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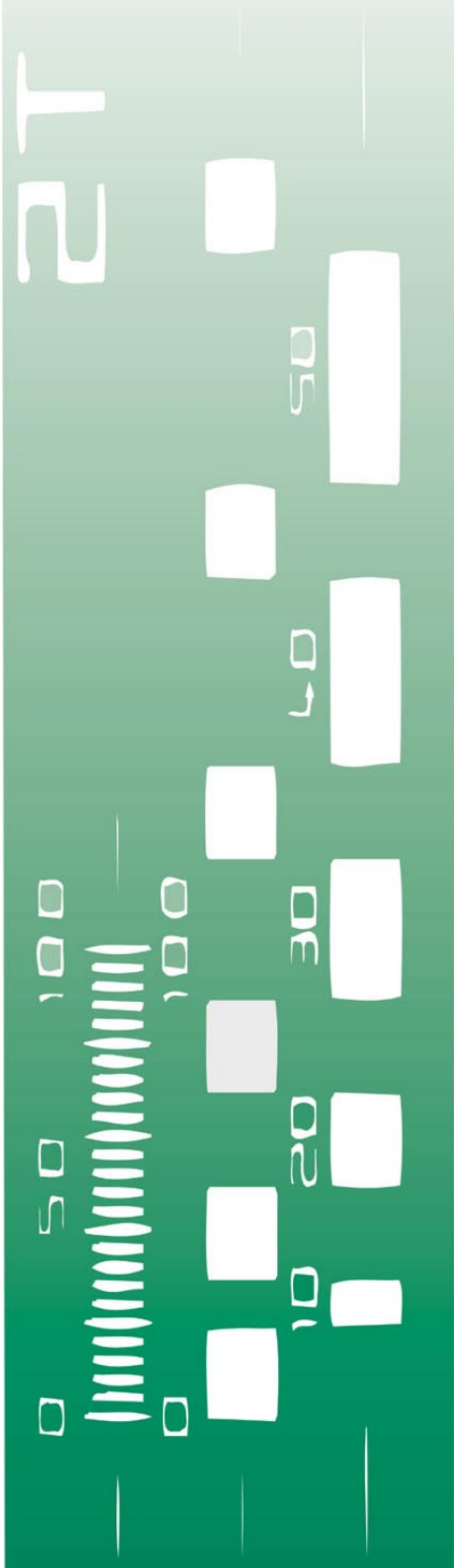
REPORT

Report on the feasibility of establishing a reference cavitating medium

Mark Hodnett, Bajram Zeqiri, Nigel Lee and Pierre Gélat

March 2001



NPL
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Report on the feasibility of establishing a reference cavitating medium

Final project report

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ABSTRACT

This study, carried out by NPL in collaboration with the Institute of Sound & Vibration Research, comprised a literature review, questionnaire survey, and feasibility experiments. The experiments explored the use of 'standard' water samples, specifically "water purified", and "AnalaR water", and also purified water loaded with hydrophobic polystyrene spheres, contained within a reference ultrasonic cleaning vessel, as a reproducible cavitating medium. A novel piezoelectric cavitation sensor was used to assess the cavitation activity.

Results indicated best prospects for reproducible behaviour using the 'standard' water samples. Further work is needed to quantify this reproducibility, by testing other water sample batches and using water containing different levels of trace particulates.

This study was carried out under the UK National Measurement System Programme for Acoustical Metrology, 1999-2001.

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CONTENTS

1 INTRODUCTION

This report describes the results of a **Study of the feasibility of establishing a reference cavitating medium**. Carried out by a consortium consisting of the National Physical Laboratory (NPL), in collaboration with the Institute of Sound and Vibration Research (ISVR), the University of Southampton, the study constitutes project 4.5.3 of the DTI National Measurement System (NMS) Programme for Acoustical Metrology (1998 – 2001). The work described was carried out over the period January – September 2000.

1.1 AIMS AND OBJECTIVES OF PROJECT

The overall aim of the project is to support UK industry by assisting in the development of measurement techniques underpinning high-power ultrasound technology. The growing requirement for measurement standards in this area is described in Annex A.

1.2 BREAKDOWN OF PROJECT ACTIVITIES

The NPL-ISVR team undertook the project in three distinct phases, and the original project tasks are listed:

Phase 1. Information gathering Phase

- Completing a comprehensive literature review relating to cavitating media;
- Developing a questionnaire aimed at key workers worldwide;
- Carrying out a questionnaire-based survey of 19 contact groups;
- Compiling literature and questionnaire-based findings to establish a list of candidate reference cavitating media.

Phase 1 culminated in an Interim Report submitted to the DTI National Measurement System Policy Unit (NMSPU), which summarised the principal findings from the information gathering phase.

Phase 2. Analysis Phase

- Analysing literature and questionnaire responses to establish the most promising candidates;
- Identifying a shortlist of the most promising candidates.

Phase 3. Experimental Phase

- Undertaking a limited number of key experimental studies using those candidate material(s) identified in Phase 2. The NPL reference ultrasonic cleaning vessel was used to undertake this;
- Holding a ground-breaking Technology Transfer meeting at NPL to which key research groups within the UK were invited to use the NPL reference ultrasonic cleaning vessel in conjunction with their speciality sensors (this activity was co-ordinated by ISVR).

This report constitutes the Final Project Report to NMSPU, and provides recommendations on the feasibility of establishing a reference cavitating medium.

2 LAYOUT OF REPORT

The Report constitutes a considerable body of work, and this Section is designed essentially as a ‘road map’ to aid the reader. The current status of cavitation monitoring is presented in Section 3. Here, the point is made, emphasised throughout this report, that the reference cavitating medium cannot be considered in isolation, rather it is one aspect of the overall construct which also comprises the sound field and the particular detection system chosen.

Section 4 describes Phase 1 of the project, which involved the gathering of information, through an email-based questionnaire (Section 4.2) and the completion of worldwide web and literature surveys. The results of these reviews of current capability culminate in the identification of candidate materials upon which some limited experimental measurements were carried out, to assess their potential use as reference media. The Test Bed used for these studies was the reference ultrasonic cleaning vessel being developed at NPL, and this facility, described in detail in Section 6, has played a key role in assessing their potential. The actual results of studies carried out on the candidate materials are described in Section 6.3.

One of the key features of the project has been the holding of a Technology Transfer activity at NPL entitled COMORAC (Comparison Of Measures Of Reference Acoustic Cavitation). ISVR were responsible for arranging the programme of measurements, and it involved a number of leading researchers within the UK being invited to NPL to test their sensors (responding to cavitation observables such acoustic emission, light emission and the production of free radicals) under nominally identical conditions. This event constitutes a ground-breaking activity and ISVR will shortly be publishing a comprehensive report on the study. The current report concludes with a discussion of the results (Section 8) and recommendations to NMSPU relating to the establishment of the reference cavitating medium (Section 9).

3 CURRENT POSITION

Whilst high power ultrasound and ultrasonic cavitation have been applied in a comparatively *ad hoc* fashion to a wide range of processes, for many years, there still remains a lack of capability to characterise these hostile fields. The difficulties in making reliable measurements of cavitation (and hence controlling and optimising cavitation-driven processes) have been identified as a barrier to industrial take-up of the technology (1).

The need for measurement techniques was highlighted in a Study Report funded by NMSPU (1, 2, 3), and as part of this exercise, several facets of the technology were identified as being important. Ultimately, to understand the science and application of ultrasonic cavitation requires the development of an overall 'reference system'. To quote the words of Apfel (4): '*Know thy liquid*; *know thy sound field; know when something happens.' S*uch a system would thus consist of three dependent components:

- the reference sound field (which necessarily includes a reference vessel in which the liquid is contained);
- a reference medium;
- reference detection system to monitor the effects of cavitation.

Progress on these three aspects to date is as follows:

- *The reference sound field/vessel.* A reference ultrasound cleaning vessel is currently being established at the National Physical Laboratory, as part of the NMS UK Acoustical Metrology Programme 1998-2001;
- *The detection system*. The development of a reference detection system is perhaps the most challenging, with a large number of measurement approaches already having been reported (2). It is worth noting that the UK has a community of internationally-recognised experts who cover between them a range of cavitation detection techniques;
- *The reference liquid*. The subject of the current project, the reference liquid is the key link between the other two components. The effectiveness of any reference vessel or detection scheme is very limited without an accompanying reference liquid.

3.1 FUNDAMENTAL CONSIDERATIONS

The phenomenon of acoustic cavitation may be described simply as the activity of small stabilised bubbles in a fluid subjected to an ultrasonic field. High intensity ultrasonic fields may cause these bubbles to collapse through a process known as inertial cavitation, whereupon locally extreme conditions of temperature and pressure may be produced. These conditions can lead to physical, chemical and biological changes in a medium, and so acoustic cavitation has been utilised in processes ranging from healthcare to sonochemistry.

Physically, the resulting experimental situation is complex, and to date, many of the difficulties in understanding and applying ultrasonic cavitation on a useful basis arise from a lack of repeatability and predictability. These may be due to insufficient control over parameters such as the stability of the sound field, but there are several principal factors that are fundamental to the cavitation process:

- the way in which a particular bubble behaves is dependent on the acoustic sound field at that bubble, which depends on the driving sound field, but which is also influenced by the presence of other bubbles, through a variety of mechanisms (shielding, scattering etc.), and also environmental factors, such as the surfaces and structures in the container. A schematic representation of these interrelated factors can be seen in Figure 3.1;

- the type of cavitation activity exhibited by a given bubble (inertial or non-inertial) is critically dependent on the size of that bubble, the acoustic frequency, and other factors such as proximity to vessel surfaces and other bubbles;

- the threshold of cavitation activity itself is hard to predict, unless care is taken in preparation and control of the liquid medium.

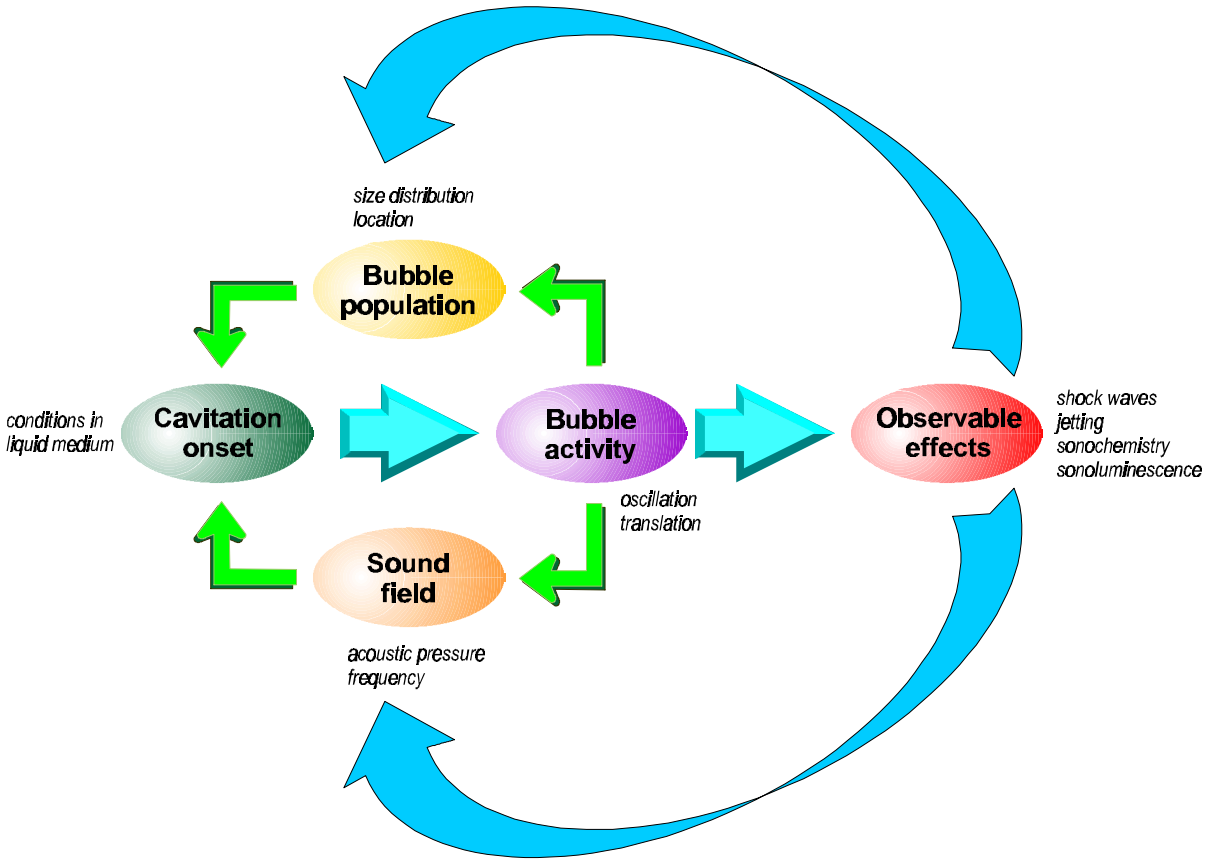

Figure 3.1: A schematic illustration of how the types of cavitation that occur in a bubble population are determined as a result of the local sound field and the bubble population. An observable effect is subsequently produced from the resulting cavitation, and this may have a feedback effect on both the sound field and the bubble population

In studying cavitation activity, the production of the sound field and the nature of the vessel are parameters that may be specified and managed. However, the nature of the liquid can be more difficult to control. As cavitation is a threshold process, its onset will depend on the

weakest link in the medium. This may be a property of the medium itself, or perhaps an aspect associated with it, such as:

- any free and dissolved gas;
- any chemical impurities or 'contaminants';
- the suspended particles or 'motes'.

The importance of the liquid itself is discussed in the following Section.

3.2 CAVITATION INCEPTION

At its simplest, the process of *initiating* acoustic cavitation can be thought of as 'tearing' the liquid apart, forming a cavity which would then fill with liquid vapour and/or any gases dissolved within the liquid. From this, it would be expected that acoustic cavitation would occur only at acoustic pressure amplitudes which generate a tension within the liquid equal to or greater than the tensile strength of the liquid. From the list above, there are several factors that may have a significant influence on the 'tensile strength', rather than just the liquid itself. The tensile strength is therefore more accurately described as that tension which a *liquid system* can support without breaking (cavitating).

3.3 PREDICTION AND EXPERIMENT

Theoretical models of the tensile strength of a liquid consider primarily the attractive forces that cause cohesion between molecules of a pure liquid. In the case of water, the theoretical tensile strength at room temperature is greater than 10^8 Pa. Therefore, the tension required to produce cavitation would reasonably be expected to be of a similar magnitude (5).

Experimental investigations of tensile strength have been carried out by several groups, with a wide range of results. In particular, static tests were performed by Bertholet (6), who performed measurements using a heated glass tube, and measured the tensile strength of his water sample to be around 50 bar. Under highly purified conditions, using a well-filtered sample, Briggs (7) obtained a breaking tension in water of 277 bar, using a centrifuge.

It has been found that non-purified water can be cavitated with stresses of as little as 10^5 Pa (8), which suggests that the presence of weaknesses or cavitation nuclei within the liquid caused significant effects. In addition to the medium properties listed above, other weak links, or cavitation nuclei can be found as gas pockets, located in crevices and cracks in the vessel walls or within free-floating particles within the liquid (9, 10, 11). The passage of ultrasonic waves of sufficient amplitude may cause the gas pockets to either expand out of their crevices, or perhaps produce microbubbles through surface wave activity. In both cases, free-floating nuclei for cavitation may be produced.

Even if microfiltration is employed to remove the majority of particles, this will not completely remove all suitable nuclei for inertial cavitation, which may be generated, for example, by the passage of cosmic rays through the sample (12).

3.4 SUMMARY

In investigating the feasibility of establishing a reference cavitating medium, there are clearly a number of ways of proceeding. The desire to produce the best reference material possible must be considered carefully against factors such as usability and accessibility, as well as compatibility with the variety of sound fields, vessels and detector systems employed by users. In general, however, particular care must be taken with the gas, solid, and impurity content of the reference liquid if it is to have the necessary stability, predictability, and repeatability properties required in a reference cavitation liquid.

4 PHASE 1: INFORMATION GATHERING

The information gathering phase of the project was subdivided into a number of distinct activities. To kick off this phase, NPL and ISVR met to compile a list of contacts and to brainstorm key questions to be posed within the email-based questionnaire. Each activity of Phase 1 will now be described in detail.

4.1 IDENTIFY KEY CONTACTS

The questionnaire provided the key mechanism to facilitate a detailed consultation with representatives of the user community. Due to the importance of the questionnaire in establishing the ultimate success of the project, the decision was made to restrict the user survey to forefront researchers who would be knowledgeable in terms of the state-of-the-art in cavitation monitoring and characterisation.

A list of nineteen contact names was compiled by the project team. In order to maximise the success of the project, the decision was made to approach all of these, even though this was greater than the number identified in the Invitation to Tender. A compilation of email recipients is shown in Appendix C.

4.2 PILOT AND REFINE QUESTIONNAIRE

Questions posed to recipients were designed to establish desirable attributes of a reference cavitating medium. One such attribute is that the onset of cavitation (the threshold) is well defined. Other key parameters to control in a reference fluid include:

- the size of the nuclei;
- the size distribution of the nuclei;
- the concentration of nuclei - are a range of concentrations required?;
- whether it is feasible to base such a material on a solid, with nucleation sites perhaps distributed evenly through a gel-like material;
- if based on a fluid, how the nucleation sites remain evenly distributed throughout the fluid suspension;
- the best way of containing the reference fluid (vial or vessel), as this itself might provide further nucleation sources;
- the need for a range of media with different cavitation thresholds.

A second tier of questions attempted to establish the usefulness of such a material, were it to be developed, in particular:

- who would use the reference fluid?
- how would a reference fluid be applied?
- what desirable attributes and characteristics should the medium have, for example what would constitute the ideal reference cavitating medium?
- what new capability would the availability of the reference cavitating fluid provide? What problems will it overcome?

Also of key importance was whether or not any of the recipients of the questionnaire had actually used any material as a reference cavitation medium. If so, was the work published and what were the benefits and problems of its application?

It was clear that the questionnaire needed to be concise and easy to complete to ensure a significant response rate. After an advanced draft has been prepared, it was piloted and refined using Dr Peter Birkin of the University of Southampton, who was not originally involved in the questionnaire formulation process. The final version of the questionnaire, shown in Appendix B, comprised ten questions and was distributed to recipients by email over the period 29 March to 7 April 2000. The original imposed deadline for replies was April 28, although, to allow more replies to be generated, this was extended to May 12. In total, 14 replies were received, constituting an overall yield of approximately 70%.

4.3 COMPLETE WWW AND LITERATURE SURVEYS

Literature searches were completed using the electronic search facilities at NPL, the aim being to identify those reference cavitation medium candidates whose use had been documented in the open literature. The results of the search are described in detail in Section 5.1. It is an important point to note that, in general, it appears that the vast majority of the literature available on the subject is applicable to the medical ultrasonics field, dealing primarily with MHz frequencies. This is reflected by the tremendous interest in the use of ultrasonic contrast agents for image enhancement. In this application, a medium is seeded with small nucleation sites which act as efficient scatterers of ultrasound. In principle, these could additionally act as cavitation nuclei in a reference cavitation medium, and information was gathered on available contrast agents, primarily through the worldwide web. The proliferation of MHz-based literature should be contrasted with the 40 kHz operating frequency of the NPL reference ultrasonic cleaning bath, which is the primary driver behind this investigation into a reference cavitation medium.

4.4 COMORAC

During the questionnaire activity, UK recipients of the questionnaire were informed of the Technology Transfer meeting to be held at NPL, just prior to the end of the project, under Phase 3. Entitled COMORAC (Comparison of Measurements of Reference Acoustic Cavitation), some details of the rationale are described in Section 7.

4.5 INTERIM REPORT

The results of Phase 1 were summarised in an Interim Report provided to NMSPU on June 1 2000.

5 PHASE 2: ANALYSIS PHASE

5.1 LITERATURE REVIEW

Cavitation nuclei are most often either gas pockets trapped in cracks or crevices on hydrophobic impurities or microbubbles which have been stabilised against dissolution. Clearly, for a given medium, the number of nuclei will play a crucial part in the amount of cavitation activity. Sea water and tap water both contain a wide distribution of bubble sizes (13). In order to improve the reliability of measurements of the acoustic amplitude of accompanying cavitation inception, it is common to seed the liquid with hydrophobic particles including dust, polystyrene spheres (14, 15). In the case of water, the majority of the "naturally" occurring nuclei can be removed by filtration and degassing. During their measurements, Atchley et al. introduced hydrophobic spherical carboxyl latex particles (1μm in diameter) at a uniform rate in water after filtration: latex particles provide large numbers of the same type of hydrophobic impurity in the region of the greatest acoustic intensity during data acquisition, hence improving measurements (14).

Deng et al. studied the effect of the concentration of nucleation agents on cavitation thresholds (15). Experiments were conducted in distilled water seeded with polystyrene particles. Focused transducers with megahertz centre frequencies (2.5 MHz and 4.3 MHz) and a clinical diagnostic ultrasound system (4.0 MHz) were used to generate pulsed ultrasound to induce cavitation. Inertial cavitation thresholds were measured for various concentrations of polystyrene particles. It was observed that the threshold decreased from 2.5 MPa at concentrations of about 10^6 particles / ml to 1.6 MPa at a concentration of about 10^9 particles / ml. For smaller changes in concentration, the effect on the threshold was not as significant. Measurements of the cavitation thresholds were then made in specially developed phantom materials to study the effect of viscosity on the cavitation threshold when surface tension and other mechanical properties of the materials are kept relatively constant. Experimental results showed that the threshold increased with increasing viscosity. Cavitation was also detected in water seeded with polystyrene particles using a clinical ultrasound system at an acoustic pressure of 3.84 MPa.

The work by Deng et al. is important in the context of this project as it involves the study of inertial cavitation in what the authors called a controlled host medium, a different phrase for a reference cavitating medium. They used a medium that was seeded with nuclei whose properties they stated should be: hydrophobic, monodispersed, and of sub-micron size. Their particular size requirements were, however, governed by the megahertz frequencies of application. Although it is not known for sure, it is surmised that the appearance of crevices and cracks on the surface of the particles act as stabilisation sites for gas. If the pressure in the fluid around the particle is reduced due to the passage of an ultrasonic wave, then the gas pockets can be released, so forming a small gas bubble, which under certain conditions may be made to cavitate and collapse violently. The polystyrene particles used by the authors were 0.1 μm diameter hydrophobic polystyrene particles. As commented by the authors, the size of the bubble released from the crevice entrapping it would be much smaller than the resonant size of the bubble (of the order of 1 μm at the frequencies indicated). The host medium which formed the matrix for the particles should also ideally be sufficiently clean such that it is impossible to initiate cavitation in the host at the highest applied acoustic pressure. In this way, any cavitation occurring could definitely be associated with the hydrophobic particles.

Another method of entrapping bubble nuclei was employed by Nyborg et al. (16), where cylindrical bubbles were trapped in the 10 μm pores of hydrophobically treated Nucleopore, polycarbonate filter membranes. The authors studied the spectra generated by bubbles, and also investigated damage to blood platelets.

Most ultrasound contrast agents that are injected into the bloodstream use encapsulated microbubbles as acoustical scatterers in order to increase the reflectivity of the blood (17). However, ultrasound contrast agents, when disrupted by an ultrasound field, still leave an environment rich in nuclei for inertial cavitation (18). For Albunex® in Isoton II solution, the threshold of inertial cavitation is inversely proportional to the concentration (18). Umemura et al. have shown that sonodynamically active cavitation is induced at low ultrasonic intensity in the presence of a certain chemical agents, especially when second harmonic superposition (SHS) on the fundamental is employed (19): Xanthene derivatives are very effective, and Albunex® can enhance *in vitro* and *in vivo* cavitation. SHS enhances cavitation effects through acceleration of the growth of microbubbles, rather than their collapse (19).

Lifshitz et al (20). tested various media for their ability to support cavitation when exposed to shock waves from a lithotripter. Damage inflicted on aluminium foil was used to quantify cavitation. The standard medium used was bicarbonate-free Ringer buffer, pH 7.4 containing (in mM):

- NaCl 122
- KCl 5
- $CaCl_2$ 1.8
- $MgSO_4$ 0.8
- Na-phosphate 0.8
- glucose 5.5
- mannitol 32
- HEPES 20

Viscous aqueous media were tested for their capacity to reduce cavitation all dissolved in bicarbonate-free Ringer:

- methylcellulose 2%
- polyvinylpyrrolidine (PVP) 10%
- dextran 10%
- ficoll 400 20%

each dissolved in bicarbonate-free Ringer.

Oils were also tested:

- castor oil
- mineral oil
- silicone oil

The influence on cavitation of degassing the Ringer buffer was also assessed. It appears that degassing significantly reduces the amount of damage to foil, hence implying that there is less cavitation activity (this could be in terms of less events, and/or lower energy events). Viscous aqueous media proved ineffective at reducing cavitation activity resulting in a high

damage index to the foil (note that this statement appears to contradict that in (15)). Oils generally inhibit cavitation. (20)

In summary, it appears that a number of groups have identified the need for some form of reference medium in the course of their work. From the studies reported above, Lifshitz's group showed that oils inhibit cavitation, which initially might suggest that this would be a possible starting point for investigating a reference material, but clearly, such viscous samples would be somewhat removed from the majority of fluids used in clinical and many industrial applications. It would seem more appropriate to consider the use of aqueous-based media, perhaps containing well-controlled levels of additives.

5.2 QUESTIONNAIRE RESPONSES

5.2.1 Overview

For the most important questions covered in the Questionnaire (see Appendix B), the responses are summarised below. From the responses to Question 1, it was clear that the email recipients covered a wide range of backgrounds, spanning industrialists to medical physics researchers. There is an even balance of those that consider cavitation to be a desirable effect, against those whom are seeking to avoid it.

Question 2 dealt with the recipients' interpretation of the phrase "reference cavitating medium", and what were the key parameters affecting the function of such a medium. Taking the first issue, there was a consensus that a reference cavitation medium should be a material which when applied in any process would generate a known level of (inertial) cavitation. Added to this is the caveat, emphasized earlier in Section 3, that the degree of resultant cavitation should be measured using a method which is readily applied and characterised by an identifiable end-point. Therefore, strictly speaking, the reference cavitating medium was only one component of an overall construct formed by the medium, applied acoustic field, detector and well-defined end-point. To some extent, the identification of the need for an associated detector system was dependent on the area of expertise of the respondent. Chemists interpreted the phrase as describing a mixture of known chemical constituents, which could directly detect the effects of cavitation.

The following is a summary of some of the key observations derived from Question 2:

- the reference cavitating medium is a material which generates the same degree of cavitation, under nominally identical acoustic excitation conditions;
- there may be a need to differentiate between reference media based on a threshold (knowing whether or not cavitation has started) and activity (the 'amount' of cavitation taking place once the onset threshold has been exceeded);

The second part of Question 2, dealt with those key parameters affecting cavitation which would need to be defined in the reference cavitating medium. The following comprises a summary of these:

- dissolved gas content;
- the properties of the medium must be stable with time;
- presence and size distribution of nuclei (predictable density of nucleation sites);

- viscosity;
- ambient pressure;
- in some medical applications, it might be useful for the material to have properties close to that of tissue;
- sound speed;
- density;
- frequency-dependent absorption;
- surface tension;
- temperature;
- nonlinear propagation parameter.

One respondent made the comment that in view of the number of variables involved, there were doubts as to whether any degree of reproducibility in cavitation activity could actually be achieved. Even the smallest variation in impurity level or gas content within a medium could potentially alter the cavitation threshold and the frequency of cavitation events. Two researchers suggested a better approach may be to develop a cavitation monitoring system (based on a type of probe which measures cavitation density), rather than a reference cavitating medium.

An important practical observation made was that the reference cavitating medium must be widely available i.e. it should either be easy to manufacture, or be readily obtainable from a well-defined source.

Of the respondents who have used any sort of reference medium (Questions 3 and 4), the majority indicated that this was aqueous-based, in some cases using well-characterised chemical additives. Tap-water, distilled water and degassed-water had all been used. One researcher commented that tap (aerated) water could be used to produce a 'worst-case' cavitational estimate, and was surprisingly robust for local use. They added, however, that well-known regional variations in tap-water obviously precluded its use as a global standard. Those materials which had been used had been applied when comparing system performance, or when trying to reproduce cavitation conditions in the process of investigating some other variable, for example lithotripter performance.

The overwhelming majority of respondents agreed that there is a definite need for a reference cavitating medium, and that the need is immediate (Question 5). Specifically, the need to intercompare cleaning processes and acoustic sources was identified as the principal driver behind this need; even to provide the capability to 'calibrate' cavitation-producing systems. It was suggested that the hitherto lack of a reference cavitating medium (alongside a reference vessel and detection technique) had perhaps inhibited the take-up of sonochemistry in industry.

A wide range of different and new capabilities were indicated if such a medium were to become available (Question 7). In particular, the possibility of developing a reference cavitation test object, suitable for assessing the likelihood of diagnostic ultrasound scanners to cause cavitation *in vivo*, would be facilitated by the availability of such a medium. The area of assessing the feasibility of industrial scale-up was also suggested. In general, the

theme of ‘enabling comparison’ was highly prevalent, indicating a widely-held view of researchers wanting to establish best practice in a variety of applications.

The question of how to realise a reference medium (Question 8) provided an interesting range of responses. Most thought that due to its ready availability and (in some cases) apparent efficacy, a water-based medium would be most appropriate. Some respondents identified chemical mixtures that would provide suitable media - these would work along the lines of providing steady quantities of free radicals, which could then be detected by a variety of techniques. Also suggested was an aqueous base containing a well-known concentration and size distribution of nucleation sites, such as contrast agents, polymer beads, or even radioactive sources. Water was identified also because the majority of cavitation-driven processes are aqueous. A well-defined nucleation density could potentially be generated using small beads made of hydrophobic materials (solid beads) or hydrophilic materials with crevices present for stabilizing gas, or even hollow spheres containing liquids or gases.

It was considered that the reference medium may necessarily be application-specific (Question 9), although two respondents suggested that if it was feasible, a universally-applicable material that responded to different situations in different ways would be attractive. One author also suggested that the shelf-life was also important, and that perhaps a standard material could be available with different ‘ratings’, perhaps depending on viscosity and concentration of included nuclei.

From the responses to the questionnaire, it was concluded that the most important aspects of any reference medium were:

- ready availability;
- ease of use;
- repeatability;
- it should be aqueous in origin;
- it should produce an easily-identifiable effect.

5.3 SUMMARY ANALYSIS

From the literature review and an assessment of the responses to the questionnaire, the aim was to identify those candidate materials which were to be studied briefly within Phase 3 of the project. However, first, it is valuable to consider, in rather generic terms, the possibilities for these media. The choice of the basic liquid needs consideration. From the responses, most respondents cited an aqueous medium, and this has the advantage that many industrial systems use it. Furthermore, the bubble dynamics within aqueous media are probably better understood than in any other liquid. The main driver for the current project is ultrasonic cleaning and here, an increasing number of systems employ aqueous-based media due to the need to move away from chemicals amid potential environmental implications.

Having accepted that the background medium should be water-based, a decision has to be made on how to treat the indigenous particles, as these will have a significant effect on cavitation inception, and perhaps also on the level of cavitation once the onset threshold has been passed. The comment has been made previously that tap water has been shown to be saturated with nucleation sites, and for local use, can itself be considered as a reference

medium. However, there is the general belief that, unless steps are taken to remove the effects of random nucleation sites, then these may produce a detrimental effect on cavitation reproducibility. Such an effect might be removed in two ways:

1) by employing a filtration / degassing system to reduce the level of these impurities as low as possible, as is used in the NPL water management system. This will then leave the onset of cavitation dependent on the impurities which have not been removed (i.e. as the tensile strength of the liquid is approached, the medium is subjected to relatively large forces: the onset of cavitation might be likened to the breaking of a chain and many of the 'weaker links' have simply been removed; having done so, the onset of cavitation depends on the breakage of one of the remaining links, so that in fact, repeatability may have been *reduced*). Ensuring that a significant, but reproducible, amount of particulate remains such that the tensile strength of the liquid has not been altered radically might reduce this effect. A second point relating to the removal of impurities is that the medium is likely to have fewer nucleation sites than any material which is used with ultrasonic cleaning vessels, so that the cavitation activity might be similarly very different. If less cavitation is produced then the cavitation field might be misleadingly simple. Whilst this might be desirable for a standard, it may become removed from the practical industrial cleaning bath;

2) by flooding the liquid with 'standard' nucleation sites – so minimising the effect of random nucleation. Several options for these sites exist: contrast agents; radiation; electrolysis; and solid particles. All have advantages and disadvantages. For example, the stability and acoustic properties of contrast agents can be compromised if they are used out of the intended host medium, or even in an inappropriate dilution; and once cavitation has begun, the very species exploited in the gas and shell of the agent to stabilise its properties become contaminants in the cavitation detritus. One drawback of this 'seeding' approach is that a great deal of cavitation activity may result, enhancing the problematic effects of multiple bubble interactions.

One compromise might be to combine both techniques, removing the random nucleation sites and adding some 'standard' ones. Some care needs to be taken to ensure that the resulting level of cavitation resembles that found in industry, which raises the issue of which detector systems will be incorporated into the reference system. The detector system used for the candidate material studies is described in Section 6.2.2, and is based on the detection of acoustic emissions generated by cavitation.

5.4 SUMMARY OF CANDIDATE MATERIALS

It was decided that two basic types of materials would be studied:

- two different types of 'standard' water would be procured and investigated;
- a source of nucleation sites would be investigated, and a small quantity procured. From the literature, along with the responses to the questionnaire, it was decided that the additives should be based on hydrophobic polystyrene spheres.

In deciding on the types of media to be studied, it was considered beneficial to assess materials that were available commercially. The NPL water management system could have been used to produce 'standard' samples of water of a specific filtration level and dissolved gas content, but

this would have had lesser general relevance. A consequence of this philosophy was that the water procured "off the shelf" would be uniformly saturated with gas. Two standard samples of water, "water purified" and "AnalaR water" were procured from the chemical supplier, BDH Ltd, and their details are given in Table 5.1.

To obtain a suitable source of uniform hydrophobic microspheres, several searches were carried out using the Internet. The particular material chosen was selected predominantly because it has a density similar to water (relative density 1.061), and would thus stay suspended in water for relatively long periods.

Table 5.1: Summary of the candidate cavitation reference materials purchased

Material	**Specification**	**Source**
"Water purified" **Product code: 307298J**	Maximum specific conductance 5 μS cm^{-1} Residue on evaporation at 110 °C (particulate level) = 2 ppm Complies with BS 3978:1987 (ISO 3696:1987) – Water for laboratory use, Grade 3	BDH Merck Limited, Merck House, Poole, Dorset
"AnalaR Water" **Product code: 102928H**	Maximum specific conductance 5 μS cm^{-1} Residue on evaporation at 110 °C (particulate level) = 1 ppm Complies with BS 3978:1987 (ISO 3696:1987) – Water for laboratory use, Grade 2	BDH Merck Limited, Merck House, Poole, Dorset
Hydrophobic polystyrene spheres **Product number: PS07N/001406**	Uniform microspheres, 10.0 μm diameter, Polymer: Polystyrene (S/0.1%DVB) 10% solids, Inv L92111C. 0.5g vial.	Bangs Laboratories, Inc 9025 Technology Drive, Fishers, IN 40638 – 2886, USA

The compilation of the table of candidate materials was based on the outcome of the main questionnaire responses, and it should be noted that it represents a departure from the original project plan, in which the intention was to formulate a scoring system. This would have been used to evaluate the range of possible options that would arise as a result of the information gathering phase. This slight change of direction was due to the nature of the questionnaire responses, which showed that although high quality data was obtained from the respondents, a comparatively limited amount of information was yielded on suitable materials, either that the

respondents had used themselves, or that they considered would be suitable for use as a reference medium. The overriding response appeared to be that aqueous media would be most suitable. From this, a small number of possible realisations were discussed, and it was considered that a comprehensive scoring system technique to evaluate their strengths and weaknesses would be inappropriate. It was judged that the project would benefit more from effort instead being directed towards studying the identified candidate media experimentally.

6 PHASE 3: EXPERIMENTAL STUDIES

6.1 THE REFERENCE ULTRASONIC CLEANING VESSEL

The Study Report (1) recommended that a reference cleaning vessel be established, and this is in the process of being developed within the current NMS Acoustics Programme 1998-2001 at NPL (Project 4.5.1). Progress in establishing the infrastructure supporting the reference ultrasonic cleaning vessel has recently been summarised (21).

Some care needs to be exercised when interpreting the term 'reference' in relation to the ultrasonic cleaning vessel. It should be understood as representing the ultrasound cleaning vessel in combination with the sensor scanning, environmental monitoring and characterisation infrastructure built around it. It was considered important that the ultrasound source itself should be representative of a commercially-available system, for the results of studies to be appropriate at the industrial user level. As the reference cleaning vessel played an important role in the proposed project, allowing testing of the reference cavitation medium candidates (this section) and providing the test-bed for COMORAC (Section 7), it will be described briefly.

Its centrepiece comprises a Branson 1 kW ultrasonic cleaning bath of internal dimensions 360 mm long, 230 mm wide and 305 mm deep. It has 12 transducers distributed evenly over the bottom surface, and the modified control and drive system excites these at a frequency of 40 kHz. The cleaning vessel is shown in Figure 6.1.

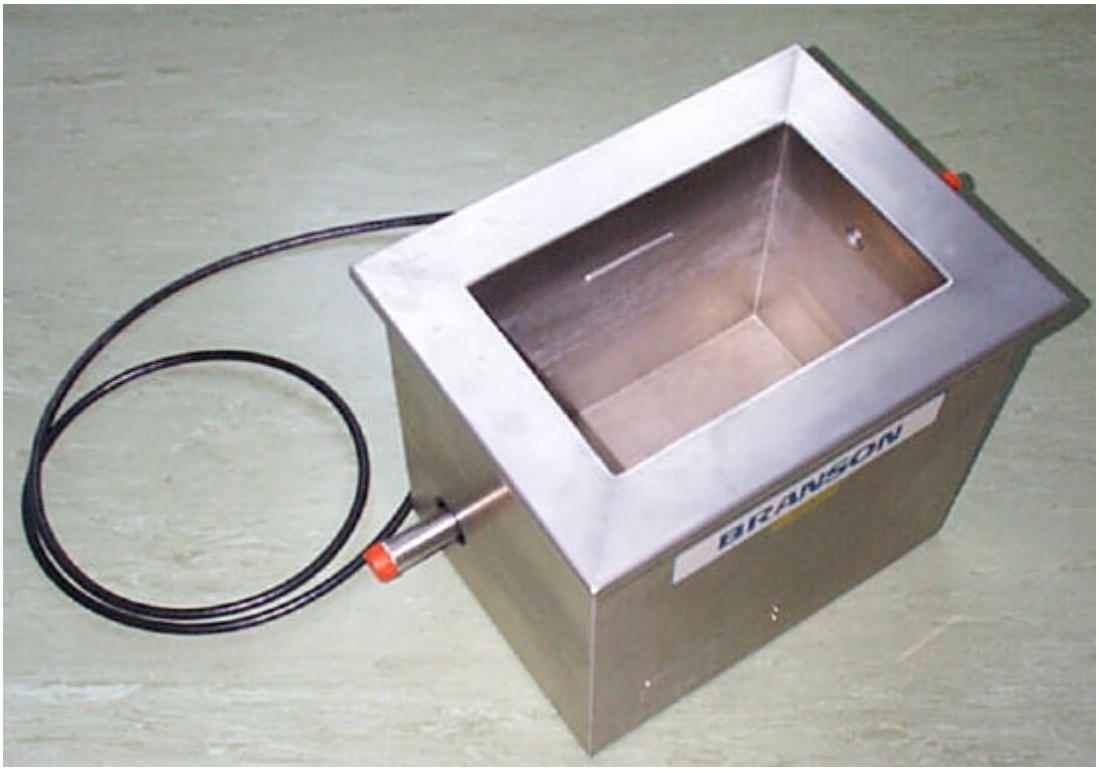

Figure 6.1: The Branson 1 kW ultrasonic cleaning vessel

In establishing such a reference vessel, the aim is to be able to reproduce the acoustic environment (defined by the driving acoustic field) giving rise to cavitation, such that measurement sensors (e.g. acoustical, thermal and optical) as well as those cavitation-related

processes (e. g. sonochemistry) may be tested and analysed under known field conditions. To this end, a number of factors have initially been identified as being important to monitor and control (21):

Environmental and mechanical:

- water temperature;
- water height;
- particulate size distribution and concentration;
- levels of dissolved gas;
- levels of dissolved ions;
- surfactant concentration.

Electrical (and hence acoustical):

- transducer drive voltage and therefore acoustic pressure amplitude;
- transducer drive frequency.

To facilitate the environmental control, a dedicated water management system provides the necessary water quality control and monitoring capabilities. Additionally, the reference cleaning vessel is located within an environmental enclosure, which minimises manual intervention and thus restricts the amount of 'atmospheric contamination'. Modifications made to the Branson drive system facilitate the control of the acoustic output of the cleaning vessel, enabling it to be varied over the range 5% to 95% of the maximum power of the vessel.

A three-axis positioning system is located over the cleaning vessel, allowing precise location of measurement sensors (±0.005 mm) and the ability to scan through the acoustic field whilst data is being captured in time and frequency domains. The scanning and data acquisition facilities are under computer control. The cleaning vessel along with some of its supporting infrastructure is shown in Figure 6.2.

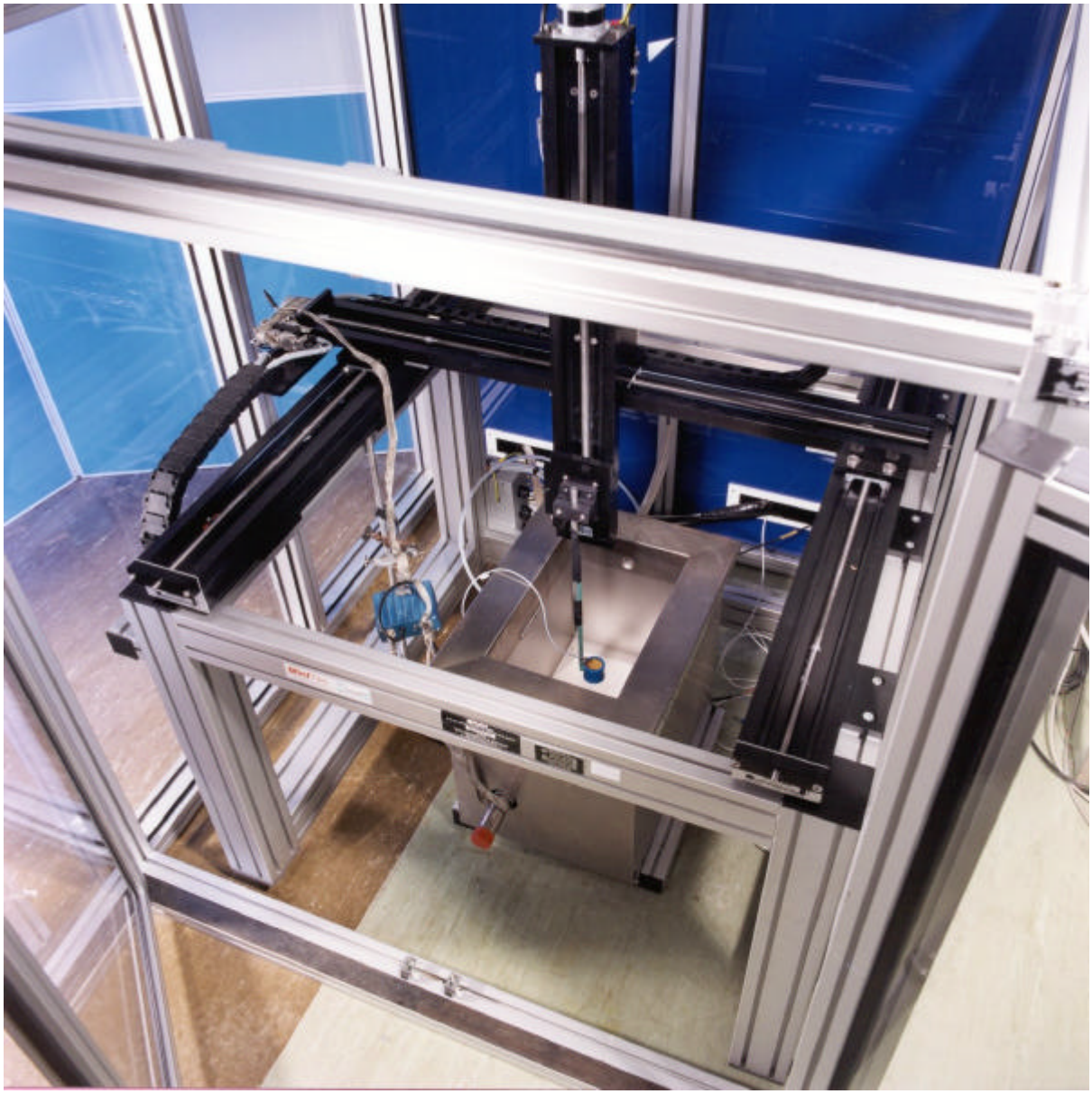

Figure 6.2: The reference ultrasonic cleaning facility, showing the Branson 1 kW ultrasonic cleaning vessel positioned within the sound proofed enclosure, with the three-axis positioning system located around it. An NPL cavitation sensor (discussed in Section 6.2.2) is shown positioned within the cleaning vessel

6.2 PHASE 3: EXPERIMENTAL STUDIES – EQUIPMENT AND METHODOLOGY

In addition to the literature and questionnaire surveys, and the critical analysis of the findings, the work carried out under this project comprised a limited set of measurements on the candidate materials identified in Table 5.1. The consortium considered that such an investigation would be crucial in feeding into the recommendations of the overall study.

6.2.1 Experimental set up

Central to the experimental facilities utilised at NPL in the experimental studies is the reference cleaning vessel described immediately above. This is being established as a reference source of cavitation at NPL, and results to date have shown that cavitation conditions are reproducible to around ±13% (21). This repeatability has allowed the reference vessel to be used as a "test bed" against which novel sensors are being developed, within a parallel-running NPL Strategic Research Project. The cavitation sensor developed at NPL was used to assess the reproducibility of the cavitation field generated by the candidate reference materials. It will therefore be described in more detail. The sensor is covered under a UK Patent: Priority Application No. 9921982.6.

6.2.2 The novel cavitation sensor

The NPL Strategic Research Project has investigated the development of sensor technologies that may be used in the assessment of the cavitation activity produced by ultrasonic cleaning vessels. The project has included extensive literature searches and theoretical modelling, all targeted towards formulating a prototype sensor. There are a number of features of these sensors, which are described below:

- they are based on a passive acoustic detection technique and utilise a thin (110 μm) film of *pvdf*, procured from a commercially available source and delivered with screen printed silver electrodes;
- the bandwidth of the *pvdf* film allows measurements of the bubble acoustic emissions to frequencies beyond 10 MHz;

(Note: within the reference vessel, acoustic signals beyond 13 MHz have been observed through the use of a reference device – a Marconi bilaminar membrane hydrophone, which has a resonance at 20 MHz).

- the *pvdf* material is formed into a right-circular cylinder which is hollow and is therefore open to the (aqueous) fluid medium in the vessel;
- the cylindrical structure of the sensor is designed so that it behaves as a focused receiver such that MHz acoustic signals originating from the fluid medium contained within the sensor are monitored, providing the sensor with a degree of spatial resolution;
- to ensure bubble-events occurring outside of the cylinder do not contribute to the device output, its external surface is coated with a 'cavitation shield' which has been produced through a specialist materials development programme. Based on a polyurethane rubber material, the 4 mm thick layer is highly attenuating to MHz acoustic signals (attenuation coefficient >70 dB cm^{-1} MHz^{-1}) but is designed to be minimally perturbing to the driving frequency of the cleaning vessel, 40 kHz.

(Note: the materials have been characterised in terms of their acoustic properties over the frequency range 20 kHz to 3 MHz. The two requirements given above, of high MHz and low kHz transmission loss, are mutually exclusive and a trade-off needs to be made. The acoustic impedance of the material has been designed to match that of water at 40 kHz, as the propagation speed in the material is highly dependent on frequency).

A second stage prototyping of the sensors is currently underway. The cylinders that make up the cavitation sensors are of diameter 30 mm and height 30 mm. They are positioned within the cleaning vessel using a thin rod made of syntactic epoxy, allowing the devices to be scanned through the tank, in principle deriving a map of the 'cavitation activity'. A schematic representation of the sensor, together with a photograph of its construction can be seen in Figure 6.3.

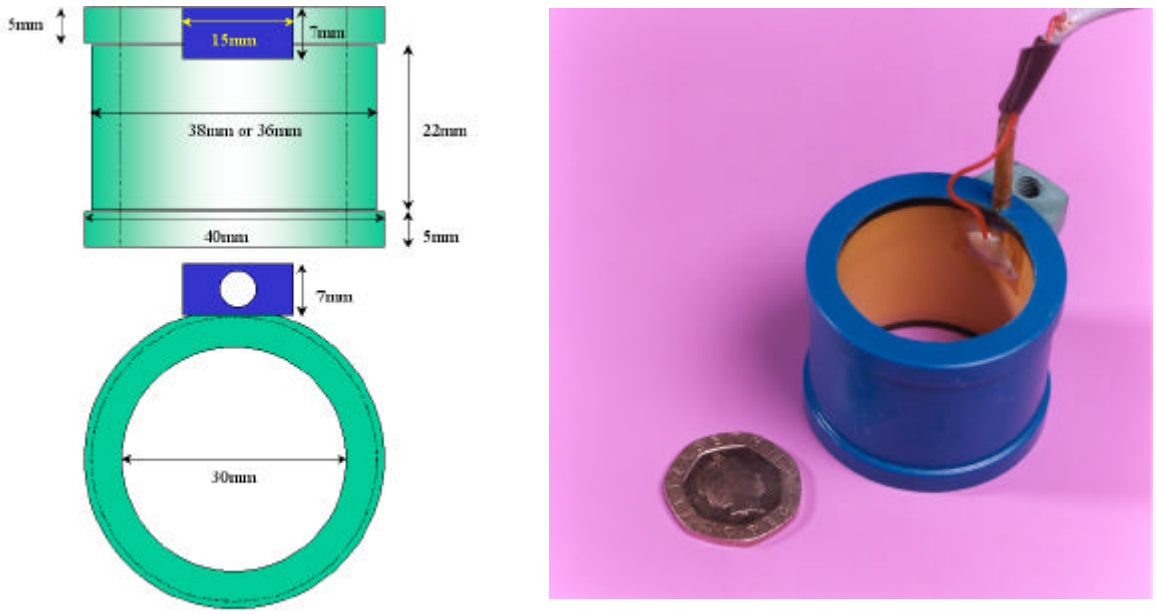

Figure 6.3: Schematic representation and actual construction of NPL Prototype Cavitation Sensor (PCS)

The device will be referred to from here onwards as the Prototype Cavitation Sensor (PCS). Although the PCS is very much in the 'proof-of-concept' stage, a large number of promising results had been achieved in aqueous media prior to the experimental phase of the current project, and consequently, it was considered appropriate that the sensor should be used in the tests of cavitation activity produced using the reference vessel and the candidate media.

6.2.3 Acoustic emission

It is considered useful to provide here a brief description of the basis of passive acoustic cavitation detection. A full description of the bubble dynamics principles responsible for the acoustic signal emitted by cavitation bubbles may be found elsewhere (22). A brief synopsis is provided here of the detection of signals produced by cavitating bubbles. Acoustic emission techniques have been used by a number of authors (23, 2) and are based fundamentally on the use of piezoelectric and other devices that respond to the acoustic signals produced by oscillating and collapsing bubbles. A brief discussion of acoustic emission measurements is given here. Figure 6.4 shows a typical acoustic spectrum acquired in a 40 kHz high power ultrasound field, producing inertial cavitation.

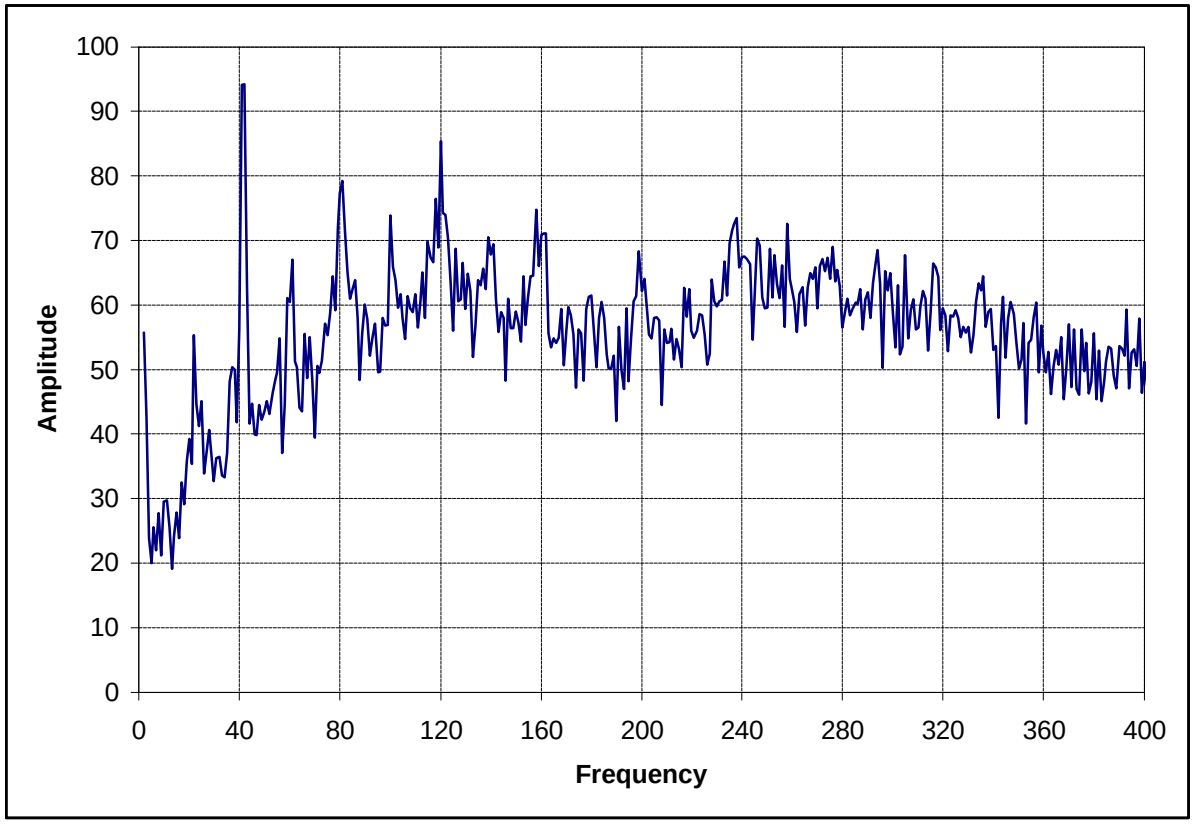

Figure 6.4: Typical acoustic spectrum produced by a cavitating bubble field

The figure shows a range of signals that may be used as indicators of cavitation activity:

- The fundamental frequency component at approximately 40 kHz – in the main, this is produced by the direct driving field, with further contributions from linear bubble oscillations (24);

- Harmonics of the fundamental, at integer multiple frequencies nf_0 (80 kHz, 120 kHz and so on) – these are the result of nonlinear bubble oscillations (25), and in some cases, acoustic signals produced by the transducers due to distortion in the drive waveform;

- Subharmonics of the fundamental, f_0/n; here the 20 kHz signal can be seen – these are sometimes interpreted as indicators of the onset of inertial cavitation activity (26), and can occur when a bubble of twice the resonant size is excited (27), or when a bubble takes two acoustic periods to reach critical radius before collapse (28);

- Ultraharmonics of the fundamental, mf_0/n, where m and n are integers; (60 kHz, 100 kHz and so on) – these are bubble events of a similar type to those producing subharmonics (29);

- Broadband signal – the 'noise' at all frequencies, produced by the shock waves emitted upon bubble collapse, and by collapsing bubbles of a wide range of sizes. This has been used as an indicator of inertial cavitation activity (23), and can extend to frequencies well into the MHz range, even for acoustic fields which are driven at tens of kHz.

The last of the listed parameters, the broadband 'noise' signal, is used in this report as a measure of inertial cavitation taking place during the assessment of the candidate materials.

Hydrophone measurements of cavitating fields may also be sensitive to electrical signals produced by the transducers themselves: these will tend to be at the fundamental frequency, and at the integer harmonics, and will thus be superimposed on the 'true' acoustic spectrum. In the case of Figure 6.4, there appears to be structure in the overall noise envelope: this is due to the frequency response of the particular hydrophone used.

The reference cleaning vessel generates an acoustic environment that is sufficient to produce at any instant a large number of bubble events, which will be occurring throughout the volume of the vessel. Measurements made using a point sensor will actually be a summation of contributions from cavitation events over a large volume, and will also be influenced by reflections from the walls and the water surface, and electrical pick-up. Phenomena such as bubble shielding will further affect the data obtained. This location uncertainty of the signal origin is largely overcome by the PCS, as it detects only high frequency cavitation signals occurring within the specific volume formed by the piezoelectric cylinder. In making measurements, the sensor output is connected to two devices:

- a spectrum analyser, with the captured data then analysed in the frequency domain. The particular parameter of interest is the broadband signal, evaluated over the range 1 to 5 MHz;
- a root-mean-square (*rms*) voltmeter, which effectively provides a monitor of the direct 40 kHz driving field: even though the meter generally used responds to signals in the range 0-10 MHz, the 40 kHz fundamental is generally 20 - 30 dB higher in level than the majority of other signals, and so dominates the measured value.

The acquired spectra are digitised and imported into Microsoft Excel, where they are converted to voltage amplitude spectra. The broadband power is then calculated by summing the squared voltage contributions at each frequency over the range 1 MHz – 5 MHz, to produce a figure with units of V^2 Hz. This is carried out for each drive setting, and results are plotted as a function of this.

6.2.4 Experimental protocol

It was considered to be important that all of the experimental work carried out by NPL in the project (both in the candidate material studies and the COMORAC Technology Transfer Activity) should be performed on as an identical a basis as possible. To this end, an experimental protocol was drawn up. A version of this (produced for COMORAC) can be found in full in Appendix D, and in essence it comprises precise details on equipment set up, sensor deployment, media preparation and instrument control settings.

For all tests performed on the candidate materials, the sensor was deployed in a vertical configuration (see Figures 6.2 and 6.3), and was kept at a fixed position in the reference vessel. This position was readily reproducible, to ± 0.3 cm, using the positioning system around the vessel. It was felt that a full assessment of the spatial distribution of the cavitation activity in the reference media would be beyond the scope of the current project. The PCS was thus located at the centre of the vessel in the horizontal plane, with the centre

of the device 9 cm below the water surface. In all cases, the vessel was filled with liquid to the manufacturer's indicator, providing a medium depth of 22 cm.

6.3 CANDIDATE MATERIAL STUDIES 1: STANDARD WATER SAMPLES

Once the candidate materials had been decided upon (see Table 5.1), two 25 litre bottles of each of the two types of 'standard' water were procured. The reference vessel holds 18.1 litres of liquid when filled to the indicating line, and so the procured quantity provided enough for two independent runs on each type of water, which was considered to provide a reasonable idea of the likely reproducibility. For all studies, the cleaning vessel was thoroughly rinsed with filtered water prior to being filled with the sample under test. The PCS was pre-soaked in water containing a small amount of washing-up liquid before measurements were made, to aid wetting. This pre-conditioning was normally carried out for 30 minutes. The PCS was then attached to the positioning system, and lowered into the reference vessel to the position described above. In most cases, the temperature (T) and level of dissolved oxygen (DO_2) were measured using appropriate meters (see Appendix D), before and after each full run. The procedure for measurements was very similar to that described in the COMORAC protocol (Appendix D), with the exception of the medium preparation.

6.3.1 Results – "water purified"

The two separate, nominally-identical "water purified" bottles are described as sample 1 and sample 2. Experimental runs were first carried out on sample 1, with sensor PCS-11 located at the reference position. Measurements were made as a function of vessel drive setting, with three types of readings taken:

- the DC voltage produced by the vessel drive unit interface card, related to the electrical power supplied to the transducers;
- the acoustic spectrum, acquired in the range 1 – 5 MHz, and averaged over 40 sweeps;
- the *rms* voltage produced by the sensor.

The acquired spectrum was then analysed as described in Section 6.2.3, producing a value for broadband power in the range 1 MHz to 5 MHz. Data was acquired for ascending and descending powers, to provide an assessment of any system hysteresis. Figure 6.5 shows the amplitude spectra for the full range of powers, with the numerical results from the first pair of runs tabulated in Table 6.1. Figure 6.6 shows the broadband signal as a function of DC voltage, and Figure 6.7 the *rms,* again as a function of ascending DC voltage. Figures 6.8 – 6.10 show the analogous graphs for the descending DC voltage set.

Table 6.1: PCS-11, "water purified" sample 1

Initial conditions: DO_2 level = 8.69 ppm, T = 21.3 °C			
Nominal % power level	DC voltage (V)	*rms* (mV)	Broadband power (/ 1000 V^2 Hz)
5	1.009	133	0.20
10	1.389	145	0.29
15	2.131	171	0.75
20	2.85	178	1.24
40	4.67	184	2.14
65	7.18	175	2.33
85	9.42	165	2.36
95	10.17	159	1.59
Middle conditions: DO_2 level = 8.57 ppm, T = 22.3 °C			
95	10.03	164	2.57
85	9.39	169	2.06
65	7.25	179	1.90
40	4.69	184	1.66
20	2.8	161	0.96
15	1.85	146	0.53
10	1.459	143	0.42
5	0.999	134	0.28
Final conditions: DO_2 level = 8.51 ppm, T = 23.0 °C			

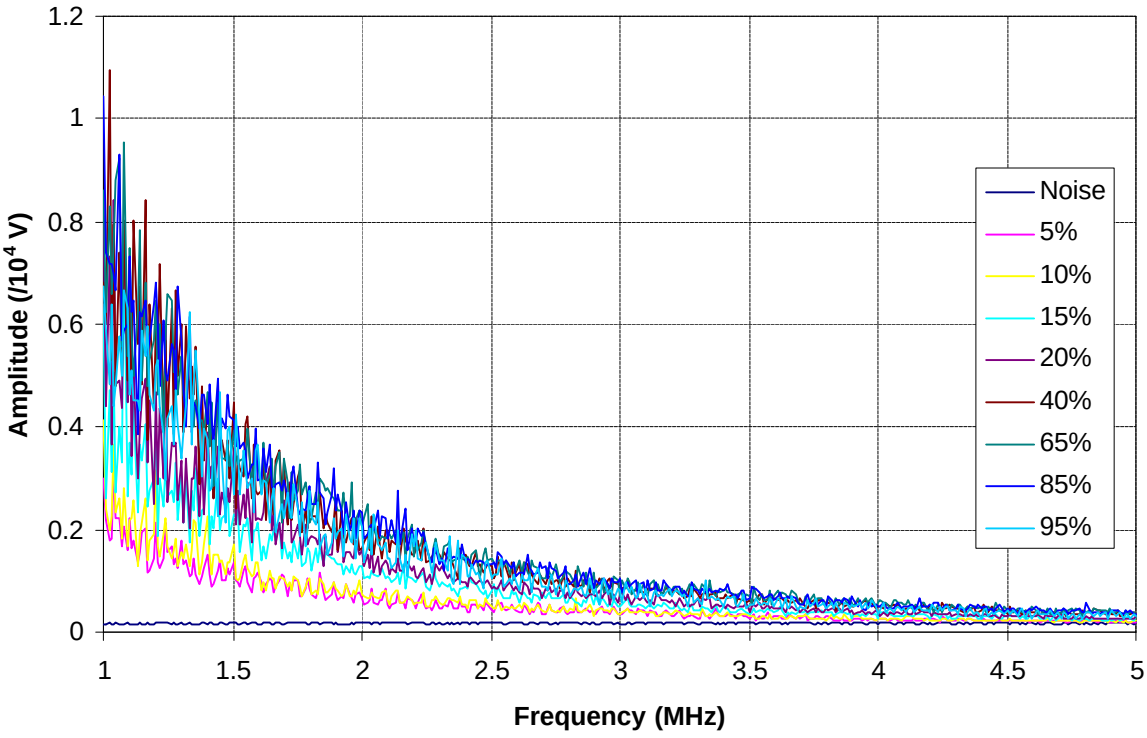

Figure 6.5: Acoustic spectra acquired as a function of nominal drive setting using PCS-11, "water purified" sample 1, ascending data

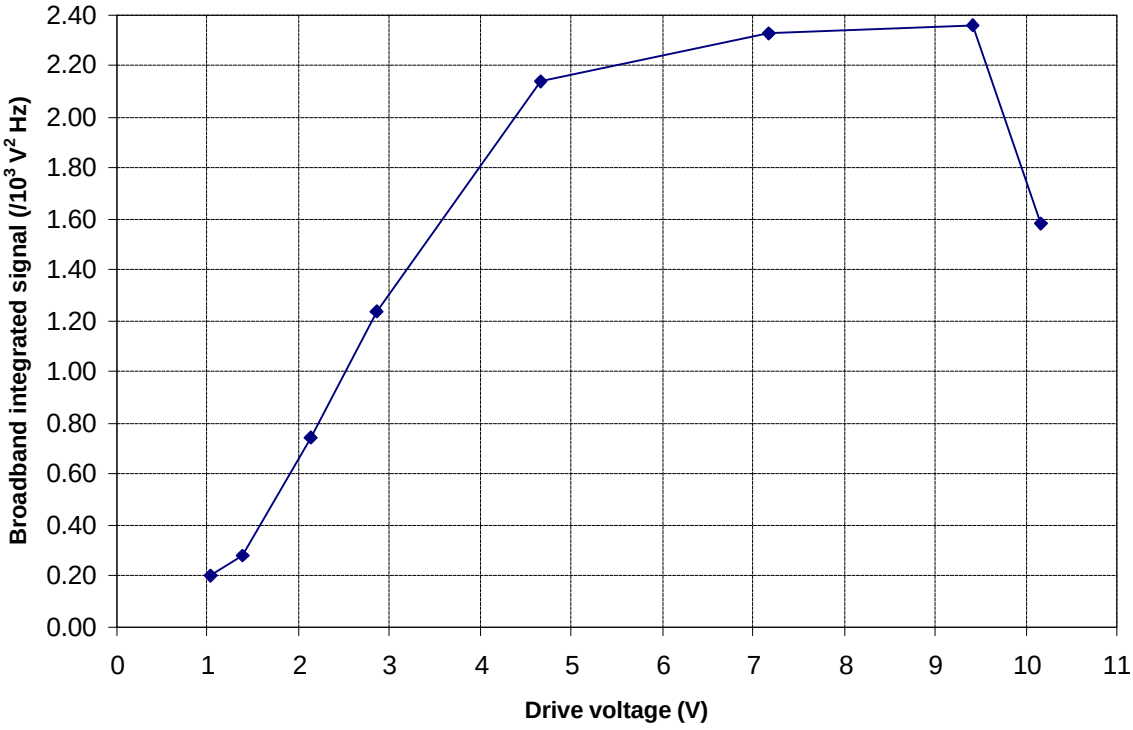

Figure 6.6: Broadband integrated signal as a function of actual DC voltage using PCS-11, "water purified" sample 1, ascending data

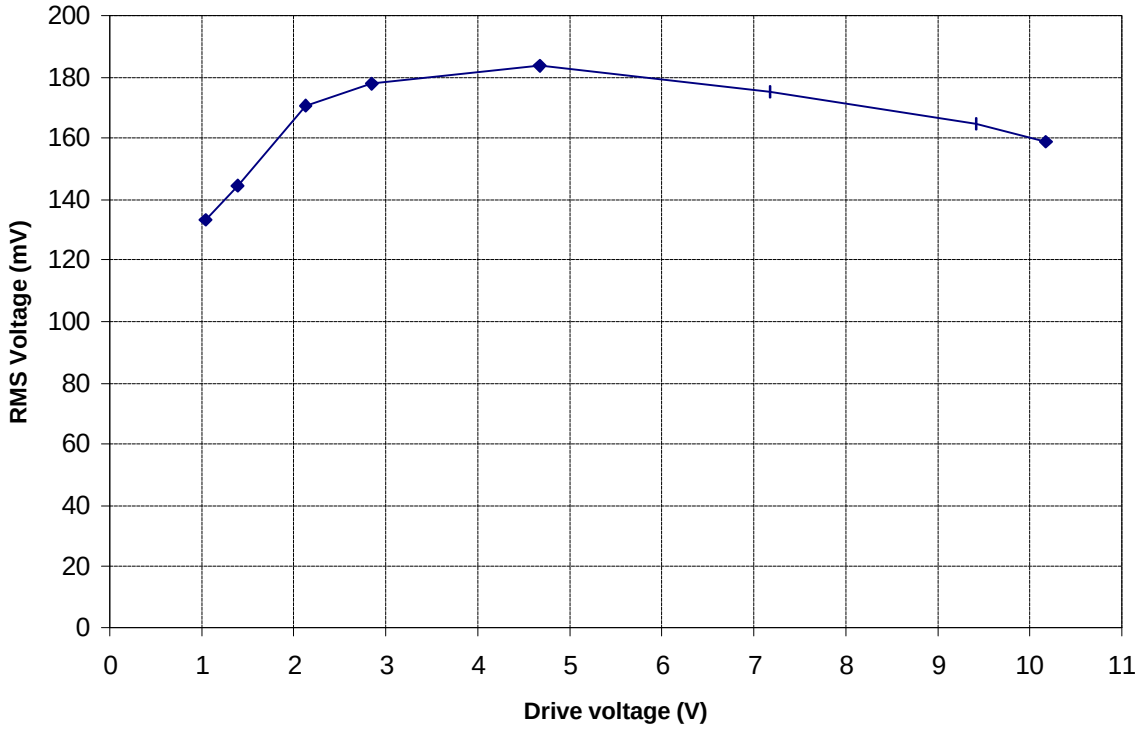

Figure 6.7: *rms* voltage as a function of actual DC voltage using PCS-11, "water purified" sample 1, ascending data

Figure 6.5 shows that the acoustic spectra increase in level, both in terms of the broadband envelope and the harmonic levels. The presence of the harmonics in the spectra may indicate that there is significant stable bubble oscillation occurring, implying that a high number of cavitation nuclei are available. This would probably be expected for the medium in this case, as there is a substantial amount of dissolved oxygen, which will 'seed' cavitation. The increase in the broadband activity, manifested by the area under the spectra increasing as the vessel power is increased, indicates an increase in inertial cavitation activity.

The spectra acquired for the three highest settings are difficult to distinguish on the amplitude plot, suggesting that the extent of cavitation activity reaches a limit. Examining the broadband signal derived from the spectra (Figure 6.6) shows that the level of inertial cavitation activity appears to start increasing rapidly, before evening out and reaching a peak level for the nominal 85% drive setting, and then decreasing again at the highest drive level. This confirms that the cavitation activity detected by the sensor can reach a peak for a lower power level than the maximum available. There are thought to be two main processes that may account for this:

- cavitation shielding, in which bubbles in the medium scatter the direct ultrasound field and the acoustic cavitation emissions, meaning that the sensor detects an apparently lower level of acoustic activity – this is likely to occur readily for the 'standard'-type water samples, as the levels of dissolved oxygen approach those of potable water. Although the turnover in the trend of *rms* as a function of drive voltage (Figure 6.7) might suggest that the direct 40 kHz field may be affected in this way, such a trend is also seen for other types of water sample;

- energy re-distribution into higher frequencies – cavitation is a nonlinear process, and at higher drive settings, energy may be moved from lower frequencies into higher orders outside of the acquisition bandwidth, meaning that the energy measured in the limited envelope (1-5 MHz) can level off, an effect which would be most pronounced at the highest drive levels.

Generally, from the many other measurements made using the PCS sensors and conventional hydrophones, data acquired at the lower powers are considered to be more reproducible, where cavitation conditions are less intense.

Figures 6.8 – 6.10 show the analogous graphs for the descending DC voltage set.

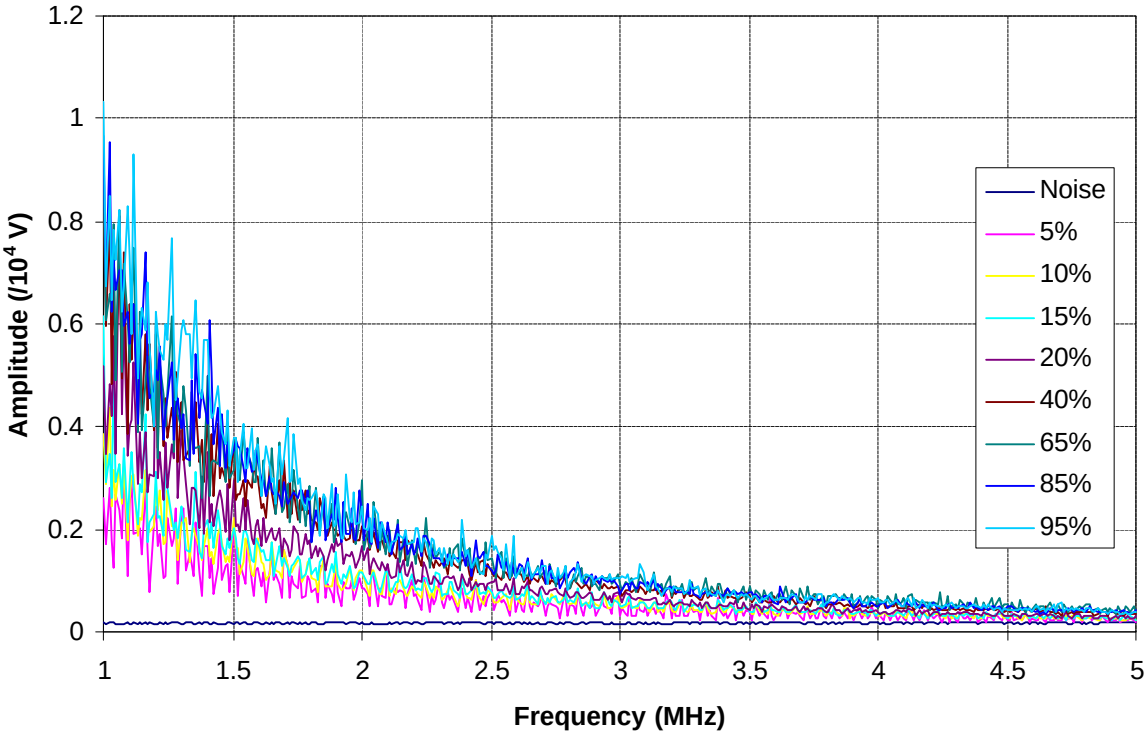

Figure 6.8: Acoustic spectra acquired as a function of nominal drive setting using PCS-11, "water purified" sample 1, descending data

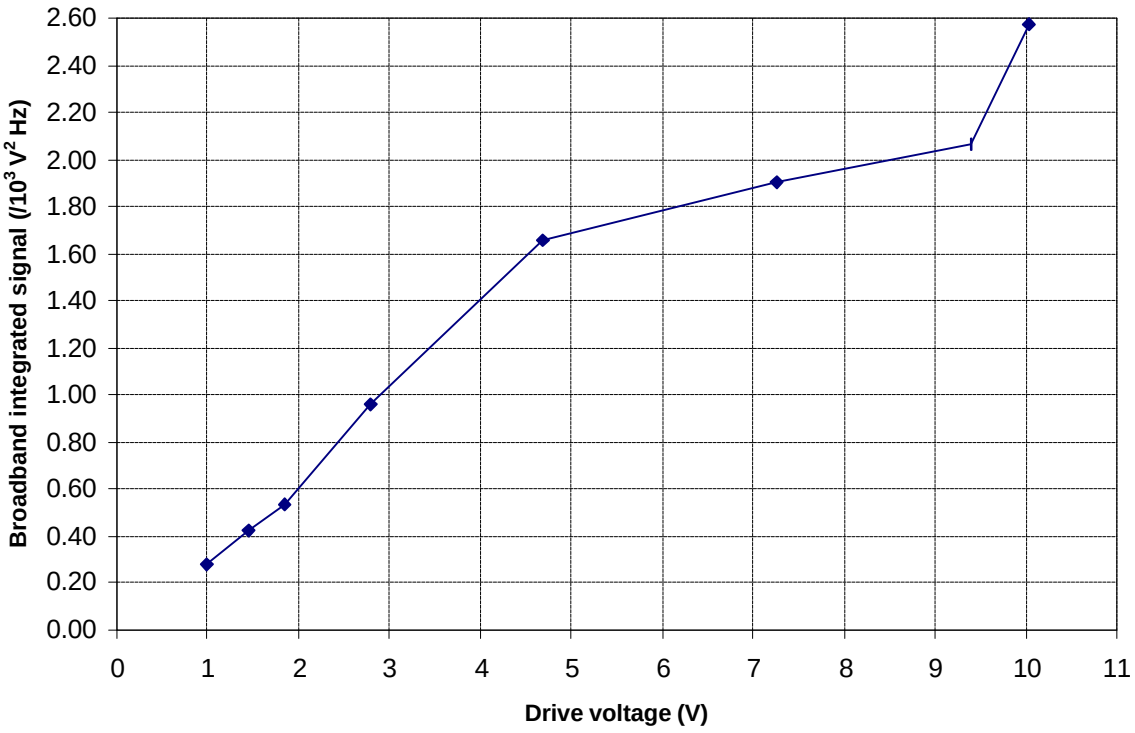

Figure 6.9: Broadband integrated signal as a function of actual DC voltage using PCS-11, "water purified" sample 1, descending data

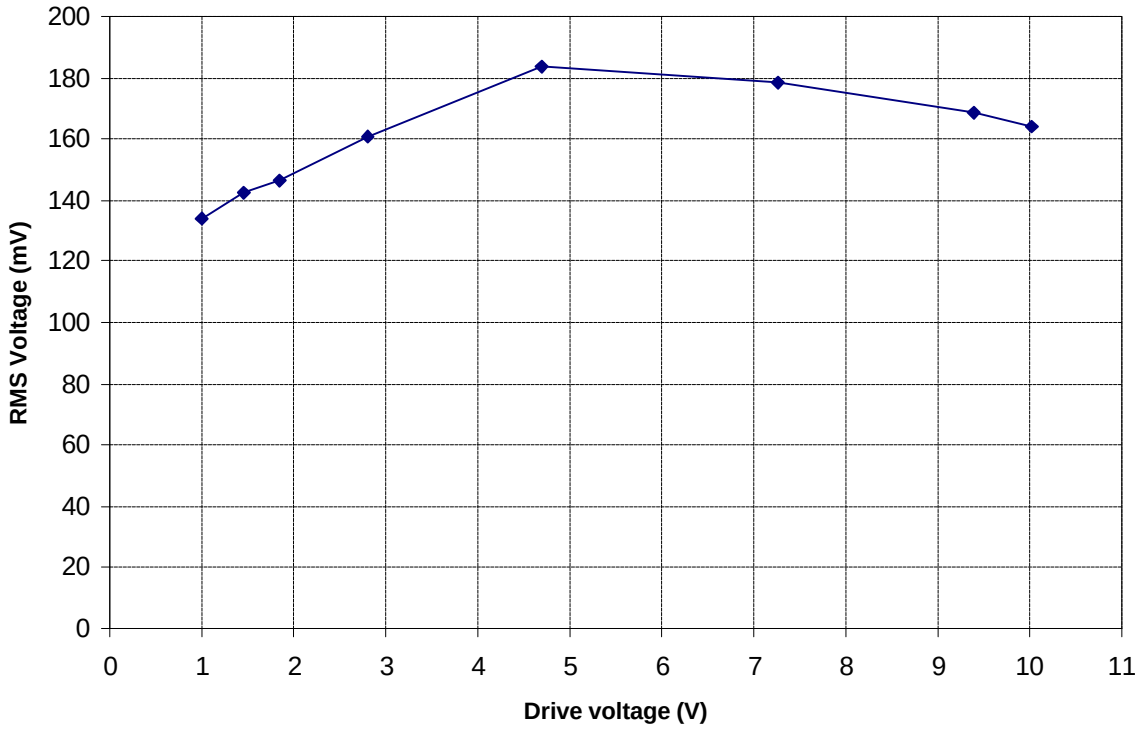

Figure 6.10: *rms* voltage as a function of actual DC voltage using PCS-11, "water purified" sample 1, descending data

Comparing the acquired spectra from the ascending and descending data sets (Figures 6.5 and 6.8) shows reasonable agreement at the lower power settings in terms of general levels, although it is apparent that the descending data set appears to have frequency harmonics of greater magnitudes, particularly for the 5% case. When examining the two sets of broadband power data (Figures 6.6 and 6.9), the descending set shows a similar trend to the ascending set, with the initial gradient decreasing as the DC voltage is increase. However in this case, the 95% power setting measurement shows a further increase, a different result to the ascending data. The *rms* plots (Figures 6.7 and 6.10) agree very well between the two data sets, both in measured levels and the trend seen, and this is interpreted as an indication that the cleaning vessel direct field is behaving in a reproducible manner. In general, the ascending and descending runs exhibit quite reasonable reproducibility. After this first pair of runs, a second set of measurements were completed on the same sample, and the broadband power data obtained in these cases can be found in the summary graph, Figure 6.12.

To assess the reproducibility of cavitation activity measured in the 'water purified' sample, the cleaning vessel was then emptied, and the second sample used. Sensor PCS-11 was left at the same position, and the measurement process repeated. Results for this set of data are provided in Table 6.2, and a comparison graph of the broadband power measured for the ascending and descending data sets is shown in Figure 6.11.

Table 6.2: PCS-11, 'Water purified' sample 2

Initial conditions: DO_2 level = 8.53 ppm, T = 20.7 °C			
Nominal % power level	DC voltage (V)	*rms* (mV)	Broadband power (/1000 V^2 Hz)
5	1.045	118	0.20
10	1.494	134	0.34
15	1.921	158	0.64
20	2.788	165	0.95
40	4.75	182	1.88
65	7.18	172	2.12
85	9.33	160	2.30
95	10.06	144	1.32
Middle conditions: DO_2 level = 8.56 ppm, T = 21.6 °C			
95	10.06	148	1.61
85	9.35	161	2.34
65	7.27	176	2.48
40	4.85	184	1.96
20	2.749	154	0.61
15	1.909	146	0.49
10	1.499	139	0.39
5	1.094	126	0.31
Final conditions: DO_2 level = 8.51 ppm, T = 22.4 °C			

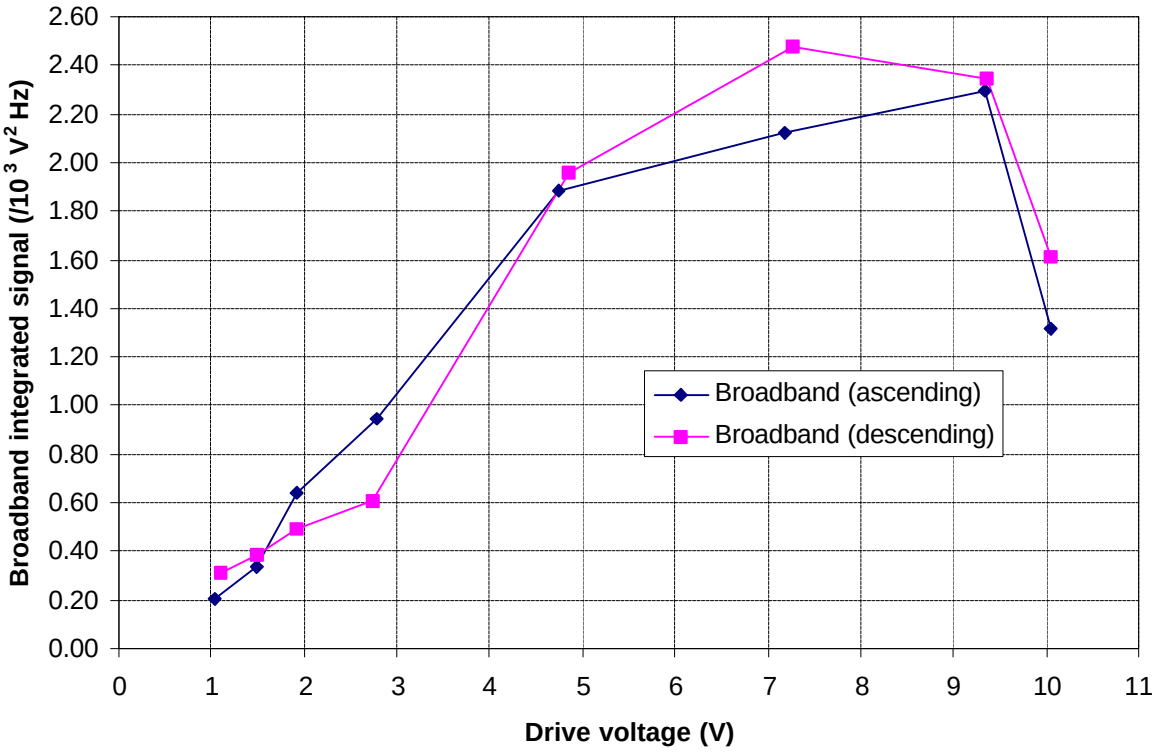

Figure 6.11: Broadband integrated signal as a function of actual DC voltage using PCS-11, water purified sample 2, ascending and descending data

Figure 6.11 illustrates that the second sample shows excellent agreement between the ascending and descending data in terms of the broadband signal, with no clear evidence of any hysteresis seen. Examining the data in Table 6.2 shows that the *rms* data is similarly well-represented.

The very similar environmental conditions (temperature, DO_2 level) measured in the two samples allows comparison of the data obtained from separate runs on a reasonable basis. The broadband data for all runs carried out over the two samples produces the plot seen in Figure 6.12.

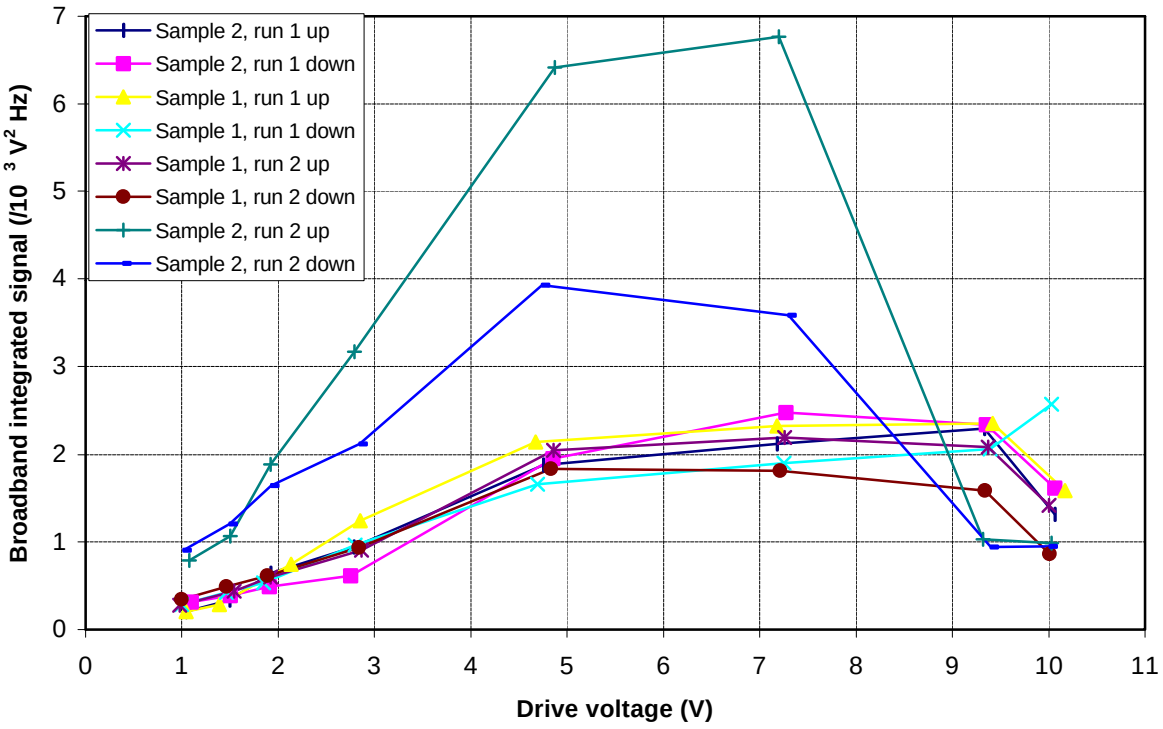

Figure 6.12: Broadband integrated signal as a function of actual DC voltage using PCS-11, data compilation of measurement runs for 2 independent samples

Figure 6.12 shows eight data sets in total, with ascending and descending data sets for each of two runs on each of the two samples. The six data sets corresponding to the measurements made on sample 1 (runs 1 and 2) and sample 2 (run 1) are readily superimposed, and illustrate good agreement. Examining the broadband power values at each of the drive levels measured for these six data sets shows that as a percentage of the mean, the average standard deviation over the full power range is 18%. This compares quite favourably with the cavitation activity reproducibility figures derived from sonar hydrophone measurements in the reference vessel (21), and suggests that the 'water purified' samples constitute a 'reference medium' approaching the quality of the degassed, de-ionised filtered water used in that case.

Two data sets stand out as showing a markedly different response: those acquired for the ascending and descending directions for the second measurement on sample 2. Examining the spectra acquired for these two data sets shows that in comparison to the other six data sets, there are a significant number of integer harmonics present in the frequency range 1 - 1.5 MHz, particularly for the data acquired at the nominal 40% and 65% drive settings. In all cases, these components have a substantial effect on the derived broadband power, so affecting the traces in Figure 6.12. The origin for these harmonics may be attributable to the following effects:

First, and probably most significantly, the second pair of measurements performed on Sample 2 were carried out after a three day break, during which time the sample was left in the reference vessel. Although located in the environmental enclosure, the water surface was not covered, and so it is possible that some particulate may have contaminated the sample, thereby increasing the likely number of cavitation nucleation sites.

Second, PCS-11 was not pre-wetted in washing-up liquid prior to these last two measurements, although it was immersed in the medium for 30 minutes prior to measurements being started.

Overall, however, the agreement seen in the majority of the measurements is quite acceptable, with the two exceptional data sets showing that there are still aspects of the vessel and sensor performance that need further study to home in on the reasons for occasional discrepancies seen. This dependence of the cavitation activity on the history of the medium is well established in the literature (8).

6.3.2 Results – "AnalaR water"

Measurements on the two "AnalaR water" samples were carried out in exactly the same way as the "water purified" samples, with the sensor located in the same position in the bath, and the same settings used for the spectrum analyser, using the COMORAC protocol outlined in Appendix D. Data was acquired in ascending and descending directions, and results are presented in a similar manner to those in Section 6.3.1.

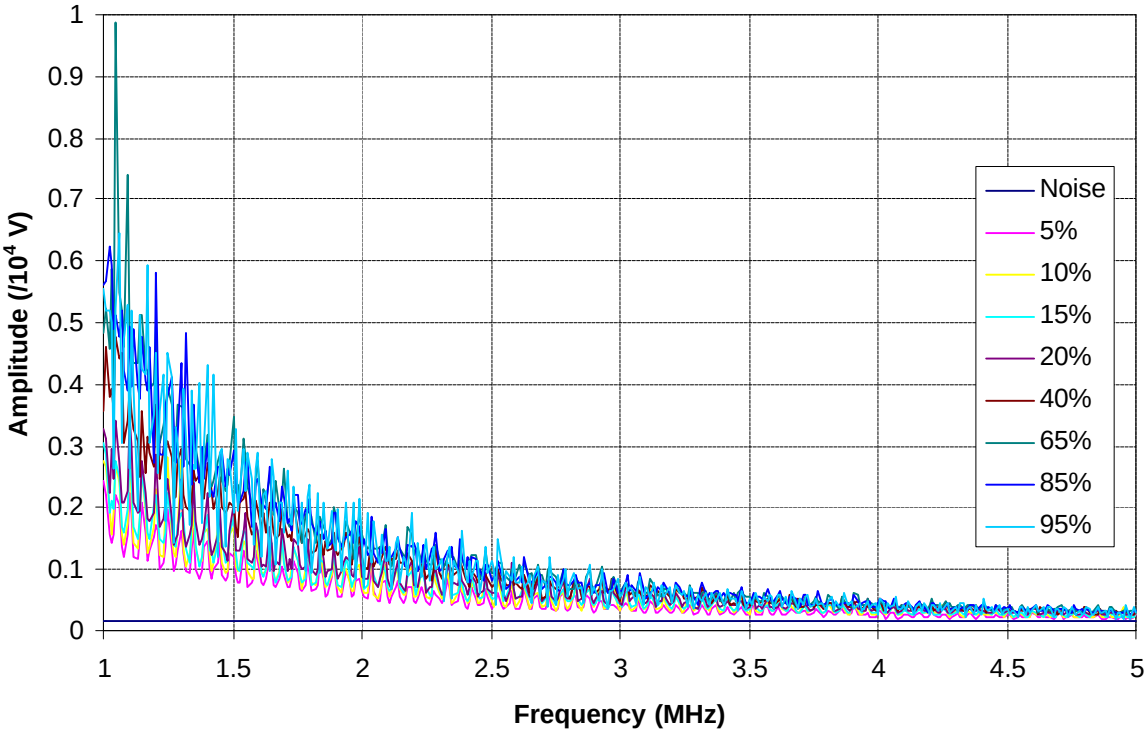

Figure 6.13: Acoustic spectra acquired as a function of nominal drive setting using PCS-11, "AnalaR water" sample 1, run 1, ascending data

Table 6.3: PCS-11, "AnalaR water", sample 1, run 1

Initial conditions: DO_2 level = 6.86 ppm*, T = 20.8 °C			
Nominal % power level	DC voltage (V)	*rms* (mV)	Broadband power (/1000 V^2 Hz)
5	0.97	97	0.18
10	1.49	101	0.27
15	1.89	112	0.30
20	2.77	137	0.38
40	4.77	142	0.69
65	7.24	141	1.20
85	9.34	140	1.10
95	10.03	132	1.04
Middle conditions: DO_2 level = 6.56 ppm*, T = 21.6 °C			
95	10.02	148	1.29
85	9.36	142	1.15
65	7.27	140	0.93
40	4.88	151	0.64
20	2.84	155	0.42
15	1.93	165	0.37
10	1.41	132	0.30
5	0.99	137	0.25
Final conditions: DO_2 level = 5.86 ppm*, T = 22.2 °C			

* NB – it was discovered subsequently that these measurements of DO_2 level are incorrect, due to problems with deploying the meter. Spot checks showed that the 'true' values are very similar to those seen with the "water purified" sample, i.e. in the range 8.4 – 8.7 ppm.

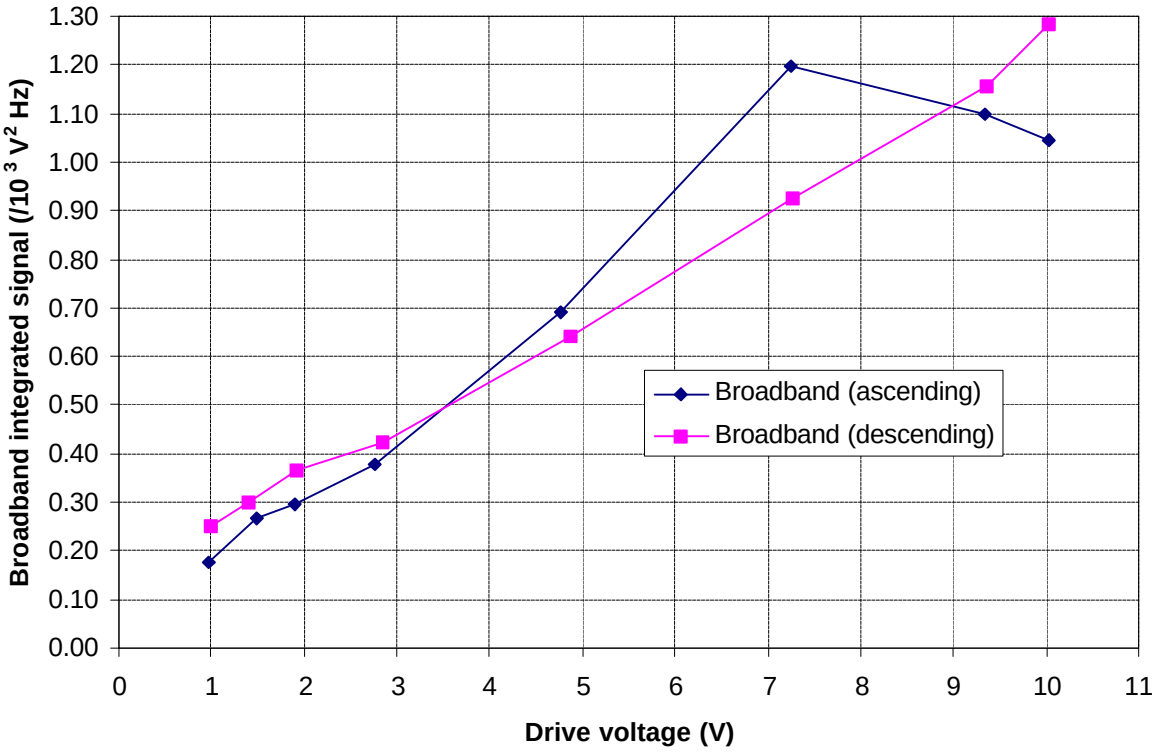

Figure 6.14: Broadband integrated signal as a function of actual DC voltage using PCS-11, Analar water sample 1, run 1, ascending and descending data

The broadband power plot, Figure 6.14, shows that for the ascending run, the trend as the drive increases is as seen for the majority of data sets acquired using the "water purified" sample, with the value at the nominal 95% level being lower than that seen at the 85% setting. The descending run, however, shows a steady trend, decreasing almost linearly from 95% to 5%. Comparing the two sets of *rms* data in Table 6.3 shows that the overall levels between the two half sets are of a similar order at the higher drive levels, although they deviate at the lower drive levels. The reason for this is unclear.

After measurements had been completed, the Analar water was left in the cleaning vessel overnight, with some film used to cover the water surface and thus prevent significant environmental impurities getting into the medium. The following day, another set of ascending and descending measurements were carried out on the sample, with PCS-11 at nominally the same location. Data is shown in Table 6.4 and Figures 6.15 and 6.16.

Table 6.4: PCS-11, "AnalaR water", sample 1, run 2

Initial conditions: DO_2 level = 5.56 ppm*, T = 21.2 °C			
Nominal % power level	DC voltage (V)	*rms* (mV)	Broadband power (/1000 V^2 Hz)
5	1.06	170	0.23
10	1.51	192	0.28
15	1.98	187	0.28
20	2.82	185	0.37
40	4.92	155	0.63
65	7.21	149	0.92
85	9.34	138	1.18
95	10.02	145	1.23
Middle conditions: DO_2 level = 6.06 ppm*, T = 22.0 °C			
95	10	143	1.40
85	9.35	150	1.41
65	7.21	160	0.95
40	4.88	160	0.67
20	2.85	162	0.45
15	1.91	134	0.43
10	1.5	125	0.37
5	0.96	110	0.30
Final conditions: DO_2 level = 5.70 ppm*, T = 22.7 °C			

* NB – incorrect values of DO_2, as described previously.

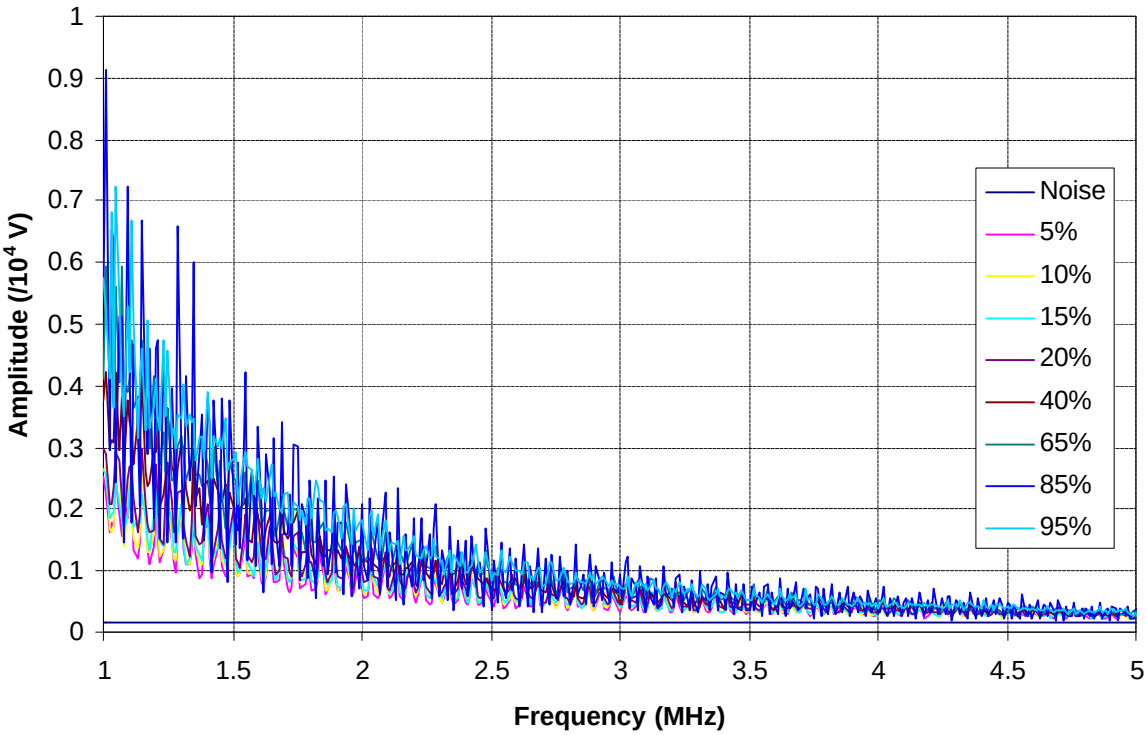

Figure 6.15: Acoustic spectra acquired as a function of nominal drive setting using PCS-11, "AnalaR water" sample 1, run 2, ascending data

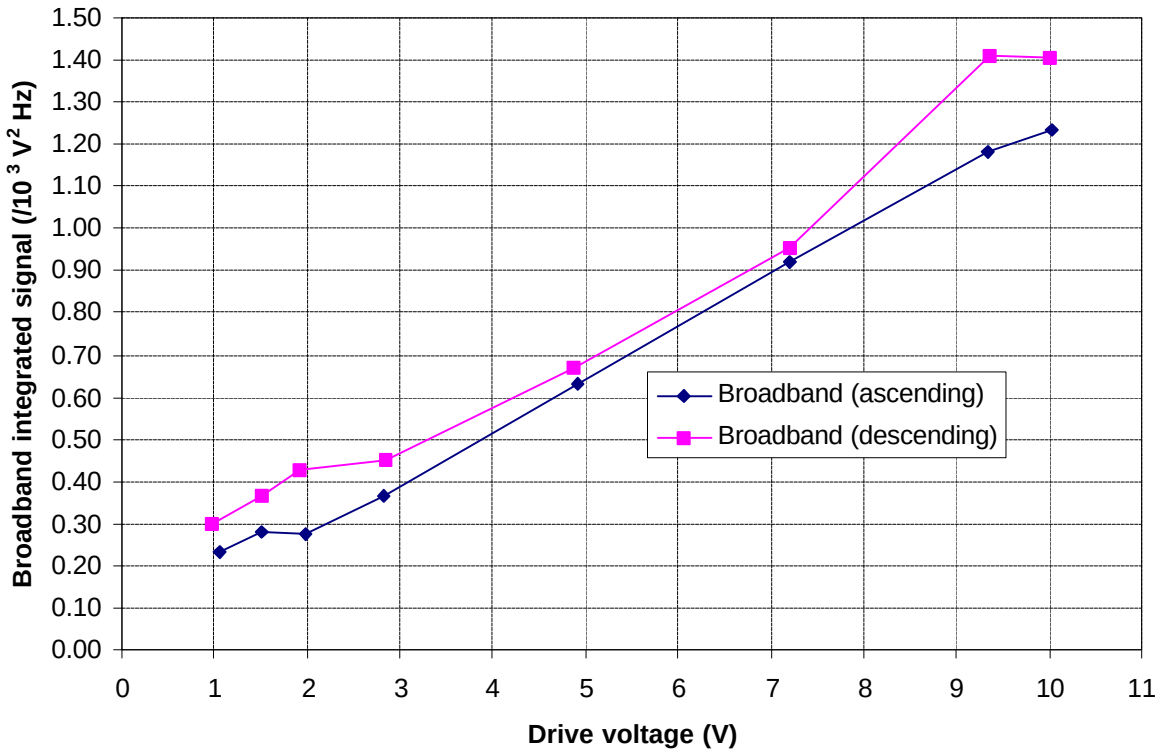

Figure 6.16: Broadband integrated signal as a function of actual DC voltage using PCS-11, "AnalaR water" sample 1, run 2, ascending and descending data

The broadband integrated data from run 2 (Figure 6.16) shows good agreement between the ascending and descending data sets, with a smaller deviation between the signal levels at the higher drive levels than seen in run 1. The actual levels themselves are also in quite good agreement with those seen in run 1, indicating that the activity in the medium shows good reproducibility day on day. Figure 6.17 shows a comparison of the broadband power for all four data sets.

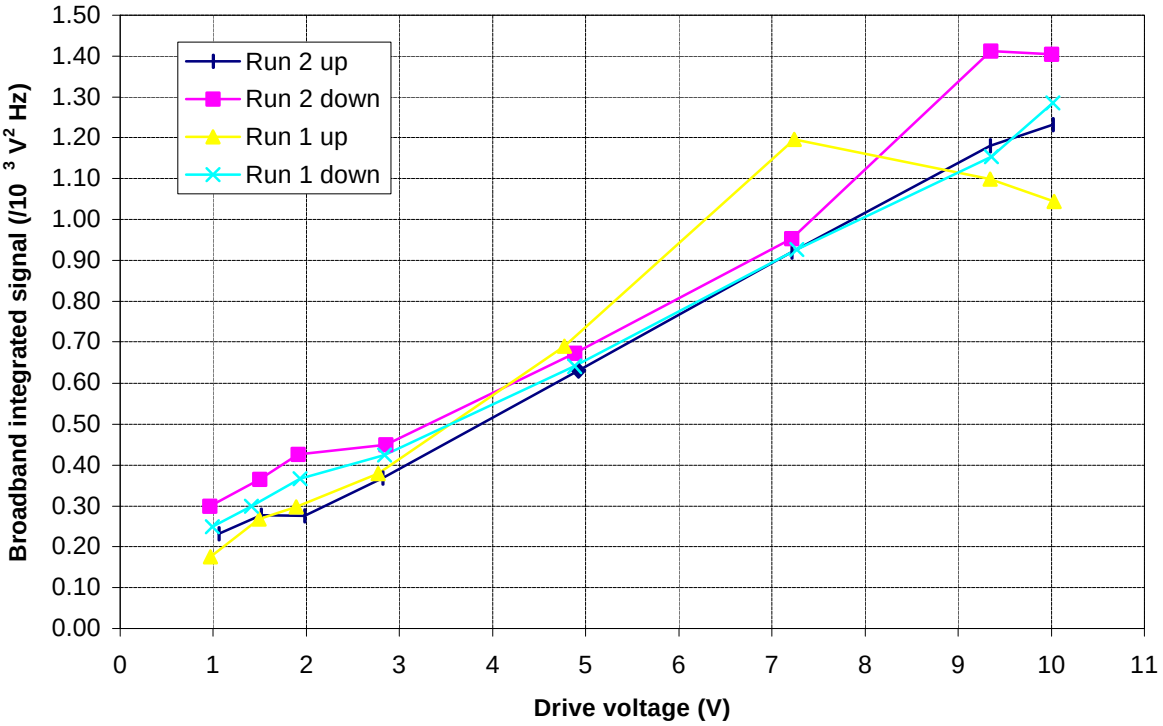

Figure 6.17: Comparison of four separate runs on "AnalaR water", sample as a function of actual DC voltage using PCS-11, broadband power

Again, as seen for the "water purified" sample, the four runs show good agreement, particularly at the lower powers, with an average standard deviation over the full range of 13%. Interestingly, the general trend as a function of DC voltage this time appears to show a systematic increase, with the first ascending run being the only data set to show a downturn at the higher powers.

Comparing Figures 6.12 (six main sets) and 6.17 in terms of overall levels shows that the "AnalaR water" used here produces broadband power levels that are typically 30% less at the lower powers, and 40% different at the higher orders. Generally, there is quite a difference in profiles, with the "AnalaR" sample showing a more linear variation with DC voltage. This, and the difference in overall signal levels, could be related to the differing levels of particulate content in the two samples. The fluid specifications in Table 5.1 show that the properties of the "water purified" medium include a specification on the particulate level of 2 ppm, compared to the 1 ppm specified for the higher grade "Analar water". However, it is considered that this relatively small difference in the particulate level is unlikely to be the sole cause for the differences seen in broadband power levels between the two samples, and so suggests that other contaminants present in the standard samples at source may also contribute to the differences. These differences are considered worthy of further investigation.

The *rms* levels seen for this sample are around 30% lower than those seen for the "water purified" sample. The reasons for this difference are again unclear, as for better-filtered media, the *rms* level would be expected to increase (due to less significant shielding effects). Again, examining this difference could be considered for further experimentation.

Before measurements were made on the second "AnalaR water" sample, some repairs had to be made to Sensor 11, after erosion damage caused by cavitation was noticed. Some limited evaluations of sensor PCS-11 in degassed, filtered water before and after the repairs showed that the device produced an output of a similar frequency range, but at a lower overall level. The second sample of Analar water was measured a few days later, this time as an 'ascending' data set only, again with Sensor 11 in nominally the same location. Data is shown for acoustic spectra and broadband power only, Figures 6.18 and 6.19. Given the change in sensor response, it is considered inappropriate to compare the data acquired here with the two runs obtained using Analar water sample 1 on an absolute level, but comparing trends as a function of power may still be done on a reasonable basis.

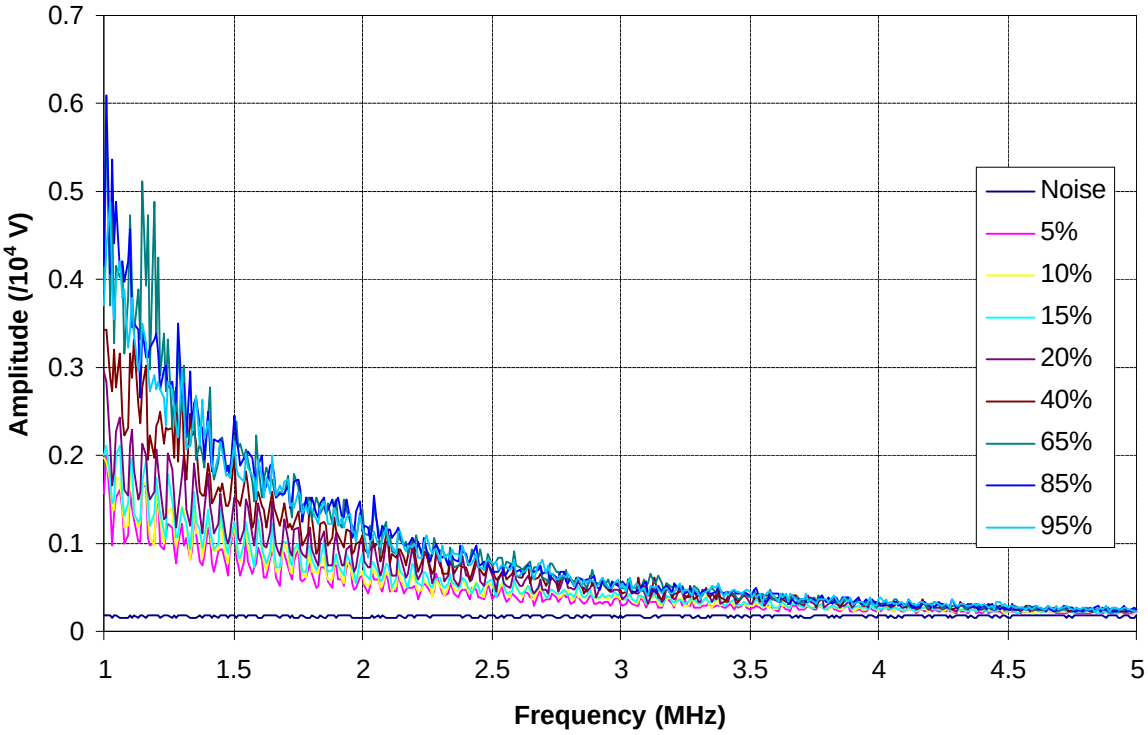

Figure 6.18: Acoustic spectra acquired as a function of nominal drive setting using PCS-11, Analar water sample 1, run 2, ascending data

The spectra in Figure 6.18 show a more pronounced harmonic-based behaviour than the previous measurements (e.g. Figure 6.13), illustrating that the high levels of DO_2 in the medium can provide plenty of nucleation sites from which stable cavitation may initiate. The 65% setting produces a spectrum that has some local structure at around 1.2 MHz: this has been seen sporadically in other measurements, both prior to and since repairs were effected to PCS-11. Its origin has been tested to be acoustic (simply by removing the PCS from the medium with the vessel activated), but the exact nature of the processes that cause it, cavitation or otherwise, are unclear. Figure 6.19 shows the derived broadband power data.

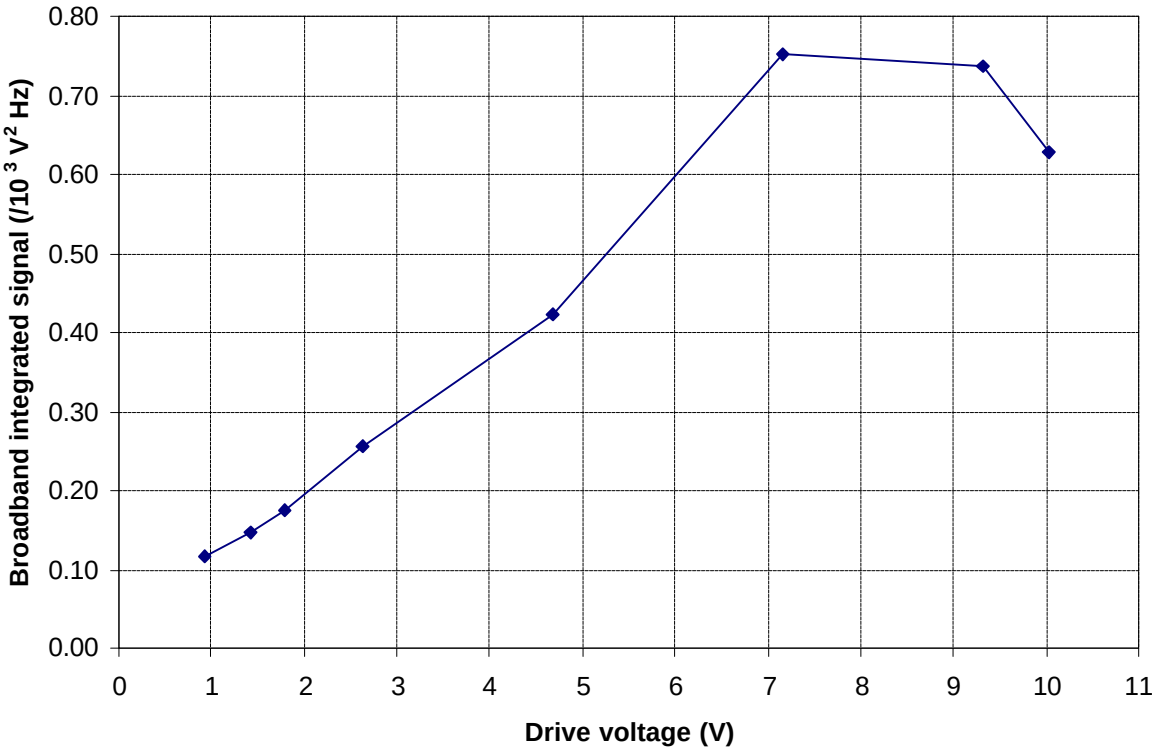

Figure 6.19: Broadband integrated signal as a function of actual DC voltage using PCS-11, Analar water sample 1, run 1, ascending and descending data

Examining the broadband activity in this case shows again the trend initially identified with the "water purified" sample, with the values at the two highest powers being lower than the 65% value. The spectral feature identified at 1.2 MHz in the 65% setting spectrum will increase the broadband energy value calculated, and so may be distorting the trend somewhat; however, the near-linear fit seen in previous data for this sample is also observed here, up to the data gathered at the 40% setting. In terms of a profile, it agrees quite well with the traces shown in Figure 6.17, with the repairs made to PCS-11 seeming to have reduced the response in broadband power by around 35 - 40%.

6.3.3 Discussion of standard water results

Even over the relatively limited number of measurements performed, both the standard water samples showed an acceptable level of reproducibility. Due to some uncertainties over the performance of PCS-11 because of the erosion damage spotted during the "AnalaR water" tests, the data gathered using the "water purified" samples is considered more reliable, and Figure 6.12 shows encouraging levels of reproducible activity in most instances when the broadband power is considered.

In the context of a reference medium, it is considered that a larger number of nucleation sites may be desirable, as it would appear to increase the sensitivity of the sensor response, and hence give a larger dynamic range of results for a given set of DC drive voltages. However, there probably exists a threshold above which increasing the number of nucleation sites can saturate the response of a given sensor, thus giving results that are more difficult to interpret. The studies carried out on the hydrophobic polystyrene spheres in principle allowed more control over the number of nucleation sites, and the systematic studies of activity as a function of drive setting and particle 'concentration' are reported in Section 6.4.

6.4 CANDIDATE MATERIAL STUDIES 2: HYDROPHOBIC POLYSTYRENE SPHERES

6.4.1 Background

The polystyrene spheres could potentially form a whole area of study on their own, but such an exercise is beyond the scope of the current project. Specific, targeted measurements were hence made on a limited number of samples, in keeping with the 'feasibility' aspect of the project.

6.4.2 Preparation

The spheres were supplied as detailed in Table 5.1, with 0.5 g of the particles dissipated in 4.5 g of deionised water. Based on the information supplied by the manufacturer, this 5 g of fluid contained approximately 780 million microspheres, of mean diameter 10.50 μm. The relative density of the spheres is 1.061. To enable the studies to be carried out, a 'master solution' was prepared, consisting of 2.5 g of the procured particle solution (thoroughly mixed) added to 1000 ml of degassed, deionised filtered water (from a measuring cylinder), and then decanted into a clean beaker. The mixture was stirred using a clean glass rod, and the beaker covered at all times to minimise contamination from atmospheric impurities. The concentration of this master solution was thus (to a good approximation) 778 million particles per litre.

To evaluate the candidate material, it was decided that tests would be carried out on small volumes of the fluid, located around the sensor. It was considered that this approach would be more controllable than filling the reference vessel with the microsphere solution. To effect this, polyethene sample bags were obtained, approximately 150 mm high and 100 mm wide, with a clip seal at the top. For most tests, the cleaning vessel was filled with the "AnalaR water" used for the experimentation reported previously, as it was felt that it had showed relatively good reproducibility in terms of the cavitation activity measured in it, and the propagation of low frequency ultrasound through it. This suggested that it would act as a suitably reproducible coupling medium.

6.4.3 Initial testing

To test the influence of the bag on the PCS-11 signal, the sensor was submerged initially at the 'reference' depth (9 cm) below the centre of the cleaning bath, a sample bag lowered into the vessel, so that it filled with the "AnalaR water", and then moved up so that it surrounded the PCS-11. The bag was attached to the epoxy mounting rod holding the PCS-11 using fabric tape, such that the clip seal and approximately 15 mm of the bag was out of the water (Figure 6.20) The level of the water in the bag was equivalent to that in the vessel. The vessel was then activated, and spectra were acquired at the 20% and 65% drive settings. The bag was then removed, and the volume of its contents was checked using a measuring cylinder, showing that the bag contained 250 ml. The water from the bag was then returned to the bath, and the same drive settings measured again in the absence of the bag. The acquired spectra for the four measurements are shown in Figure 6.21.

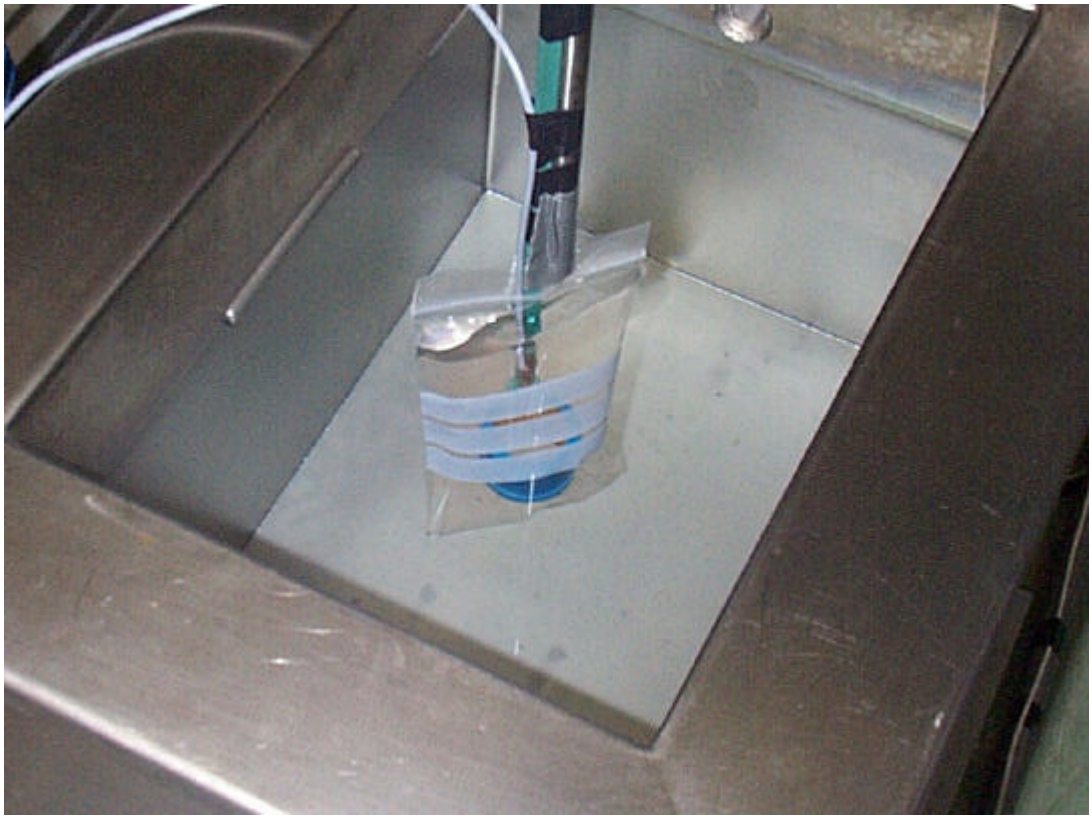

Figure 6.20: PCS-11 located inside polyethene sample bag in reference vessel

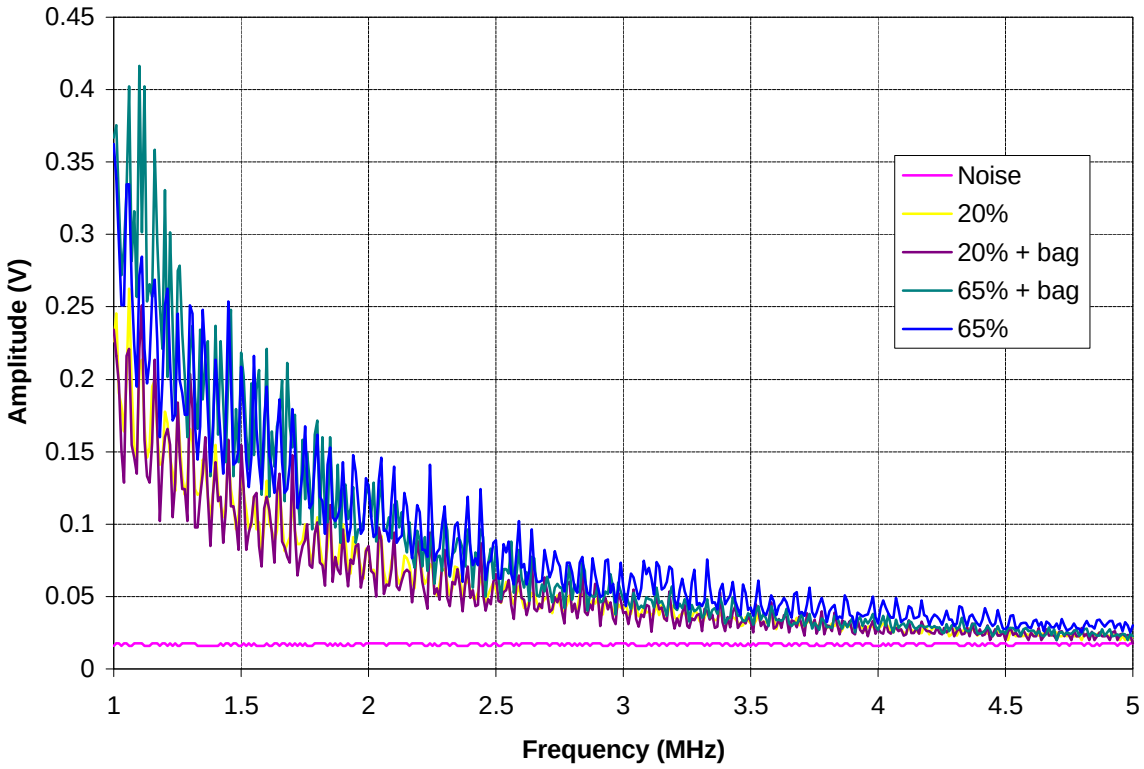

Figure 6.21: Comparison of acquired spectra at two drive levels, with and without the sample bag present, PCS-11, Analar water

Figure 6.21 shows that there is good agreement seen for the two pairs of measurements: the data acquired with PCS-11 in the bag shows more 'peaky' behaviour, particularly at the lower frequencies, but generally, the traces are quite similar. The derived broadband power values for these spectra agree to 4% and 13% for the 20% and 65% settings respectively. This shows that the bag has no significant effect on the cavitation signal measured by the sensor when the medium in the vessel and the bag are the same. On this basis, the particle studies were carried out using this experimental arrangement.

6.4.4 Experiment 1 – first test of effect of particles

The beaker containing the 'master solution' was stirred thoroughly, to homogenise its contents, and 50 ml of the mixture was poured into a measuring cylinder. The cylinder was then topped up with 200 ml of fresh degassed, deionised water, producing a mixture containing approximately 39 million particles. A separate beaker of the degassed, deionised water was obtained at the same time, to check the DO_2 level and the temperature (measured at 0.78 ppm and 23.3 °C respectively). The 250 ml of diluted particle solution was again stirred, and carefully poured into a fresh sample bag. Taking care not to allow any of the vessel medium to come into contact with the particle solution, the bag was secured around the PCS-11 in the same manner described above, with approximately 15 mm of the bag above the water surface. As above, the level of fluid in the bag coincided with that in the vessel.

Acoustic spectra were then acquired for four power settings spanning the range available: 20%, 40%, 65% and 95%. This initially limited range was used to seek to identify trends, without necessarily exposing the solution to significant amounts of cavitation. A noise trace was also captured. After these five spectra, the 20% measurement was repeated, to examine the reproducibility, and to check if there was any early indication of any change occurring to the medium: it was considered that cumulative ('dose') effects of cavitation would perhaps reduce the particle size through collisions and erosion, effectively increasing the number of particles, and thus change the medium. The acquired spectra are shown in Figure 6.22.

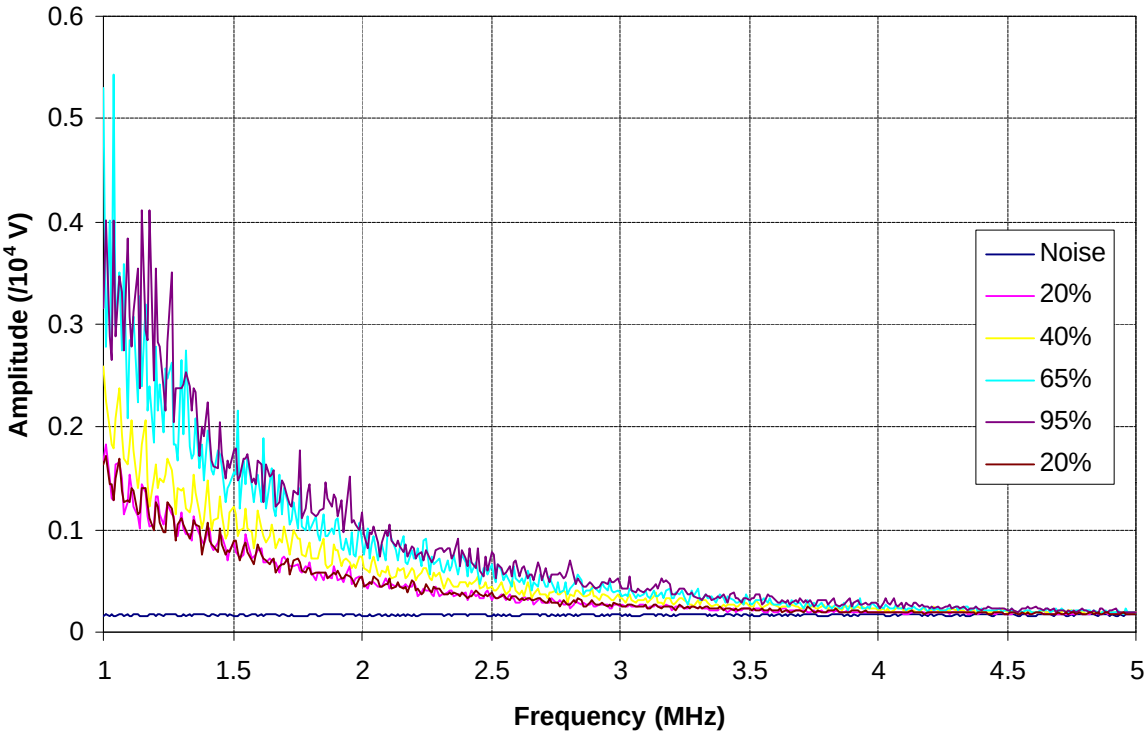

Figure 6.22: Acoustic spectra acquired using PCS-11 as a function of drive setting for 20% concentration of 'master solution'

Figure 6.22 shows that there is excellent reproducibility in the measurements, with the two 20% setting measurements agreeing very well. The overall appearance of the spectra is perhaps less harmonic-rich than the data seen in Figure 6.18, which is of interest: the latter were acquired in gassy Analar water, whereas in this case, the background medium is degassed water.

Further measurements were made at the other power settings, to provide more detailed information over the full operating range. This data was then analysed to produce the broadband power curve seen in Figure 6.23.

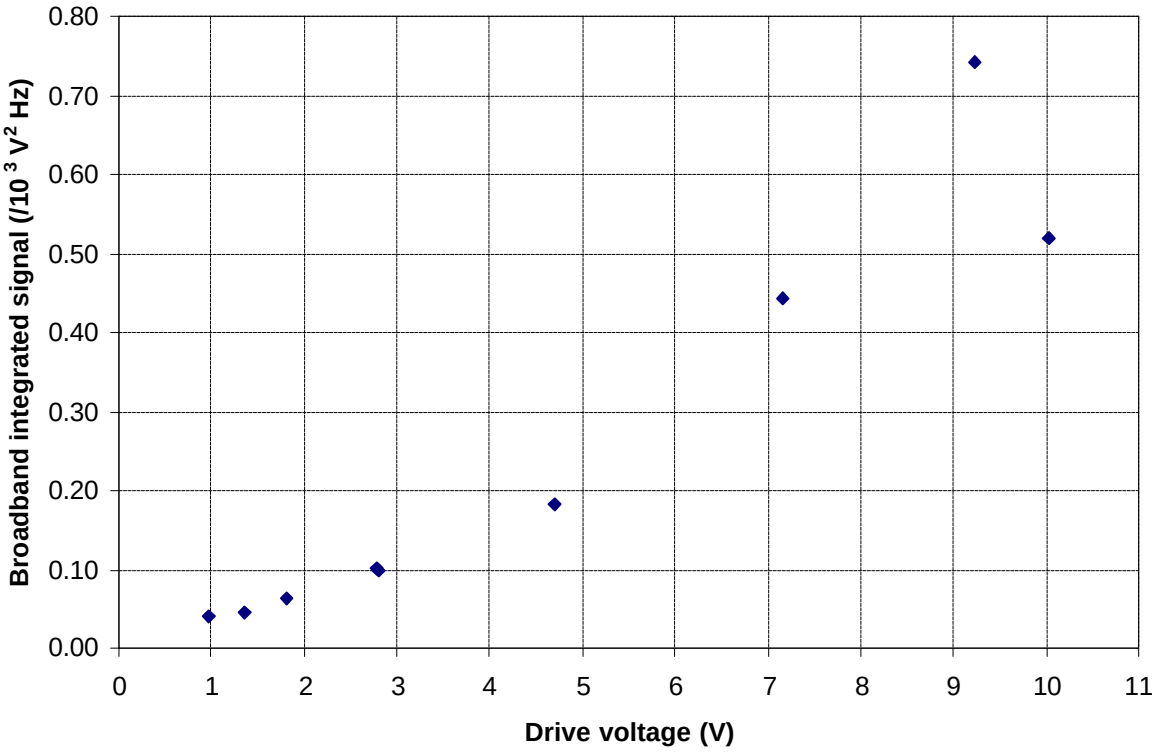

Figure 6.23: Broadband power as a function of DC drive voltage, PCS-11, for 20% concentration of 'master solution'

The acquired spectrum producing the broadband power value seen in Figure 6.23 at the 85% setting (9.2 V dc) was noticeably different from the other measurements, in that some additional structure was noted in the noise envelope between 1.5 and 2.5 MHz. The 85% measurement was the last in the set to be taken, and it is postulated that the apparent change in activity might be due to changes in the medium, as mentioned above. Generally, though, the trend of the broadband power with DC voltage is quite similar to the results seen with the 'standard' water samples, with the initial increase as a function of DC voltage slowly tailing off (if the 85% measurement is considered separately). Significantly, the measured broadband power levels (c.f. Figure 6.19) in what amount to two very different experimental set-ups are of a similar order. This suggests that the concentration of particle solution used here, with degassed water, produces a medium with similar attributes to the "AnalaR water" used above, but with very different measured spectra.

Further measurements were made on this first sample at the first four settings used, and then additional checks carried out at 20% and 85%. The overall plot of broadband power for all data is shown in Figure 6.24. The data shown in Figure 6.24 in red is the 'second' set at 20%, 40%, 65%, 85% and 95%, and suggests further that a relative increase in broadband cavitation activity is being seen as the overall cavitation 'dose' applied to the sample increases. It appears likely that the phenomenon is threshold-related, as the three measurements taken at the beginning, middle and end of the run at the 20% power level (drive level approximately 2.7 V) all agree to within a few per cent.

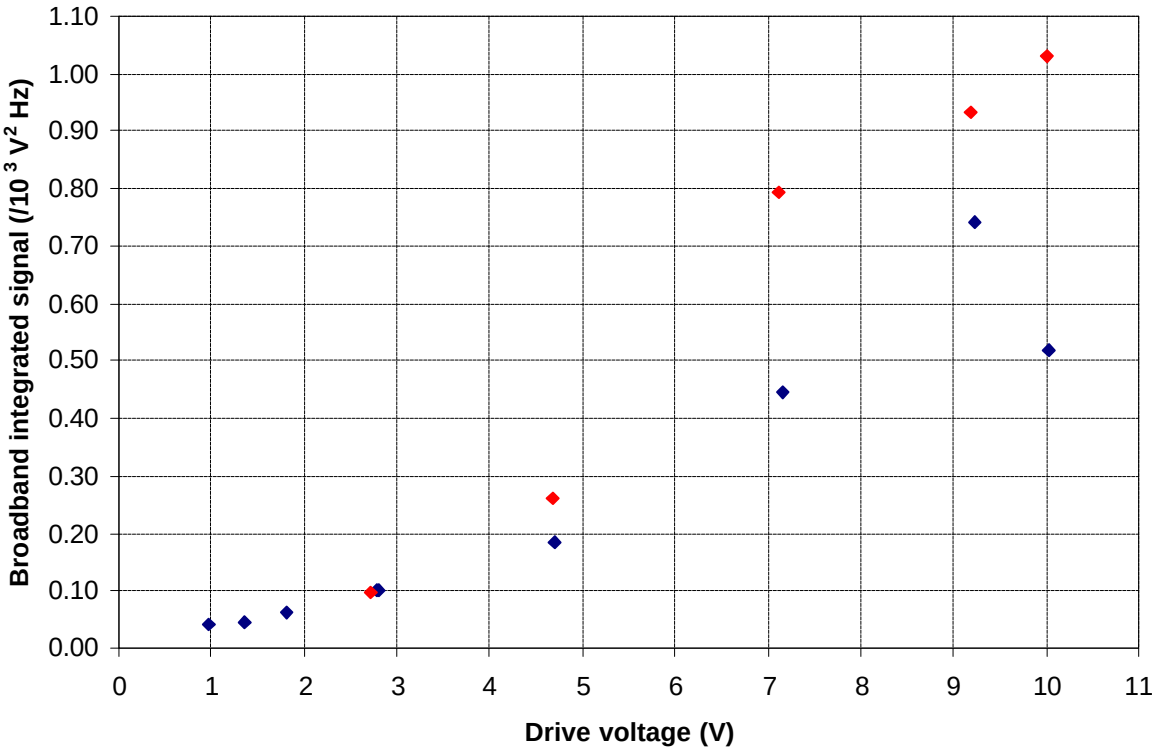

Figure 6.24: Broadband power as a function of DC drive voltage, PCS-11, for 20% concentration of 'master solution', further measurements

6.4.4.1 Investigation of particle 'ageing'

To investigate the cumulative dose effect, also described as the 'ageing' effect alluded to above, further measurements were performed at the 65% setting using the same sample. The spectra from these, together with the data acquired previously, are shown in Figure 6.25. The data is presented in the order that it was acquired; the maroon trace was first. There appears to be a steady increase in the magnitude of the spectra as a function of 'run number', and this is borne out by the derived broadband power plot, shown in Figure 6.26.

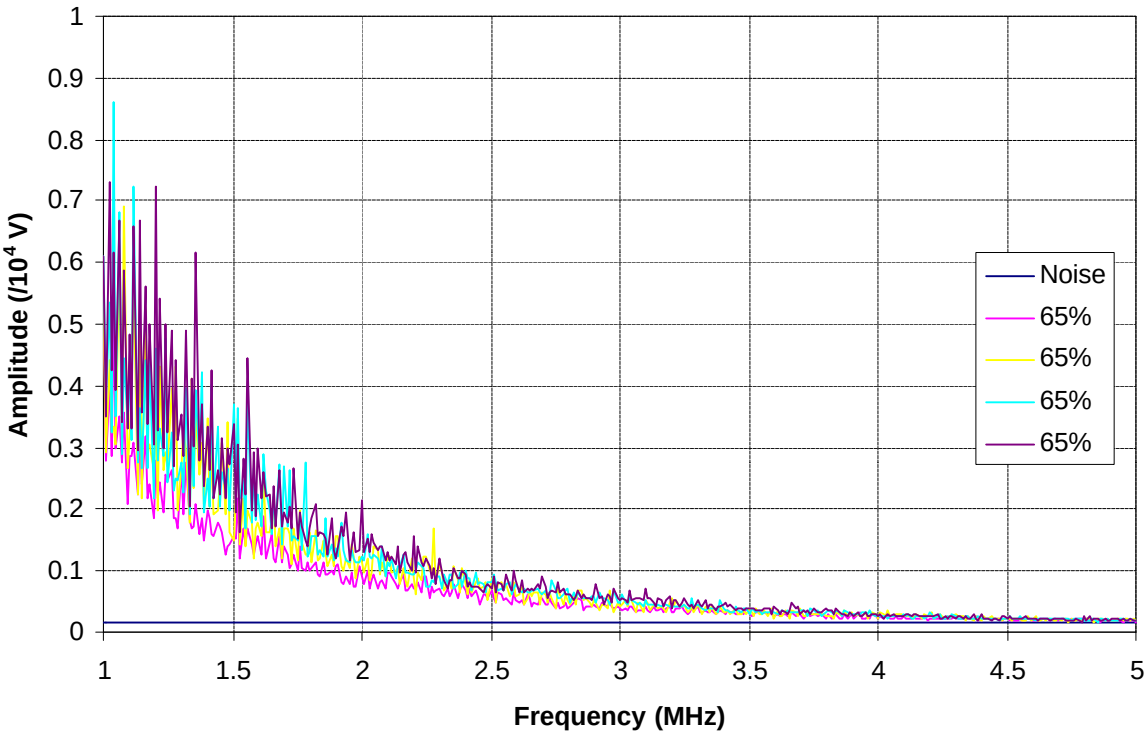

Figure 6.25: Four acoustic spectra acquired as a function of 'dose', 65% setting, PCS-11, 20% concentration of 'master solution'

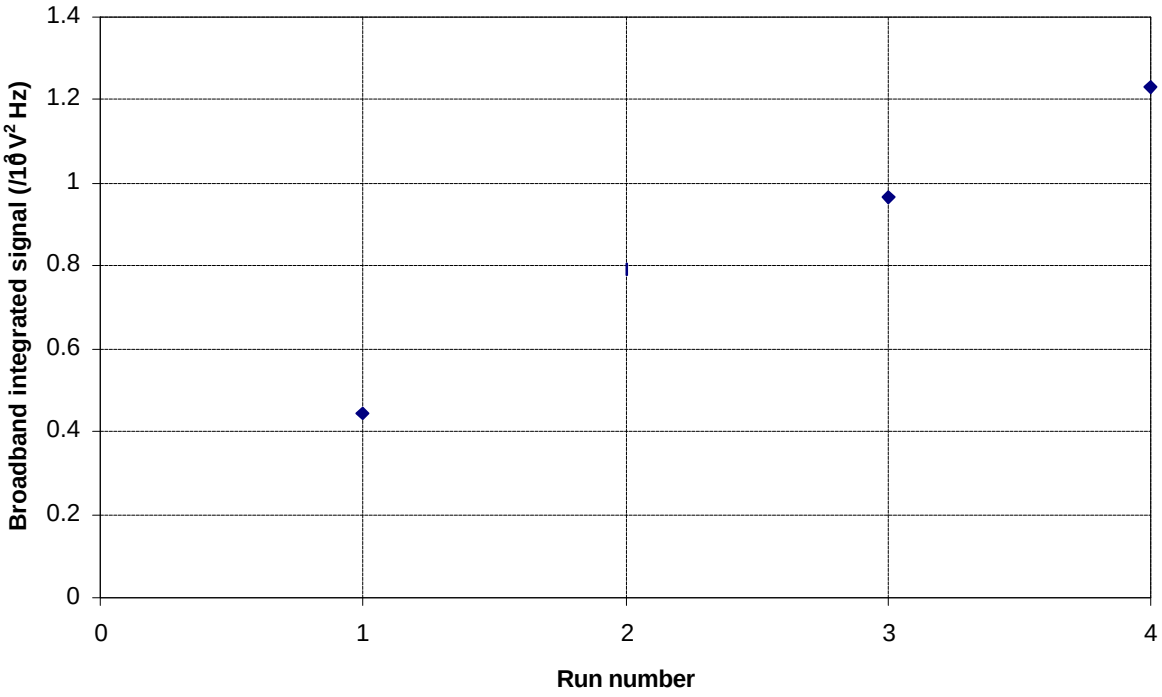

Figure 6.26: Broadband power as a function of 'dose', 65% setting, PCS-11, 20% concentration of 'master solution'

Figure 6.26 shows a steady increase in the measured broadband power through the successive runs carried out, showing that the cavitation signal from the particle-rich medium increases, by approximately a factor of 3. This increase was not thought to be due to temperature, as after the last measurement was made, the vessel temperature had increased by less than 1.5 °C (and the fluid in the bag was considered to be in reasonable thermal equilibrium with it). Instead, the possibility of cumulative changes to the particles themselves (and correspondingly, perhaps the number of particles) was considered more likely.

Also possible is that at the start of the measurements, the polystyrene particles may be gathered together in clumps, and these may be slowly de-agglomerated by the action of cavitation, so effectively increasing the actual number of sites from which cavitation may originate. Note that over the course of all of the first-stage particle solution measurements, the *rms* level remained steady and reproducible, not showing any systematic increases in sympathy with the broadband power changes seen in Figure 6.26.

6.4.5 Second experimental run

To evaluate the reproducibility of the data, a second, nominally-identical run was carried out, using the same medium in the bath, the same level of dilution of the master solution, and with a fresh sample bag and 20% concentration solution. The broadband power values derived from the measured spectra are shown in Figure 6.27.

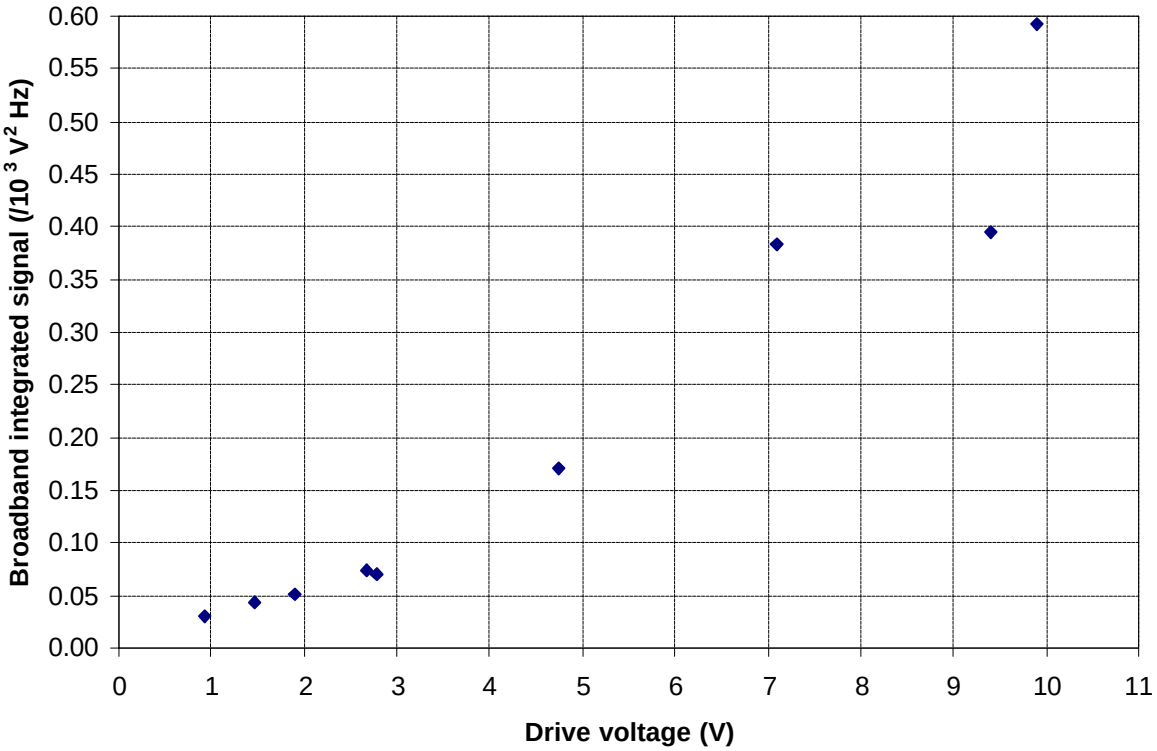

Figure 6.27: Broadband power as a function of DC drive voltage, PCS-11, for 20% concentration of 'master solution', repeat run

In this case at the lower drive levels, the profile of the trend is very similar to that seen in Figure 6.23, with the respective signal levels across the two runs, particularly in the range 5-65%, being in good agreement. As seen with the standard water samples, the differences

between the two runs occur at the higher power levels, this time with the 95% setting result providing the peak response. The *rms* values over the full power range for both runs were also in good agreement.

Extensive further measurements were also carried out to examine the 'dose' effect as described above, again concentrating on the 65% power setting. For these measurements, as previously, a stabilisation time of approximately 2 minutes was allowed between successive vessel excitations, in line with the COMORAC protocol. Figure 6.28 shows the variation. In this case, the steady increase seen previously is not obviously repeated, with the greater number of measurements completed in this case showing an apparent increase and subsequent turn over in the broadband power as a function of 'dose'. This suggests further that there is a level of 'dose' beyond which the ageing effect changes, and the nucleation attributable to the microspheres starts to lessen.

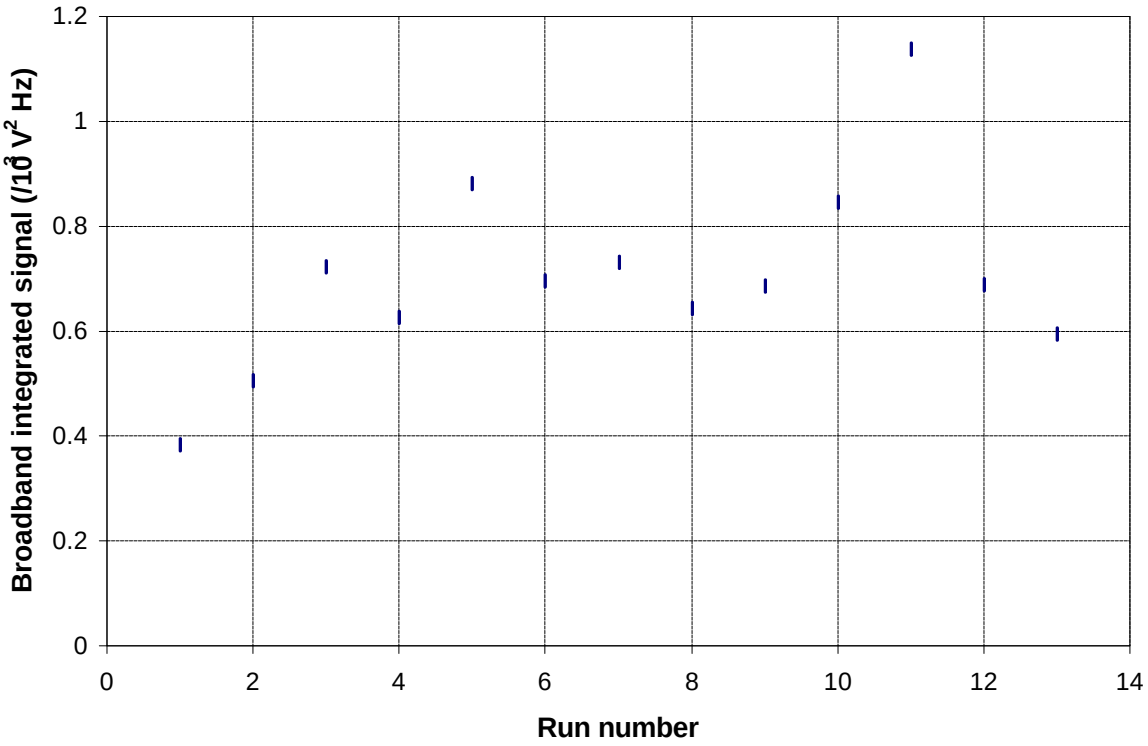

Figure 6.28: Broadband power as a function of 'dose', 65% setting, PCS-11, 20% concentration of 'master solution'

6.4.6 Investigation of changing particle concentration

Keeping the same propagation medium in the bath, a fresh sample bag was filled with 250 ml of the neat master solution, equivalent to a five-fold increase in the particle concentration in comparison to the first two investigations, and giving a volume containing approximately 200 million microspheres. Before the bag was put around the sensor, sensor PCS-11 was thoroughly rinsed off into a suspended beaker, to remove any adhering particles. Measurements were then carried out on a similar basis to those above, working initially over a selected range of drive levels (Figure 6.29).

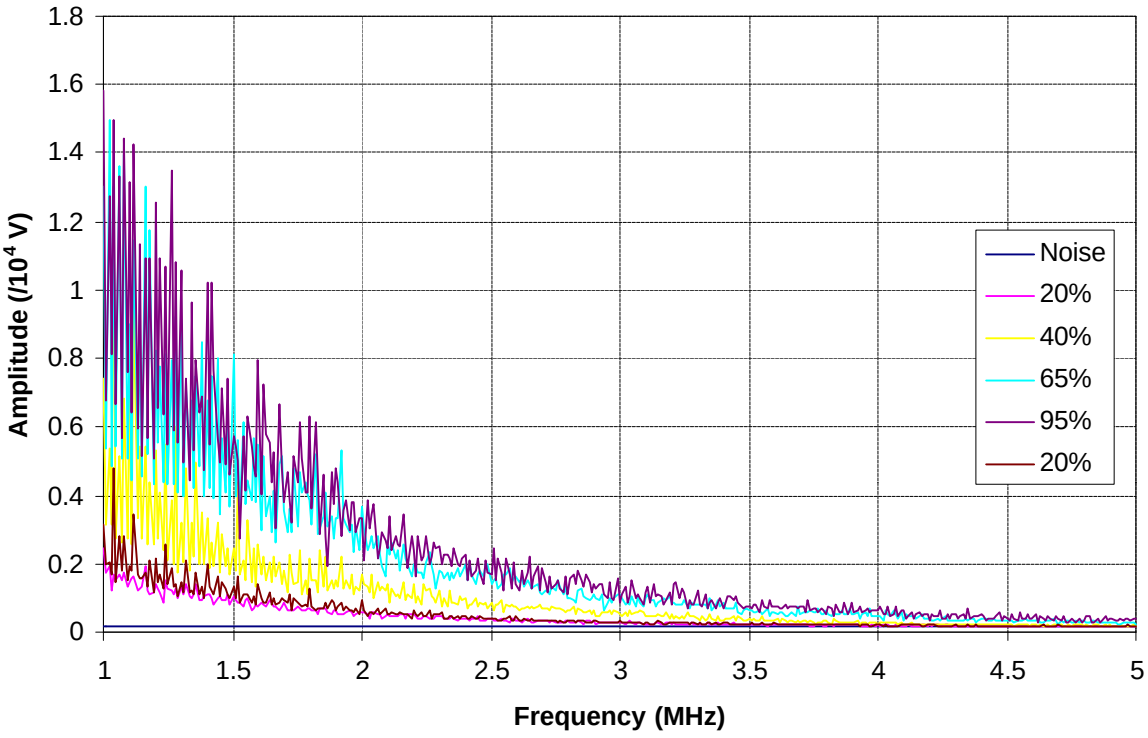

Figure 6.29: Acoustic spectra acquired as a function of drive setting for neat 'master solution'

Comparing the spectra in Figure 6.29 with those in Figure 6.22 shows that there is little evidence of harmonics in this instance, in contrast to the 20% concentration spectra. The five-fold increased concentration of polystyrene spheres manifests itself as an approximately three-fold increase in the level of the spectra at the respective drive settings. The lack of harmonics here indicates that only inertial cavitation is occurring for this higher concentration of microspheres. The *rms* voltage also shows an increase with the change in concentration (Figure 6.30), which is indicative of the direct field measured by the sensor being of greater magnitude.

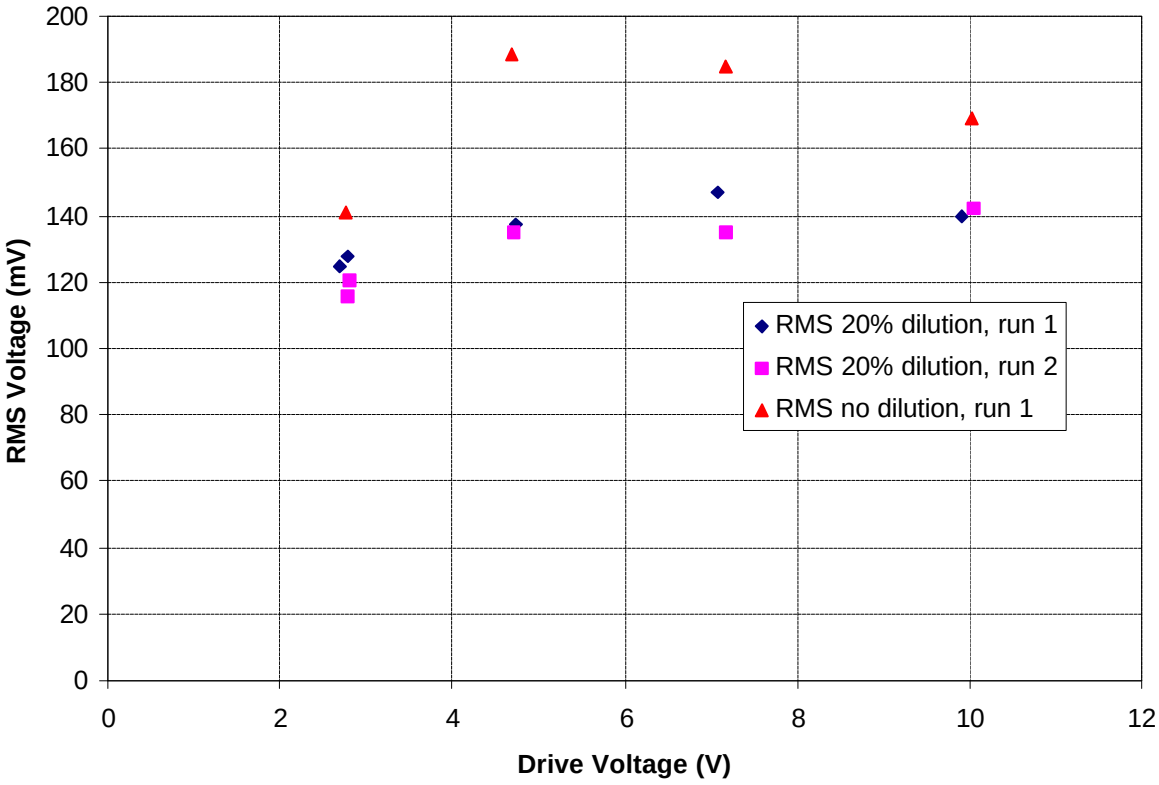

Figure 6.30: *rms* voltages measured from PCS-11, two different polystyrene sphere concentrations

A repeat set of measurements at the same drive settings was completed straight afterwards, and the broadband power data for both data sets is shown in Figure 6.31. In this case, the second half of the data set was acquired in descending order. The ageing effect seen previously would appear to be manifesting itself here also, with descending data set (shown in red) showing higher levels of activity. In this case, as the second half of the run was performed in descending order, the 95% values show better agreement with each other, before the two trend lines deviate through the remainder of the runs.

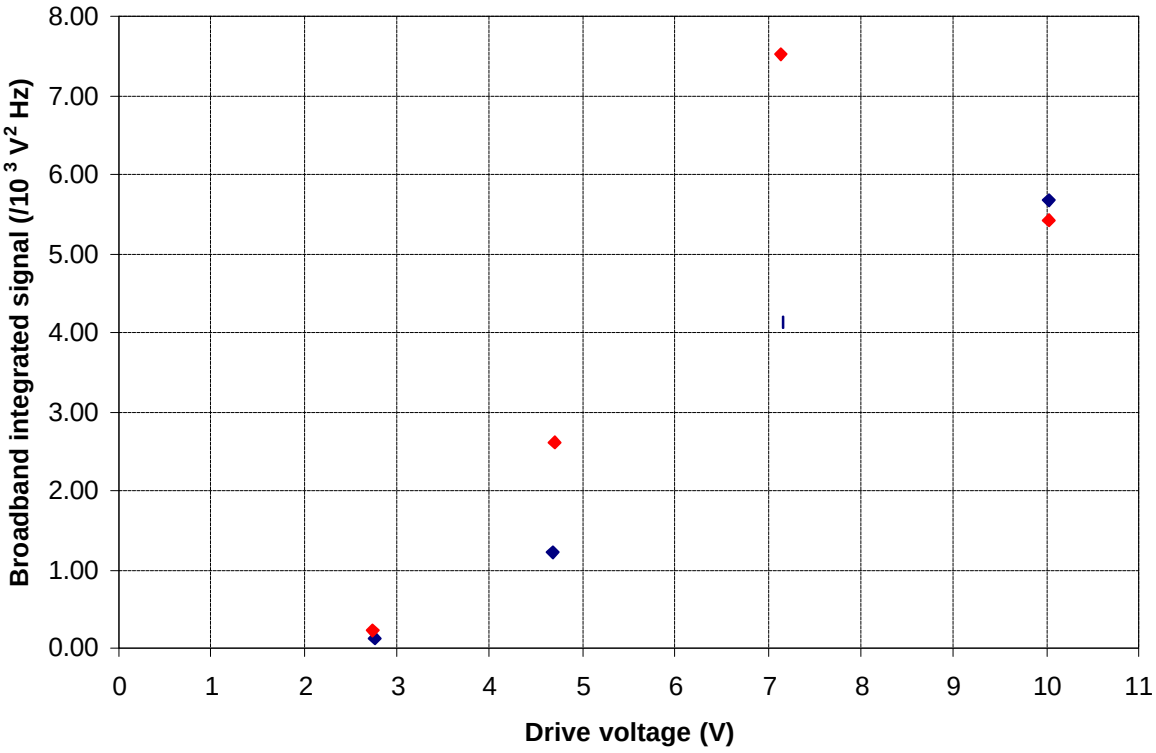

Figure 6.31: Broadband power over the full range of drive settings, ascending and descending data in neat 'master solution', PCS-11

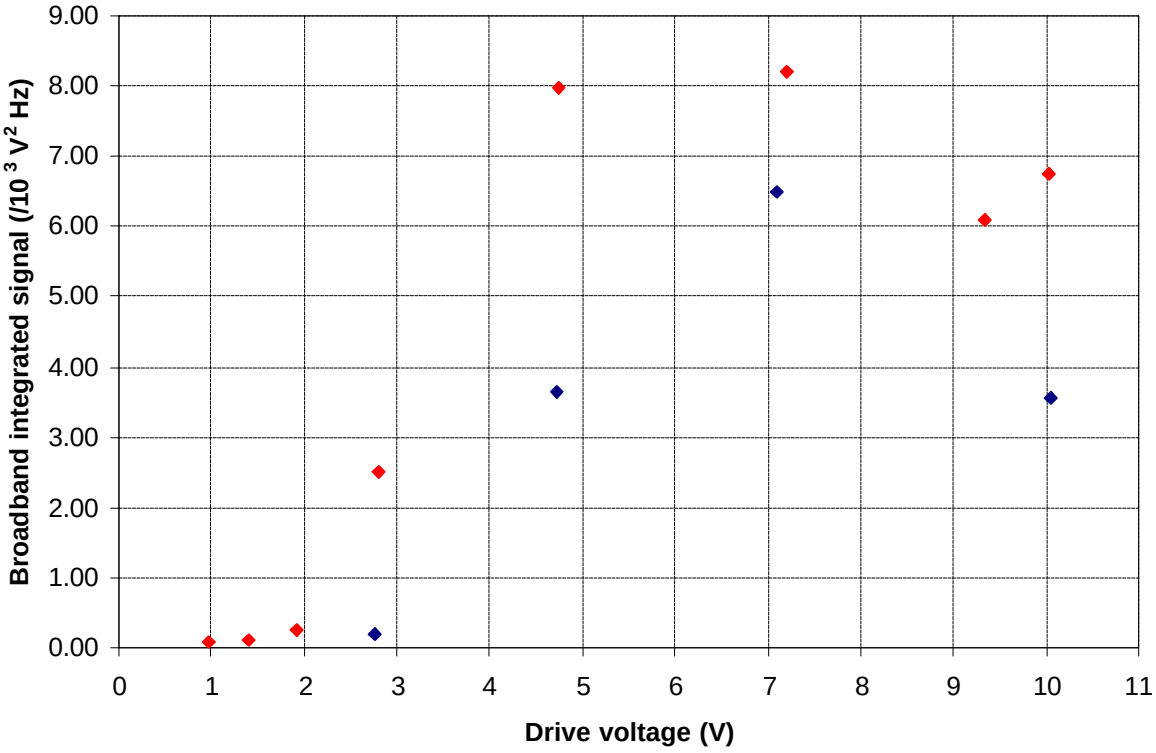

Figure 6.32: Broadband power over the full range of drive settings, ascending and descending data in neat 'master solution', PCS-11, second run

Using the same sample, another set of ascending and descending measurements were carried out straight after, producing the data shown in Figure 6.32. Again, the descending data set is shown in red. The agreement in levels between Figures 6.31 and 6.32 is good, and there is some evidence again of a hysteresis type effect, with the descending data in both cases showing characteristically higher broadband power levels. In both cases, the peak broadband power is seen at a drive level less than the maximum: however in the majority of measurements made using the "Analar water" alone (Figure 6.17), the trend in broadband power with drive setting is almost linear. It may be inferred thus that the apparent turnover seen repeatably for the 'neat' master solution in the bag, with Analar water used as the coupling medium, is a feature of the particle medium itself, with some form of local shielding perhaps occurring at the highest drive levels, effectively reducing the signal measured by the PCS-11.

Note the significant difference in the broadband signal levels when compared to the lower concentration measurements (see Figure 6.24) – the difference is approximately a factor of nine, which is consistent with the square of the three-fold change seen in the amplitude spectra. This significant change can probably be ascribed simply to the higher number of microspheres in solution, which will produce a larger number of sites from which cavitation may initiate. The comment was made previously that there was no significant difference in the measured broadband power levels for 20% concentration when compared to the "AnalaR water" sample. The higher value seen for broadband power for this greater concentration shows that at higher concentrations, the microspheres do produce a substantial effect. The increase in levels is not directly proportional to the change in concentration, however.

6.4.7 Discussion of particle studies

Although carried out on a limited basis, the experiments completed on the polystyrene microspheres showed some interesting results. The brief investigation of the change in concentration had a marked effect on the broadband power data, and so it appears that the particles have some form of nucleation effect. The experimental arrangement, whilst appearing to have had a small effect on the measured cavitation activity when using "AnalaR water", is certainly different to that used for most other studies in the project. It is expected that the presence of the bag as a mechanical barrier to the replenishment of nucleation sites does have an effect, particularly when looking at the longer-term repeatability of cavitation activity in a medium.

The trend of the increasing and then possibly decreasing levels of cavitation activity with 'dose' may be related to the use of the bag, although deagglomeration and particle size changes may also be of relevance. Where repeat measurements have been performed, the use of the microspheres has shown quite good reproducibility, and so it is thought that further measurements using particles with a wider scope should be considered for future investigation.

7 PHASE 3: COMORAC

During the initial project meeting held at ISVR, plans for the COMORAC (Comparison of Measurements of Reference Acoustic Cavitation) meeting were discussed in detail. In total, five centres within the UK were approached. All expressed a willingness to participate. Additionally, due to opportune timing, a researcher from Belarus State University took part in some of the trials. They had been one of the participants in a European Union INTAS project to develop an acoustic emission based cavitation meter.

7.1 RATIONALE

The original aim of COMORAC was to use the candidate cavitation reference materials (described in Section 6), within the reference cleaning vessel, with various detectors and sensors supplied by key centres within the UK. Each participating researcher was asked to devise one or more detection techniques that gave a quantifiable parameter to associate with a given level of cavitation. The comparative performances of these techniques when presented with a given set of cavitation conditions at NPL (produced by the reference vessel) were then assessed.

7.2 COMORAC PROCESS

A complete comparison study was clearly beyond the scope of the current project, and it was originally envisaged that the COMORAC process would essentially consist of a feasibility test carried out over a period of one or two days. However, due to the interest shown in the project by the participants, and the number of different techniques they wished to apply, COMORAC took place over a period of 8 days, 6 of which were spent at NPL, during the period 17 August to 27 September 2000. The additional 2 days of testing took place at ISVR, where a facility producing single-bubble sonoluminescence was used to test the response of two techniques prior to their deployment at NPL. The time period over which measurements were carried out was not ideal, especially as severe problems in scheduling suitable time-slots arose due to the UK fuel crisis.

7.3 COMORAC REALISATION

A full report of the measurement techniques, protocols adopted and results obtained will be published shortly by ISVR.

8 DISCUSSION

During this project, the questionnaire played a key role in obtaining up-to-date views from the scientific community. The questionnaire was restricted to leading-edge researchers in the area of cavitation detection and monitoring, and the high response rate (70%) is probably indicative of the importance attached to the project by the recipients. Approaching the relevant experts ensured that the quality of technical information was high.

It has also been important to be flexible within the project. As commented previously, the original project plan involved developing a scoring system through which the various options for the reference medium could be evaluated and the most promising candidate subjected to limited experimental study. However, it became clear that the number of possibilities for such a medium were limited, and the implementation of this rigorous process would not be worthwhile. Probably for reasons of expediency, respondents chose to use aqueous-based systems for their reference liquids. This fact indicated that the studies within the current project should be targeted towards a water-based host system.

The experimental studies, both of the candidate materials and the technology transfer activity (COMORAC), played an essential role the project. It needs to be underlined that these tests were limited in scope but, nevertheless, were key in feeding into the recommendations given in Section 9.

The limited scope of the measurements, in keeping with the 'feasibility' nature of the project needs to be borne in mind when drawing definitive conclusions from the experimental work carried out. Nevertheless, the overall remit of the project was to establish the feasibility of developing a reference medium for cavitation. The results obtained, particularly on the 'standard water' samples, have established that such a medium is technically feasible. The degree of agreement between different samples of water was typically ±14%: a very encouraging level, considering the lack of metrology and historical difficulties in this area of measurement. A few comments are appropriate when assessing these results:

- The reference cavitating medium, as commented by a number of questionnaire respondents, forms only one component of the overall system comprising the applied acoustic field, the cavitation medium and the detector. The level of agreement observed for different samples of nominally identical specimens of water implies that the applied field and the detector are reasonably stable. It needs to be emphasised that each of these two components is at a relatively early stage of development. The cavitation sensor (PCS), used to characterise cavitation emissions of the reference medium has been the product of an NPL Strategic Research project - prototyping of the sensors has just been completed, and experience is now being garnered about the response of the devices in characterising cavitation. Similarly, the reference cleaning vessel has only recently been developed with NMS funding and has yet to be fully characterised. As described in Section 3, at its core lies a commercially available ultrasound cleaning vessel rather than a custom made specialist facility: it might be envisaged that a bespoke system would offer improved performance;

- In using the PCS to characterize the cavitation signal, one particular means of cavitation detection has been used, based on the observation of acoustic emissions from bubbles. Furthermore, a particular feature of the acoustic spectrum has been

examined – the broadband integrated signal in the frequency range 1 MHz to 5 MHz – as being indicative of violent inertial cavitation. Other spectral features, such as the half-harmonic, might conceivably have some utility. Even more important is the need to evaluate the efficacy of water samples using other monitoring techniques, of the type used with COMORAC, based on optical and chemical detection.

Despite these comments, the results from the standard water studies are encouraging. What is of interest is the very different behaviour of the two types of water: the ”water purified” sample has a particulate content of 2 ppm, and, as shown in Figure 6.11, the broadband power level attains a maximum value of 2.4 x 10^3 V^2 Hz at 65% power, before decreasing to 1.6 x 10^3 V^2 Hz at 95% power. This turnover has been seen in a range of measurements carried out using the PCS in situations where the dissolved gas content approaches saturation level.

The conventional interpretation of the decrease in cavitation level has been that its origin lies in cavitation shielding. In contrast, the “AnalaR water” samples contain only 1 ppm particulate level, and the ‘broadband integrated signal’ increases virtually linearly over the indicated power range, achieving 1.4 x 10^3 V^2 Hz at 85% power (Figure 6.16). It should be noted that both water samples showed a similar DO_2 level, determined using the Hanna meter – approximately 7.8 to 7.9 ppm. It seems reasonable to think that this different behaviour arises from the differences in the composition of the two samples, including both dissolved chemicals and suspended particulate. It would also tentatively suggest that for these studies, the medium is the most important source of cavitation nucleation sites, rather than impurities from the vessel or the atmosphere. One final point to bring out regarding acoustic spectra generated by the two types of water lies in the fact that the “AnalaR water” results appear to have a greater number of distinct harmonics (Figure 6.13), perhaps indicative of stable (non-inertial) cavitation, although the significance of this needs to be studied further.

Again, due to the limited nature of the measurements made on the standard water samples, it is clearly of interest to extend the measurements to include further specimens obtained from BDH, or from other sources, potentially including other particulate levels, outside the two similar levels for the samples tested here. The samples of water, both “purified” and “AnalaR” were each taken from the same batch, so it would clearly be of interest to establish the effect of the ‘batch-to-batch’ variation on the cavitation activity . Furthermore, it would be useful to approach the supplier to establish the likely variation in particulate level between batches.

It is worth bearing in mind that this degree of agreement for measurements of cavitation is surprisingly good, and indeed goes against a number of views of the questionnaire respondents, as highlighted in Section 5.2. Some respondents felt that there are simply too many variables which affect cavitation for it to be reproducible. This study appears to show that, providing the three components of the overall system are controlled, then a degree of cavitation repeatability can be achieved. It is worth noting that a number of respondents work in medical diagnosis, using MHz ultrasound. It is fairly certain that this situation differs significantly from the cleaning vessel considered here. For the diagnostic configuration, the likelihood of cavitation depends on the suitable nucleation site being positioned at the correct point within the acoustic field, which in many instances may be a focused region of small volume. The likelihood of cavitation therefore takes on some of the attributes of a random process. In contrast, because the ultrasonic cleaning vessel contains a large volume of fluid and produces an acoustic field with both modal and diffuse

characteristics, and as the time-averaged intensities of the acoustic field are so high, cavitation will undoubtedly occur throughout the majority of the vessel.

A suggestion made by a couple of the questionnaire respondents was that rather than concentrate on developing a reference cavitating medium, effort may be more fruitfully applied to the development of a validated cavitation sensor. The results of this project have shown that there is distinct benefit in proceeding with two separate such studies in parallel. Both the standard water studies and the COMORAC results have been valuable in obtaining some degree of confidence in the PCS results, notwithstanding the comments made regarding sensor erosion which were made earlier.

The measurements made by seeding the background medium with 10 μm diameter hydrophobic polystyrene spheres, were more ambiguous. Deng et al. (15) similarly doped samples of an aqueous based background with 0.1 μm diameter hydrophobic polystyrene spheres, the size difference from the current study being a consequence of the MHz frequencies of interest. The paper stated that the background medium used should ideally be completely non-cavitating at any of the applied acoustic conditions. For the 40 kHz cleaning vessel studied here, this is clearly not possible, and it is likely that the results obtained for some of the diluted microsphere solutions are the result of the degassed water used as the background matrix. It may be possible to suppress this background cavitation by increasing the viscosity of the medium. The most concentrated solution used within this report (corresponding to 7.8×10^5 microspheres cm^{-3}) clearly showed significant increases in the degree of cavitation. The resultant acoustic spectra showed significant differences from the standard water samples, with little or no harmonic signals seen. Again, the significance of this needs to be investigated, but it may indicate that inertial cavitation is exclusively produced from air trapped on the surfaces of the microspheres. Purely for information, the ranges of particle concentration used in the Deng et al. study were 9×10^7 to 9×10^9 microspheres cm^{-3}, i.e. above those used in the current study. Potential problems of working with artificial nucleation sites were highlighted in Section 5.3, in particular the effects of cumulative exposure causing damage to the microspheres. To a certain extent, the results of this study have borne this out, with the amount of cavitation activity depending on previous ultrasonic treatment of the solution to an extent not generally exhibited by standard water samples. Nevertheless, the microspheres merit further study as this may be the best way of generating reference samples with a range of cavitation activities.

Finally, it is worth making a few comments regarding the COMORAC programme of measurements. This constituted the first exercise of its type within the UK, and was very valuable in bringing researchers together. The final written report, to be published by ISVR, should stand as a valuable contribution to the technical field.

9 RECOMMENDATIONS

The project achieved its overall aim of establishing the feasibility of developing a reference cavitation medium. Of the studies carried out, the standard water samples appear to be the most promising, and these follow-up studies should be given highest priority:

- studies on “water purified” and “AnalaR water” samples should be repeated, when greater confidence has been obtained in the performance of the reference cleaning vessel. This should involve liasing with BDH to establish the physical and chemical nature of the water samples, along with the ‘batch-to-batch’ variation in their behaviour;

- the possibility of obtaining water samples with different levels of particulate should be investigated (outside the 1 - 2 ppm range examined here).

The above items should be considered for inclusion in the Acoustical Metrology Programme 2001-2004. Furthermore:

- standard water samples need to be tested using alternative cavitation monitoring schemes, probably through collaboration with Universities;

- the results of the research undertaken here should be fed into the International Electrotechnical Commission (IEC) TC87 “Ultrasonics” Working Group 3.

Of lowest overall priority:-

- careful studies should be carried out of the seeding of a cavitation medium with polystyrene microspheres. This needs to be done carefully, as potentially it could involve a vast number of measurements of varying particle size, concentration etc. A schedule for a series of targeted and careful measurements would need to be generated.

10 ACKNOWLEDGEMENTS

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APPENDIX A: BACKGROUND AND PROJECT RATIONALE

A.1 CURRENT STATUS

To date, the development and dissemination of UK standards for sound in water has been undertaken in essentially free field conditions and at sufficiently low acoustic pressure levels to avoid cavitation. There are, however, major industrial applications of high-power ultrasound such as cleaning, welding and materials processing and medical applications such as surgery and therapy, where cavitation can be an important factor. Over the last five years, these applications of high-power ultrasound have shown consistent annual growths of between 10% and 30%. The importance of high-power ultrasound was first highlighted in the UK in 1991 in a DTI-commissioned report, which identified it as being one of six 'hub technologies' whose implementation would lead to a cleaner environment. Since then has come a recognition of the need for reliable methods of measurement of high power ultrasonic fields in order that processes driven by such fields, and by cavitation, may be monitored, controlled and extended to industrial scale. Indeed, the strategic importance of developing reliable characterisation methods for high power ultrasound and acoustic cavitation was recognised within the Measurement Foresight Programme, where it was rated one of the top five requirements in the field of acoustics.

A.2 THE REQUIREMENT

The need to develop new characterisation techniques for high power and cavitating ultrasound fields was confirmed through an NMSPU-funded study undertaken in 1996 by NPL in collaboration with ISVR. During this study, a survey of the views of 70 experts worldwide was undertaken which demonstrated that industrial users require methods for quantifying high power and cavitating fields in order to:

- control, replicate and scale up industrial processes;
- ensure consistent quality of manufacture and production;
- enable the development of new products;
- control safety hazards;
- satisfy regulations;
- provide customer service.

The results of the survey have been summarised in three reports: (references: 1, 2, 3). The latter two reports respectively represent extensive reviews of cavitation monitoring and field measurement techniques. The first of these documents, referred to hereafter as the Study Report, presented the findings of a sectoral analysis of measurement requirements, covering the following market segments:

- medicine;
- sonochemistry;
- ultrasonic cleaning vessels;
- industrial (non-cleaning).

The Study Report showed that the whole area of high power ultrasound technology is one in which the demand for reproducible characterisation techniques is increasing, and is driven principally by the growing importance of quality systems such as ISO9000 and the attendant stringent demands these place on industrial process control. In particular, methods of

measuring or estimating the strength or violence of collapse cavitation were identified in the Study Report as a priority, and further, there was a need for methods of determining the spatial and temporal extent of cavitation activity.

Whilst the monitoring and potential measurement of cavitation in all of these areas is undoubtedly important, the Study Report proposed that a relatively low-level start be made in this new area of acoustic metrology. The aim was to develop an understanding of the physics of cavitation, although ultimately, the development of reliable characterisation methods would lead to a means of quantifying the ultrasonic cleaning process itself. It was proposed that this understanding would be facilitated by the establishment of a reference ultrasonic cleaning vessel.

A.3 THE CONCEPT OF A REFERENCE CAVITATING MEDIUM

Whether or not cavitation actually occurs in a medium will depend predominantly on the driving amplitude of the acoustic field and the existence of bubble nuclei, which may be stabilised in solution through attachment to small impurities in the fluid medium. Clearly, on this basis, cavitation is a difficult phenomenon to control in any realistic industrial environment. One of the findings of the Study Report was that further study of the effects and applications of cavitation would be expedited by the availability of a material in which cavitation was predictable, controllable and reproducible. This conclusion was reinforced by the Focus Group meeting held at NPL which discussed the formulation of the 1998-2001 NMS Programme for Acoustical Metrology.

One potential way of developing such a reference cavitation fluid would be to ‘seed’ a previously-purified medium with well-defined nucleation sites. It is not envisaged that this material would be used within the cleaning medium to enhance or control the cleaning process but rather that it could be used as a standard medium, in conjunction with other sensors or combinations of sensors.

A.4 ULTRASONIC CLEANING SYSTEMS

As outlined in Section A.2, the Study Report advocated a fairly low-level start in this new area of metrology. The specific application area of ultrasonic cleaning was chosen partly due to its industrial importance, which can be appreciated when the market information is considered. There are over 20 manufacturers and suppliers of equipment within the UK. The value of the UK market alone is estimated to be in the range £50 to £70 million per annum, and this forms part of a world market estimated to be £1.5 billion per annum. The turnover at the ‘user’ end will be very much larger and it is the users who will benefit from the new metrology capability, enabling processes to be applied and controlled more readily.

A.5 SUMMARY

The need for measurement methods that are able to characterise reliably high power/cavitating ultrasonic fields has recently been acknowledged during an NMSPU-funded study (Reference 1). Certain key recommendations of the report have begun to be implemented under the framework of the 1998-2001 NMS Acoustical Metrology Programme, and this primarily involves the setting up of a reference ultrasound cleaning vessel at NPL. In order to facilitate the study of cavitation occurring within the vessel, it is of benefit to establish a cavitation medium which is predictable, controllable and reproducible.

To this end, a feasibility study was carried out into establishing a reference cavitating medium. This report presents the findings of this study.

APPENDIX B: QUESTIONNAIRE DISTRIBUTED TO CONTACTS

The questionnaire is presented in the exact form supplied to the experts selected.

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High power ultrasound and acoustic cavitation are important across a wide range of application areas, from industrial processing through to medicine. Whilst significant progress has been made in recent times to understand the mechanisms responsible for the effects seen, there is still a lack of standardised characterisation techniques in this technically-demanding area. In 1996, the National Physical Laboratory (NPL), UK, in collaboration with the Institute of Sound and Vibration, UK, was commissioned by the UK Department of Trade and Industry (DTI) to produce a Study Report, examining the need for standardisation of measurement methods for high power/cavitating ultrasound fields. The results of this project, which included views from over 40 experts worldwide, demonstrated that there is a significant need for standardisation. Based on these findings, DTI has commissioned two further projects in the area of high power ultrasound. The first of these is currently being carried out at NPL, where an ultrasonic cleaning vessel is being used as a reference cavitation source (*in the words of Apfel - "know thy sound field"*). The second project is another Study Report, this time examining the feasibility of establishing a reference cavitating medium (*"know thy liquid"*). The project objectives are:

to gather information from learned experts and the technical literature relating to cavitating media;
to analyse this information and identify candidate reference materials;
to complete some initial experimental studies on these candidate materials in conjunction with the NPL reference ultrasonic cleaning vessel.

In the information-gathering phase, NPL is contacting a number of recognised experts in the area of high power ultrasound and cavitation, and requesting their views on the establishment of a reference cavitating medium. The questions which follow relate to your expertise in this area.

Name:

Organisation:
Address:

1. Please describe your background, and your interests in the area of high power ultrasound and cavitation. Is cavitation a requirement of your process, or an unwanted occurrence?

2. How would you interpret the term 'reference cavitating medium'? What properties of the medium would be most relevant to your application area?

3. Have you used, or do you use, any form of reference cavitating medium? *(if your answer is NO, then go to 5)*

4. How was this applied? How did it perform? *(Are there any publications/technical reports which describe the results of using such a material?)*

5. Do you consider that there is a need for a reference cavitating medium? Is this an immediate requirement or of future importance?

6. How might such a medium be applied and used?

7. What capability would it provide? How would it benefit your application?

8. What sort of materials would be suitable? How would the medium be realised?

9. Would the medium be process-specific? Why and how?

10. Please add any further comments below:

Thank you for your input to this project - your contribution is greatly appreciated.

APPENDIX C: LIST OF QUESTIONNAIRE RECIPIENTS

Name	Institution
Gareth Price	University of Bath
Steve Pye	University of Edinburgh
Ken Suslick	University of Illinois
Ron Roy	Boston University
Rob Hekkenberg	TNO, Leiden, Holland
Bob Apfel	Yale University
Larry Crum	APL, Washington
Tim Mason	SNES, Coventry University
Andy Coleman	Guys and St Thomas' Hospital
Wesley Nyborg	University of Vermont
David Hunicke	ASP Systems Inc.
John Fuchs	Blackstone Ultrasonics Inc.
Ron Manna	Misonix Inc.
Alan Broadwin	Ainslie
Tim Leighton	ISVR
Adrian Richards	CSIRO
Brad Barber	Bell Labs Inc.
Gary Ferrell	L-tech Corp.
Charlie Church	Acusphere Inc.

APPENDIX D: COMORAC PROTOCOL DOCUMENT

D.1 MEDIUM TO BE USED FOR COMPARISON

The BRANSON cleaning vessel must be filled to the same mark (the middle of the horizontal flange within the bath positioned about 70 mm from its top) with filtered water from the supply unit. Degassed water should not be used.

(Note: the BRANSON bath has been shown to be less stable when operating with highly degassed water (DO_2 < 1 ppm) and so the system should be run with a level within the range 2 to 3 ppm. This will have to be generated using a mix of filtered, degassed and undegassed water – 50:50 mix should be adequate.)

D.2 MEASUREMENT OF WATER PROPERTIES

The dissolved oxygen content of the water and its temperature must be measured using the HANNA HI 9145 meter. This must be completed both at the beginning and end of the measurement runs.

(Note: the specification on the accuracy of the HANNA HI 9145 temperature measurement is only ±0.5° C so strictly this is not the most accurate means of temperature measurement. However, to speed measurements up, this figure will be used and corrected once the sensors had been checked using a calibrated thermometer).

D.3 SETTING DRIVE LEVELS

The drive voltage to the BRANSON vessel and the resultant acoustic power levels, are adjusted using the LD3205 regulated dual dc power supply. The I/O card is wired through the SIB1 box used to switch the ultrasound ON and OFF, and generates a dc output signal related to the electrical power being delivered to the twelve transducers in the cleaning bath. This dc output voltage is measured using an ISOTECH digital voltmeter. Guideline figures for the relationship between the power supply setting, the resultant dc output voltage and the electrical power (expressed as a % of the maximum output power), are shown in Table D.1. Settings tabulated in Table D.1 should be regarded as reference conditions for reproduction under the various tests which make up COMORAC.

D.4 POSITIONING OF SENSORS

Unless agreed otherwise, the position of the sensors should be arranged such that they are disposed centrally within the tank. Although some automatic manipulation is possible using stepper motor control, this positioning will have to be done largely ‘by eye’.

Table D.1: Tabular summary of the relationship between the power supply voltage set and the resultant power delivered to the BRANSON cleaning vessel, expressed as a percentage of the maximum power

Power supply voltage setting (V)	DC output voltage level (V)	Nominal power level (% of maximum)
9.6	1.0	5%
9.3	1.5	10%
9.0	1.9	15%
8.4	2.8	20%
7.0	4.8	40%
5.0	7.1	65%
3.1	9.3	85%
2.5	10.0	95%

(Note: in general, if sufficient time is available, measurements should be attempted at all eight settings. For some techniques, however, it may be necessary just to carry out measurements at one or more specified drive settings).

D.5 NPL PCS CAVITATION SENSORS

The comments below are relevant to the use of the cylindrical cavitation sensors:

D.5.1 Earthing

It is essential that the sensor is adequately earthed, through linkage of the earth braid to the :

- outer casing of the BRANSON cleaning vessel;
- input BNC of the KENELEC (buffer) amplifier;
- earth connection to the steppers.

It is also critical that all measurements using the cavitation sensors are made with the stepper motors OFF.

The 'noise-level' (the sensor output determined with the ultrasound OFF), provides a sensitive test of whether or not these connections have been made adequately. Using the HP 3589A spectrum analyser, determine the 'noise background' under the following conditions:-

- frequency range 1 to 5 MHz;
- B/W = 1200 Hz
- number of averages = 40 (takes 70 seconds)

The resultant background 'noise-level' should be –102.5 ± 0.5 dBm. If it is any higher, then it is highly likely that the earth connections made above are inadequate (a common problem, if

the connection is not made secure enough, is for the earth-braid to come away from the casing of the cleaning vessel due to the vibration).

(Note: in restricting the frequency range between 1 and 5 MHz, some of the high frequency 'energy' is lost using the sensors. For the time being, the loss is being accepted in the interests of obtaining better quality, more repeatable data, which requires a good frequency resolution and large number of averages).

D.5.2 A measurement 'set'

During COMORAC, a typical run with the NPL sensor will involve spectral acquisitions at each of the eight powers given in Table D.1, with the data being stored for later processing. This will take between 20 to 25 minutes.

For consistency of protocol, power measurements must be made in order of increasing drive levels, switching OFF (for two minutes) in between measurements.

After the completion of a set of measurements, the HANNA HI 9145 dissolved oxygen meter must be used to record the final DO_2 and temperature readings.

D.5.3 Measurement of cavitation sensor *rms* output signal

In addition to measuring the frequency spectrum over the range 1 to 5 MHz, the *rms* output signal level of the sensors must be recorded. This is measured using the Racal-Dana 5002 *rms* meter. The signal is related to the direct acoustic field generated at 40 kHz and represents a consistency check for the performance of the cleaning vessel.

(Note: this can be determined simultaneously, by teeing off from the at HP 3589A analyser and having a 50 Ω at the input of the Racal Dana 5002 *rms* meter).