a wider central focus then a greater separation would be permitted and this would again increase the accuracy of the results.

The wedge design is a clever method of combating the effects of divergence; however the inclusion of this device was only the answer to the problem of the specific transducers that were available and not to the overall design. Ideally transducers would be used which had a wider centre frequency, therefore allowing a greater distance between the transducers to be experienced without the loss of signal quality experienced by the inclusion of these aluminium wedges.

A significant error comes from ignoring the effect that the tank walls have on the speed of sound and density. As the specification denotes that the device must operate on tanks of different materials and the operator cannot know anything about the material before testing, then this issue escalates further. For thin walled tanks and metals, where the speed of sound is high, the error is small, but on different media such as plastic or glass with low speeds of sound or thick metal tank walls then this error becomes significant. There is also a delay in the transducers, which is small but becomes significant again when the tank diameters are small. The method of combating this effect comes from analysing the initial peaks returned from the time domain response technique, hence harnessing the overall time delay and subtracting these from the reflected TDR propagation times. However, the initial returned peaks could be multi-reflected longitudinal waves, Rayleigh waves or a combination of the two, which makes accurate modelling and analysis very difficult.

It is therefore proposed that the ideal system would combine both time domain response and acoustic resonance spectroscopy. Time domain analysis would be done initially to determine the effect of the tank wall and this would then be followed up by an FFT analysis of the tanks contents. To gain a fuller picture of the tanks contents, the attenuation of the signal should also be considered. If the errors in the propagation times do not allow accurate analysis of the speed of sound and density required for precise characterisation of the liquid, then establishing a further unique characteristic may compensate for this. The best application for this design will be to test to see if the sealed container is hiding anything unexpected, as these analyses will vary greatly from previous tests. Arriving at a completely unknown container with this device may not provide the required ultimate characterisation of the contents but will certainly highlight the state of what is contained inside. If some anomaly is found with this test, then the suspect container should be removed and sent to the laboratory for further examination.

## 10. Recommendations

Part of the aim of this report was to produce a base from which further investigation into the ultrasonic densitometer could be performed. This section of the report should be considered as an aid for future research.

### 10.1 Time Domain Response - Further analysis

This method shows extremely good, reliable results and further research on this technique should only be considered within:

- Testing numerous different substances to help build an empirical database to cross-reference results provided against stored data.
- Using a higher voltage amplifier to create a larger amplitude from the signal transducer.
- Investigating methods of creating a higher degree of automation for the overall process.

### 10.2 Acoustic Resonance Spectroscopy – Further analysis

As little access was provided during my period of testing to any frequency domain data capture equipment then there has not been significant progress from S. Fowler's (2005) report. However there are a number of suggestions that can be put forward following a study of the theory behind the principle and on reported difficulties experienced by S. Fowler.

If the same sweep frequency signal was used as in S. Fowler's report (2005) which varied 50 kHz over a time interval of 1 second, then a full standing wave of one single frequency could not be set up in the tank. It is the author's belief that S. Fowler's unusual results stemmed from the fact that the peaks on the FFT diagrams were not well pronounced. To combat this estimation of the peak separation needs to be found with more precision; hence more constructive feedback is needed. Therefore the signal should be allowed to create a standing wave by increasing the time taken for the analysis. A minimum time interval is therefore needed and can be calculated as follows:

$$t_{standingwave} = \frac{2 \times d_{tankdiameter}}{c_{speedofsound}}$$

If this was to be implemented across the 50 kHz frequency range quoted earlier, sampling every 1 Hz, then  $t_{standingwave}$  would have to be multiplied by a factor of 50,000. This would create a very long test time, but could ultimately provide very accurate results.

## References

- BHARDWAJ, M.C, (2001) *Non-contact Ultrasound: The last frontier in non-destructive testing and evaluation* (New York: John Wiley & Sons)
- CROWELL, J (2006) *Vibrations and Waves* (Licensed under the Creative Commons Attribution-ShareAlike license: <http://www.lightandmatter.com>)
- DAVID, J, CHEEKE, N (2002) *Fundamentals and Applications of Ultrasonic waves* (Montreal: CRC Press)
- European Commission Directorate General for Agriculture (2004) *The Common Agricultural Policy Explained* (Germany: European Commission)
- FOWLER, S. (2005) *Operational proof of the Ultrasonic densitometer for non-invasive detection of toxic liquids in metal tanks*. Unpublished MEng Thesis, University of Glasgow.
- HERZFELD, F.F, LITOVITZ, T.A, (1959) *Absorption and dispersion of Ultrasonic Waves* (London: Academic Press INC. (London) Ltd.)
- JAKKIDI, J. (2004) *Fast Fourier Transform*, Online presentation, <http://www.ni.com/swf/presentation/us/fft/> [Accessed 10<sup>th</sup> March 2006]
- JANSSENS-MAENHOUT, G, DECHAMP, L (2002) *Densitometer for infield inspection of tanks containing liquids*. Unpublished thesis, Institute for Protection and Security of the Citizen, European Commission, Joint Research Centre Ispra.
- LOPRESTI, V (2002) *Sound Solutions for Safety, Health, and Security* (Los Alamos Research Quarterly) p. 18-21
- LURTON, X. (2002) *An Introduction to Underwater Acoustics, Principles and Applications* (Chichester, UK: Praxis Publishing)
- MATERIAL PROPERTY DATA (1998) *Physical properties of Water*, <http://www.matweb.com/search/SpecificMaterial.asp?bassnum=DWATRO>, [Accessed 16<sup>th</sup> March 2006]
- MATSON, J, MARIANO, C.F, KHRAKOVSKY, O, LYNNWORTH, L (2002) Ultrasonic Mass Flowmeters Using Clamp-On or Wetted Transducers. In: *5<sup>th</sup> International Symposium on Fluid Flow Measurement, Arlington, Virginia, 8<sup>th</sup> April 2002*.
- NATIONAL PHYSICS LABORATORY (2004) *Technical Guides – Speed of Sound in Pure Water*, <http://www.npl.co.uk/acoustics/techguides/soundpurewater/content.html#BILANIUK>, [Accessed 1<sup>st</sup> February – 31<sup>st</sup> May 2006]
- REDWOOD, M. (1960) *Mechanical Waveguides* (Bristol: Pergamon Press Ltd.)
- SINHA, D.N (1992) *Acoustic resonance spectroscopy*, extract from Institute of Electrical and Electronics Engineers database.
- SINHA, D.N (2002) *Multi-purpose Acoustic Sensor*, Slides from presentation given in Morgantown, WV, USA, September 16<sup>th</sup>, Los Alamos National Laboratory.

SINHA, D.N, OLINGER, C.T, LYON, M.J, STANBRO, W.P, MULLEN, M.F (1993) *Acoustic resonance spectroscopy in Nuclear Safeguards*. In 34<sup>th</sup> annual Meeting of the Institute of Nuclear Materials Management, Scottsdale, Arizona, USA, July 18<sup>th</sup>, Los Alamos National Laboratory.

THE ENGINEERING TOOL BOX (2005) *Bulk Modulus and Fluid Elasticity*, [http://www.engineeringtoolbox.com/bulk-modulus-elasticity-d\\_585.html](http://www.engineeringtoolbox.com/bulk-modulus-elasticity-d_585.html) [Accessed 28<sup>th</sup> March 2006]

VAN DEVENTER, J. (2001) *Material Investigations and Stimulation Tools Towards a Design Strategy for an Ultrasonic Densitometer*. Unpublished PhD thesis, Lulea Tekniska Universitets Tryckerie University.

**Appendix**

## Appendix A - Acoustic Resonance Spectroscopy (ARS)

Another method of capturing the propagation time of the transmitted wave is by studying the tank's acoustic resonance spectrum.

### A.1 Vibration and Resonance

All systems possessing mass and elasticity can vibrate. There are two general classes of vibration, free and forced. Free vibrations occur under forces contained within a system, whereas forced vibration occurs due to excitation from external forces. Free vibrations are excited in any mechanical system that is disturbed from equilibrium and then left alone. Natural frequencies (or normal modes) are associated with free vibration and depend on physical system properties; these properties are the systems mass, stiffness and distribution. When a system undergoes forced vibration, at certain frequencies the energy transfer between the external exciting source and the vibrating system will reach a maximum. This phenomenon is called resonance and coincides with the system's natural frequencies, therefore allowing some key physical properties of the system to be found.

### A.2 Standing Waves

The modes of vibration associated with resonance in an extended system have characteristic patterns called standing waves. These standing wave modes arise from a combination of reflection and interference such that the reflected signals interfere constructively with the sent signals. Waves which undergo reflection experience complex transforms (for the moment we will consider only perfect reflection) through which the incident wave is reflected in a direction symmetrical to its direction of arrival, with no loss of amplitude. When a signal is sent with a wavelength of exactly half the diameter of the tank the first harmonic is created and this is called the fundamental harmonic. The frequency of the fundamental harmonic can be easily calculated using the following equation:

$$f_{\text{fundamenta}} = \frac{2 \times c_{\text{speedofsound}}}{d_{\text{diameterofank}}}$$

Following the fundamental frequency, the successive harmonics can be found:

$$f_{\text{harmonic}} = n \times f_{\text{fundamenta}}$$

where  $n$  is an integer.

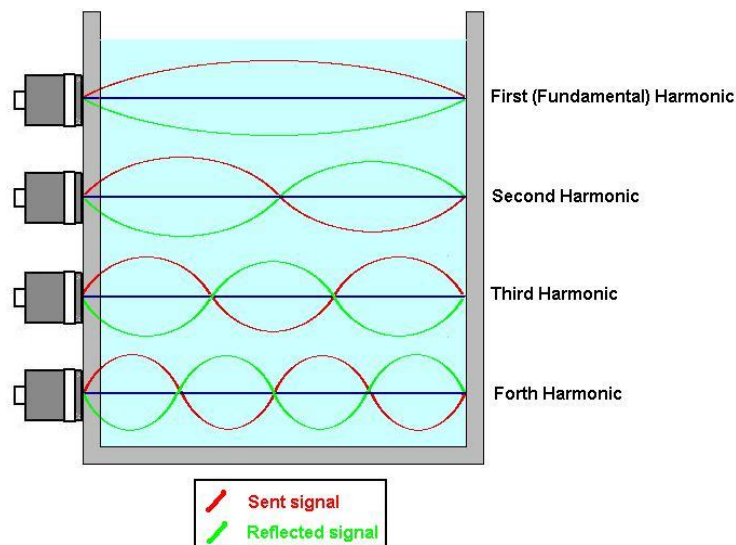


Figure A.1, Example of the first four harmonics of a system.

A.3 Forced Vibration and Resonance Exploitation

Forced vibration is experienced when a system is excited via external forces. When the frequency of the excitation is periodic, the system responds by exhibiting the same vibration frequency as the excitation frequency. If the vibrating system resonates at one of its natural frequencies, the amplitude of the steady state response of the system raises to a maximum. This signifies that constructive interference has been achieved which constitutes a positive feedback loop creating resonance. If the damping of the system is at a sufficiently low level, violent oscillations will then be experienced. The increase in feedback set up by the standing wave can be analysed and captured via Fast Fourier Transform analysis.

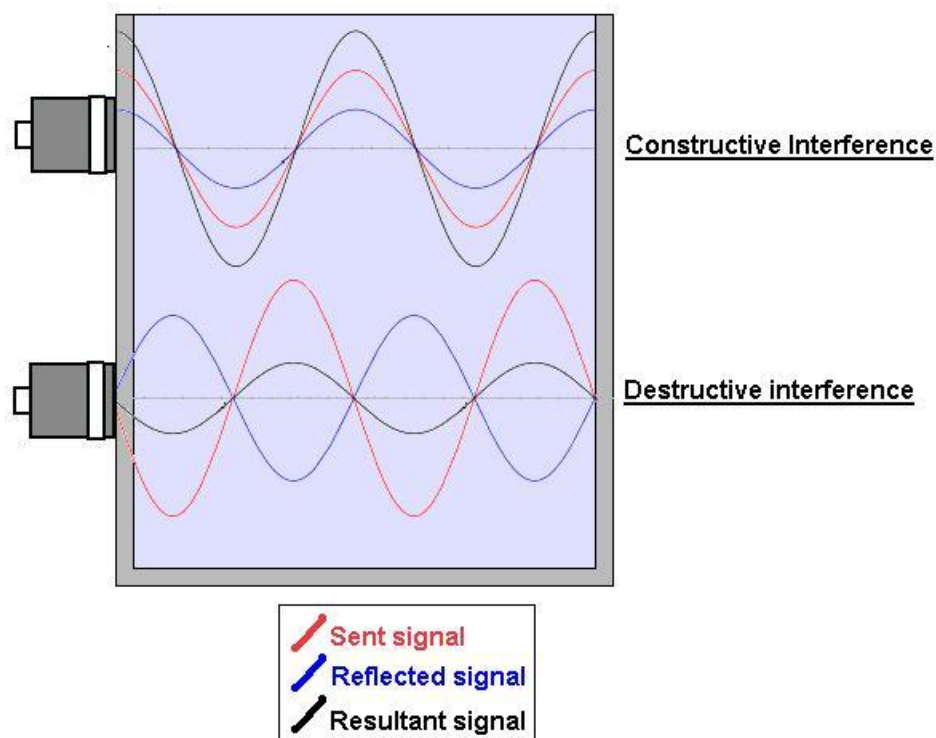


Figure A.2, Constructive and destructive interference models for pressure waves.

A.4 Fast Fourier Transform (FFT)

When measurements are considered, they are often perceived in terms of measurements in the time domain as most data acquisition hardware takes time domain measurements. However, there are some measurements that are difficult or impossible to perform in this domain. For example, sound quality is difficult to measure using only the time domain. But the frequency domain allows the quality of a signal to be analysed, helps isolate distortion and removes noise. It also allows the extraction and measurement of individual signals from complex multi-tone signals.

All periodic signals can be decomposed into a sum of sinusoids of various frequencies and amplitudes. The Fast Fourier Transform (FFT) is a class of algorithms that computes the magnitude of energy versus frequency of these periodic signals. It does this by assuming the time domain signal is composed of a sum of sinusoidal signals of various frequencies. The algorithm computes the amplitude of each of these sinusoids and the result is plotted as a magnitude versus frequency graph.

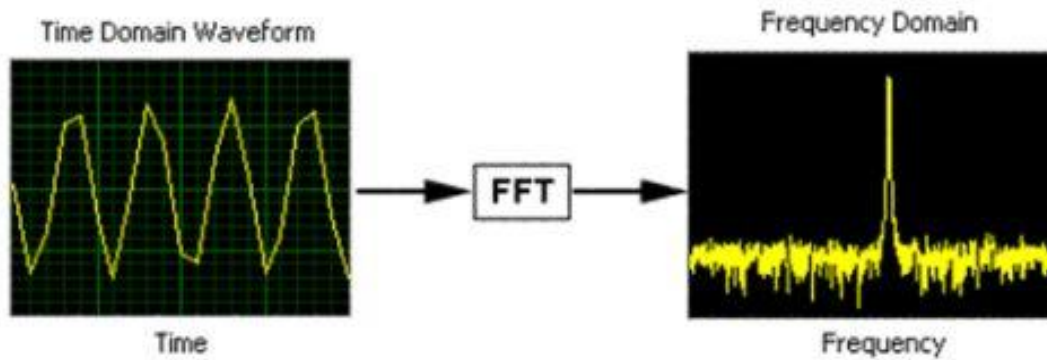


Figure A.3, Time domain to frequency domain analysis.

FFT-based spectral analysis can be applied to just about any signal, but there are certain signal characteristics that allow much more meaningful results to be obtained. The first characteristics arise from the nature of the FFT itself. In order to decompose a signal in terms of infinitely long sinusoids, the FFT extends the finite time record by repeating it periodically. If the sampled data doesn't contain exactly one cycle of an underlying periodic signal, the extension of the FFT may produce artificial discontinuities at the cut off frequencies of the sampled data. These discontinuities show up as extra frequency content in the FFT result, a phenomenon known as spectral leakage.

For the most accurate spectral analysis, the original signal should be contained within a finite frequency band. A square wave is not band-limited since it requires sinusoids of infinitely high frequency to represent it in the frequency domain. This is why truncated sine waves of an appropriate frequency are used.

An FFT does not contain any information about the time evolution of a signal. If the frequency content of a signal changes within the time record, the FFT gives no indication of when or how that change occurred. It does, however, give a summary of all frequencies contained in the sampled data.

The first relationship and one of the most fundamental rules of sampling<sup>11</sup> is called the Nyquist Theorem. The theorem states that the highest frequency,  $F_{max}$ , which can be accurately analysed in the frequency domain, is one-half of the sampling frequency rate,  $f_{samplingfrequency}$ , used to capture the time domain signal:

$$F_{max} = \frac{f_{samplingfrequency}}{2}$$

For example if the full 20 kHz audible bandwidth of the human ear is to be represented, sampling of the audio signal must be done at twice the maximum frequency, or at least 40 kHz. In reality, samples should be taken at 2.5 to 3 times the frequency of the highest component being measured. If the sample rate is not at least two times the rate of the highest frequency, these frequencies above one-half the sampling rate appear as lower frequency components and hence, an incorrect measurement. This is called aliasing. A signal is aliased when higher frequency components appear in the lower frequency ranges due to incorrect sampling rates. Aliasing creates incorrect data and is impossible to filter.

This second important relationship considers frequency resolution,  $\Delta f$ . As in the time domain, resolution determines how precisely you can examine data. However, in the frequency domain, frequency resolution is proportional to the total length of time,  $T$ , of the waveform's acquisition, not the number of sample points,  $N$ . One important note about frequency resolution is that you can get the same frequency resolution using various sample frequencies and sample durations.

<sup>11</sup> Sampling is the reduction of a signal from a continuous signal to discrete, or quantised signal.



$$\Delta f = \frac{1}{T} = \frac{f_{\text{sampling frequency}}}{N}$$

An FFT spectrum is what results when you apply the FFT algorithm to a time domain signal creating a double-sided complex-valued array. Some more processing is then required to convert this into a graph. Specifically the complex values need to be converted to magnitude and phase and the redundant negative frequencies removed from the analysis.

### A.5 Application to Density Analysis

Once data has been put through a Fast Fourier Transform program, the output graph displays sharp resonance peaks seen in Figure 3.5. The spacing between these sharp resonance peaks is constant with the frequency difference between any two consecutive peaks denoting the fundamental frequency. Once the fundamental frequency  $f_{\text{fundamental}}$ , has been found, the speed of sound,  $c_{\text{speed of sound}}$ , can then be calculated:

$$c_{\text{speed of sound}} = \lambda \times f_{\text{fundamental}}$$

where,

$$\lambda = 2 \times d_{\text{diameter of tank}}$$

Also the propagation time,  $t_{\text{propagation}}$ , of the wave can be derived by considering the fundamental harmonic, the period of this signal being equal to the propagation time:

$$t_{\text{propagation}} = \frac{1}{f_{\text{fundamental}}}$$

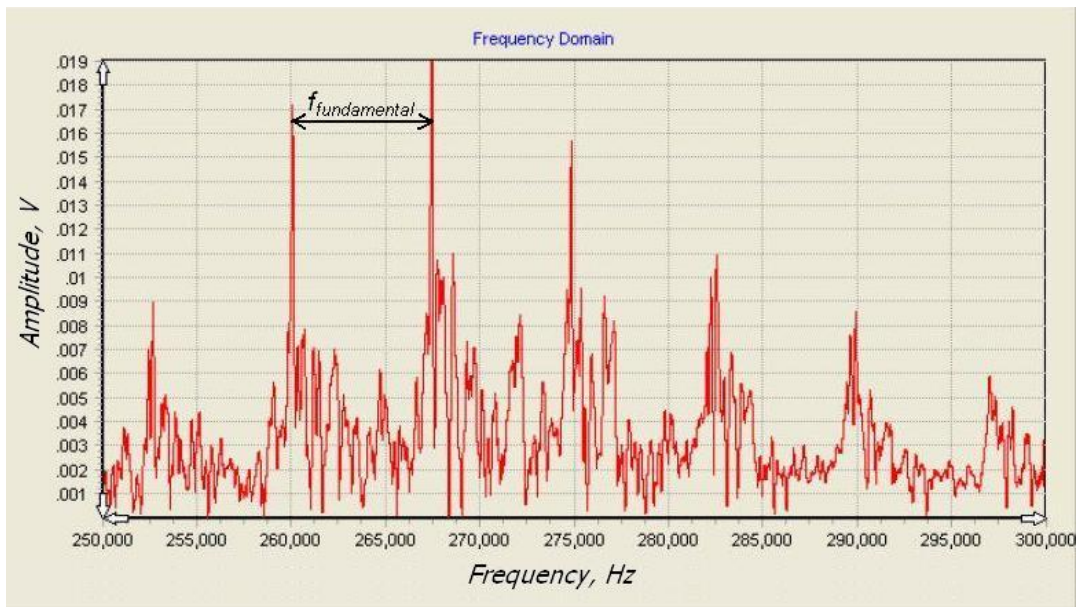


Figure A.4, FFT analysis displaying prominent resonance peaks.

This technique works very well if you already have information about the diameter of the tank undergoing test. Unfortunately, the design specification of this ultrasonic densitometer does not provide information on this and so a different set up must be considered.

### A.6 The Two Concepts

When the transducers are arranged in the same geometric set up as for the time domain response experiments, shown in Figure 3.3, then one of two phenomena are observed. The concept which was used by S. Fowler in his investigation into analysing a systems acoustic resonance spectrum it was stated that the propagating displacement wave from the signal transducer underwent divergence and was received by the receiving transducer. Altering the position of the transducer on the other side of the tank would alter the harmonic frequencies and hence change the propagation time due to the extra distance travelled by this wave. When related to the proposed reflected beam method (see Figure A.5) the speed of sound can be calculated via the following relationship:

$$c_{speedofsound} = 2 \times f_{fundamenta} \times \sqrt{d_{adj}^2 + \left(\frac{d_{separation}}{2}\right)^2}$$

However this relationship does not explain the increase in amplitude on the FFT diagram (see Figure A.4) as no constructive interference is experienced. What is proposed that happens is the displacement wave does not diverge and reflect back to the test transducer, but travels on the horizontal plane, interacts and reflects perfectly back from the wall directly across from the signal transducer. The pressure wave propagates through the liquid lagging the displacement wave by  $90^\circ$  and it is this which undergoes constructive interference, increasing with amplitude upon reflection (see Figure A.6). This signal picked up by the transducers creates the increase in feedback amplitude displayed on the FFT diagram. What is received by the receiving transducers is the wave interacting with the wall of the tank and not the diverged wave. Therefore this means that the frequency distance between the peaks is irrespective of transducer separation which in turn means that, for this technique, the speed of sound of the medium is not a function of transducer separation.

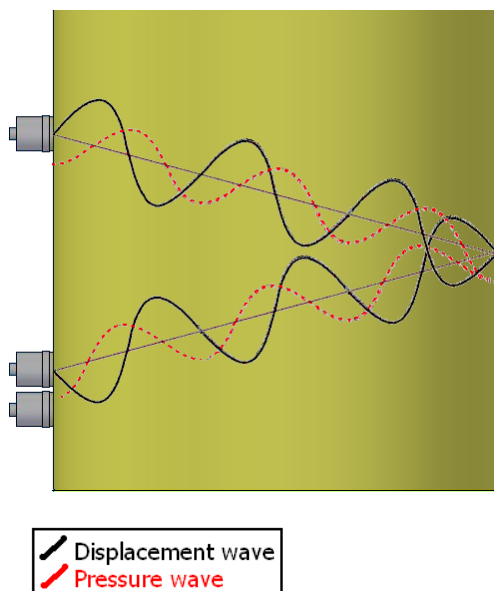


Figure A.5, S. Fowler's model of wave propagation.

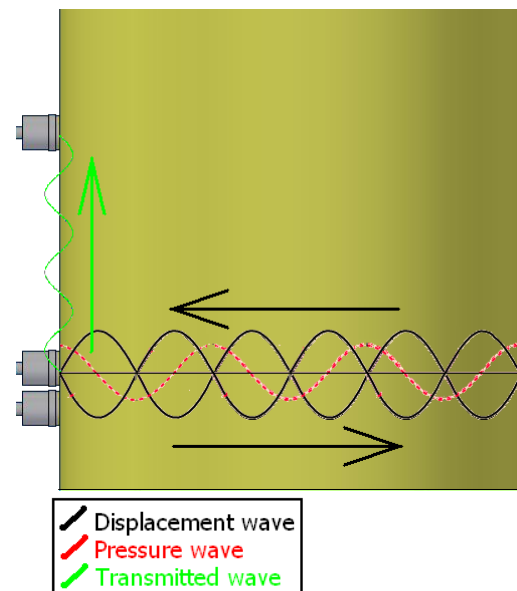


Figure A.6, Author's proposed model

### A.7 Further Applications of this Technique

This type of technique is useful for a number of analyses, far beyond just the determination of density of a liquid. At present in Los Alamos National Lab, NM, United States, research is ongoing to the application of this technique to detect the removal and replacement of tank lids from a nuclear materials drums. The acoustic spectrum of a container establishes a baseline fingerprint, referred to as the intrinsic seal, for a container. The complex stress patterns which occur within the tank and lid are impossible to maintain

following removal and replacement. Therefore acoustic resonance techniques undertaken on the seals can be used to determine whether tampering has taken place within these vessels.

Other areas of application of this type of technique encapsulate much wider and more diverse areas of investigation. This technique is being developed for use in the medical sector to solve such problems as imaging breast cancer without exposing women to the high energy radiation involved in mammography and monitoring blood-sugar levels without the need for syringes and intrusive investigation. It is also being used effectively to remotely detect structural defects in natural-gas pipelines without interrupting supply to customers.

## Appendix B - Attenuation

### B.1 Liquid Characterisation

The final step in the classification of a liquid is to perform a study of the attenuation of the propagating sound waves travelling through the liquid. When a pulse propagates through a medium it bounces backwards and forwards until absorption and scattering effects fully attenuate the signal. The rate at which this occurs is called the attenuation coefficient of the liquid. Losses or absorption of acoustic energy occur within all liquids due to characteristic properties such as viscosity and thermal conductivity. Furthermore, there are molecular processes where acoustic energy is converted into internal molecular energy. Therefore all of the loss effects in liquids can be described by a phase lag between acoustic pressure and the medium response.

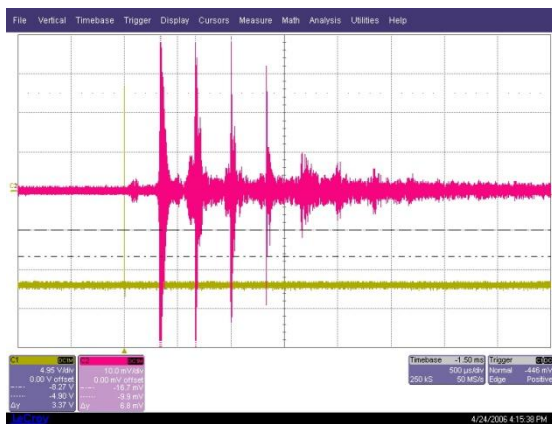


Figure B.1, Oscilloscope example of attenuation.

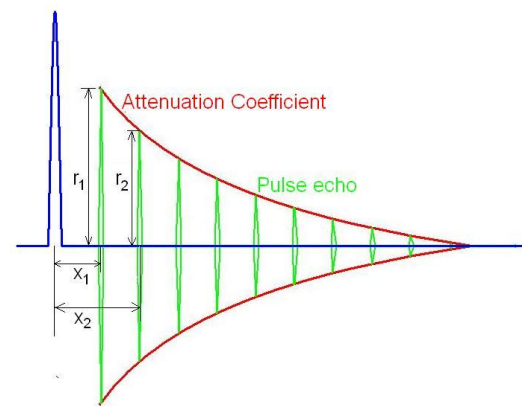


Figure B.2, Theoretical model of attenuation.

### B.2 Application of the Decibel Scale to the concept of Attenuation

Considering the displacement,  $u$ , of a propagating wave:

$$u = u_0 \exp j(\omega t - kx)$$

When we add the concept of dissipation, the wave vector,  $k$ , becomes complex transforming to  $\beta - j\alpha$ , where  $\alpha$  is the attenuation coefficient for the amplitude of the wave.

$$u = u_0 \exp j(\omega t - \beta x) \exp(-\alpha x)$$

In attenuation measurements, where plane wave conditions are standard, the acoustic intensity,  $I$ , is directly proportional to square of the displacement,  $u^2$ , and therefore the signal decays following:

$$I = \exp(-2\alpha x)$$

The factor of two comes from the difference in attenuation between the amplitude and the intensity due to the quadratic term.

### B.3 Application of the theory

When applying the theory to an actual system the attenuation of the amplitude is measured by determining the amplitude ratio,  $r_{12}$ , of the wave at two different positions  $x_1$  and  $x_2$  (see Figure 5.2):

$$\frac{r_1}{r_2} = r_{12} = \exp \alpha(x_2 - x_1)$$

The attenuation is measured in nepers and so the attenuation coefficient,  $\alpha$ , is measured in Np/m. Acoustic signals are usually represented using the decibel (dB) scale to compare acoustic intensity levels. Therefore, to transform  $\alpha$  into dB/m we must perform a further calculation:

$$\begin{aligned} \text{attenuation (dB)} &= 10 \log_{10}(r_{12})^2 \\ &= 20(\log_{10} e) \alpha(x_2 - x_1) \end{aligned}$$

#### B.4 Attenuation due to Density changes

Attenuation is normally characterised by considering the complex result of the wave vector,  $k$

$$u = u_0 e^{-\alpha x} \exp j(\omega t - \beta x)$$

Considering Stokes Law for pressure, the wave equation becomes

$$\frac{\partial^2 u}{\partial t^2} = V_0^2 \frac{\partial^2 u}{\partial x^2} + \frac{\eta}{\rho_0} \frac{\partial^2 u}{\partial x \partial \tau}$$

Substituting for the displacement,  $u$ , and separating real and imaginary parts

$$\begin{aligned} \alpha^2 &= \frac{\omega^2}{2V_0^2} \left( \frac{1}{\sqrt{1+\omega^2\tau^2}} - \frac{1}{1+\omega^2\tau^2} \right) \\ \beta^2 &= \frac{\omega^2}{2V_0^2} \left( \frac{1}{\sqrt{1+\omega^2\tau^2}} + \frac{1}{1+\omega^2\tau^2} \right) \end{aligned}$$

For most fluids undergoing investigation at ultrasonic frequencies at room temperature,  $\omega\tau \ll 1$  which therefore allows the simplification of the imaginary part of the wave vector:

$$\alpha \approx \frac{\omega^2 \tau}{2V_0} = \frac{\omega^2 \eta}{2\rho_0 V_0^3}$$

From this equation it can be seen that attenuation is not only a property of density, but also a property of a number of other medium attributes. This unique combination allows for verification of liquids to occur. Another interesting result here implies that  $\alpha \approx \omega^2$ , which means that attenuation,  $\alpha$ , rises rapidly with frequency. Therefore, when choosing a frequency for this type of analysis, careful considerations must be undertaken to make sure the correct operating frequency and transducer is selected.

## Appendix C - Tank Dimensions

### C.1 Tank 1 – Small metal tank, square base

#### Dimensions

- Breadth = 104.55 mm ± 0.025 mm
- Width = 104.55 mm ± 0.025 mm
- Wall thickness = 2.25 mm ± 0.025 mm

#### Material

- Mild steel

### C.2 Tank 2 – Large metal tank, rectangular base

#### Dimensions

- Breadth = 399 mm ± 0.5 mm
- Width = 305 mm ± 0.5 mm
- Wall thickness = 3.20 mm ± 0.025 mm

#### Material

- Mild steel

### C.3 Tank 3 – Large metal storage tank, circular base

#### Dimensions

- Diameter = 1.595 ± 5 mm
- Circumference = 5.01 m ± 5 mm
- Wall thickness = Unknown

#### Material

- Mild steel

**Appendix D – List of Abbreviations**

ARS	Acoustic Resonance Spectroscopy
CCTV	Closed-Circuit Television
EU	European Union
EURATOM	European Atomic Energy Community
FFT	Fast Fourier Transform
HEU	High Enriched Uranium
IAEA	International Atomic Energy Authority
IPSC	Institute for the Protection and Security of the Citizen
JRC	Joint Research Centre
LEU	Low Enriched Uranium
NM	New Mexico
NPT	Non-Proliferation Treaty
SNM	Special Nuclear Materials
TDR	Time Domain Response

European Commission

**EUR 22740 EN – DG Joint Research Centre, Institute for the Protection and the Security of the Citizen**

Title: The Ultrasonic Densitometer Time Domain Response: Final Report

Authors: W. Rowell, Z. Dzbikowicz, G. Janssens-Maenhout, J. Howell

Luxembourg: Office for Official Publications of the European Communities

2009 – 58 pp. – 21 x 29.7 cm

EUR - Scientific and Technical Research series - ISSN 1018-5593

ISBN 978-92-79-19318-7

doi:10.2788/50704

**Abstract**

Experiments were undertaken to investigate the feasibility of using propagating ultrasonic waves to find the speed of sound and density of solutions contained in opaque, sealed containers. A portable design is proposed which consists of 3 ultrasonic transducers aligned on a single plane along the surface of a tank. The content is then examined by measuring the time it takes for a signal to reflect off the back wall of the tank and return to another transducer. This time domain response approach delivered a very accurate analysis, with a low spread of results. This report demonstrates that by using this technique, very small changes in density can be observed. The final error in the density has been found to be less than 2%, which is adequate to reliably tell the difference between salt and fresh water.





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