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THE MEASUREMENT OF BOUND AND FREE MOISTURE IN ORGANIC MATERIALS BY MICROWAVE METHODS

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ABSTRACT

Bound and free moisture can be classified by energetic, structural, or operational schemes. We discuss these schemes and consider four methods [dynamic dielectric thermal analysis, microwave attenuation analysis, near-infrared reflectance analysis (NIRA) and nuclear magnetic resonance (NMR) spectroscopy] that have been suggested for distinguishing between bound and free moisture in organic materials. This report describes the microwave attenuation method. The theoretical basis for using microwaves for this purpose is developed. We show that microwave measurements can be used to measure the moisture in triaminotrinitrobenzene (TATB) using a microwave network analyzer down to 0.04% H_2O. The design of the apparatus necessary to extend these measurements to lower moisture content limits is mentioned. As part of this study, we measured the dielectric properties of TATB for the first time. We found that the dielectric constant ε' for TATB was 4.00 ± 0.01.

INTRODUCTION

Moisture and its effects are important in preserving the integrity of our stockpiled nuclear weapons. Water is primarily introduced as a component of the many organic materials such as explosives, plastics, cushions, etc., that are used in these devices. Of most concern is the moisture whose bonding is sufficiently weak that it can become "free" in or "available" to the interior of the nuclear weapon during its stockpile life. This free moisture can promote the corrosion of critical parts or lead to the production of hydrogen which is deleterious to system electronics.

The states of moisture can be classified in three ways: (1) energetic, and (2) structural and (3) operational [1]. In the energetic definition, water is classified according to its binding energy to the host material. This results in a continuous scale from 5 Kcal/mole (water interacting with itself in the liquid state) up to 100 Kcal/mole (the disproportion energy of simple molecules). Figure 1 summarizes the most complete structural classification scheme which we are aware of [1]. It is apparent from these two methods of classification, that calling moisture either bound or free is imprecise. In a practical sense, however, an operational definition such as "free moisture is the water that can be driven off by heating the substance at 110°C for one hour in air at atmospheric pressure" is sufficient since a complete study of the binding state of water would take too much time and consume too many resources. In the case of nuclear weapons, the operational definition is "free" moisture is that water which can become free to the interior of the weapon over the temperature range -55 to 70°C during the stockpile life of the weapon.

Four methods of distinguishing between bound and free moisture have been suggested [1]. They are: (1) dynamic dielectric thermal analysis (DDTA), (2) microwave attenuation analysis, (3) near infrared reflectance analysis (NIRA), and (4) nuclear magnetic resonance (NMR) spectroscopy. We have evaluated all four methods. We were unable to detect the effect of moisture in TATB or its plastic bonded derivative using DDTA. The NIRA method is a secondary method and was thus not studied extensively. The NMR method is described in a separate paper in this symposium [2]. This paper reports the microwave attenuation results.

In general, RF dielectric spectroscopy in the frequency range 50 to 1300 MHz is an excellent method for detecting small quantities of water in solids. The high sensitivity of the technique is based on the contrast between the relative permittivities (dielectric constants) of water (about 78) and the dry host medium (about 4). For example, simple mixture theory predicts that the relative permittivity of the above host material with 1% water will be increased to about 5. Thus 1% of water can elevate the permittivity by 25%! Furthermore, there is evidence that absorption of electromagnetic energy in most organic materials peaks in the frequency range of 0.1 to 1 GHz [3], whereas, the absorption in free water peaks at about 17 GHz. This peaking at the lower frequencies is chiefly attributed to Debye relaxation of the bound moisture and its bonding with the lattice of the host material, while absorption at higher frequencies is chiefly

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determined by a resonance phenomenon involving rotation of the free water molecule. In principle then, it should be possible to distinguish bound and free water by measuring the loss of an organic material at the UHF and microwave frequencies where the effects of bound and free water differ. Note that at frequencies lower than the UHF or microwave range the dielectric spectroscopy method is only sensitive enough to measure the effect of the increase in real dielectric constant, but is not sensitive enough to distinguish the frequency behavior of the losses. Hence, at lower frequencies it cannot distinguish between bound or free moisture.

The theory of distinguishing between bound and free moisture using microwave absorption has been described by Busker [4] among others. Note that equations (5) and (6) in his paper are missing square root signs in the second term of the denominator and that he assumes in equation (8) that dB/dW is equal to rather than proportional to the bound water.

THEORY

The physical basis underlying the electromagnetic (RF through microwave frequencies) dielectric method for the determination of moisture content is essentially determined by macroscopic polarizability. As the frequency of the applied field is scanned, the internal structure responds by absorbing energy. Microwave absorption is sensitive to the material's polarizability which arises from orientational effects within the solid. If the system is relatively disordered, it will possess a smaller directional polarization. Practically, this difference is reflected in the frequencies at which the material responds, higher frequencies are necessary to stimulate a less polarizable material.

In the particular case of bound and free moisture, the difference in frequency response may in principle be used to ascertain the relative amount of ordered or disordered water. If the water is intrinsically connected to the structure of the host material it will display bound or "ice-like" frequency response at low frequencies. Conversely, if the water resides in small pockets without participating in any bonding to the host it will be disordered or "liquid-like", and will respond as liquid H_2O throughout the frequency domain, particularly around 17 GHz.

Since H_2O is a very polar material in either form, it should be observable against the background of the host (solid) material. The variable frequency measurement technique relies upon the difference in power attenuation between the dry and wet material induced by the presence of the highly polarizable water molecules. Furthermore, the relative ordering of the water molecule themselves within the material may be detected by the attenuation between free H_2O and bound H_2O if the measurement is sufficiently sensitive.

The theoretical description of the experiment is very well established. An electromagnetic wave scatters from a material of a certain geometry. This wave has the qualitative form

$$\psi \sim \exp(\alpha + i\,\beta)$$

where α is the attenuation constant of the wave and β is the phase shift. The power attenuation is measured by the exponential loss in this function which is related to the so-called loss tangent

$$\tan\delta = \varepsilon''(\omega)/\varepsilon'(\omega)$$

$$= \alpha/\beta$$

for $\tan\delta \ll 1$. This expression is related below to the experimentally measured attenuation. ε' and ε'' are the real and imaginary parts of the dielectric function. Both are functions of the applied frequency.

The explicit expression for power attenuation as a function of frequency is given by

$$S(\omega) = aX\omega\ \varepsilon''(\omega)/\sqrt{\varepsilon'(\omega)}$$

where a is a material constant and X is the sample thickness. As emphasized above, this quantity measures the power absorbed by the material from the electromagnetic field. In order to apply this expression several reasonable approximations must be introduced to compare with experiment in the frequency domain in which all the different species - dry material, free H_2O and bound H_2O - contribute ($10\ \text{MHz} \leq \omega \leq 1\text{GHz}$).

First, the attenuation of the material with included water may be written as the sum of the individual contributions (dry material, free water and bound water)

$$S(\omega) \approx S_m(\omega) + S_f(\omega) + S_b(\omega)$$

This approximation is reasonable if the H_2O has not chemically reacted with the material to produce a new substance with dielectric properties unrelated to the previous ones. Second, the materials are assumed to be uniformly dispersed throughout the sample so that $X_i = M_i/(A\rho_i)$ where M_i is the mass of substance i, A is the area of the sample, and ρ_i is the density. For free and bound water, $\rho_f \approx \rho_b$. Third, the dry material is assumed to have a constant dielectric function with imaginary part $\varepsilon''_m \approx 0.02$ and real part $\varepsilon'_m \approx 4.0$. These values are taken from the experimental work in this paper. Fourth, the dielectric function for free water is given as a function of ω by

$$\varepsilon'_f(\omega) = 5 + \frac{75}{1 + (b\omega)^2}$$

$$\varepsilon''_f(\omega) = \frac{4.7\times10^{-9}}{1 + (b\omega)^2}$$

$$b = 6.3 \times 10^{-11}$$

and that for bound water as

$$\varepsilon'_b(\omega) = 88 + \frac{.9}{1 + (\omega \cdot 10^{-8})^2}$$

$$\varepsilon''_b(\omega) = \frac{9.0\times10^{-9}\omega}{1 + (\omega \cdot 10^{-8})^2}$$

These expressions fit the known values for liquid water and ice. The basic assumption consists of equating the included free and bound H_2O by liquid H_2O and ice. This approximation is probably valid within the confines of the measurement and is consistent with the other approximations above. These assumptions lead to the following expressions

$$S(\omega) \approx S_m(\omega) + S_f(\omega) + S_b(\omega)$$

$$\approx \frac{a\omega}{A}\left(\frac{\varepsilon''_m}{\sqrt{\varepsilon'_m}}\frac{M_m}{\rho_m} + \frac{\varepsilon''_f}{\sqrt{\varepsilon'_f}}\frac{M_f}{\rho_f} + \frac{\varepsilon''_b}{\sqrt{\varepsilon'_b}}\frac{M_b}{\rho_b}\right)$$

with $\varepsilon_m \approx 4 - i(.02)$ and $\varepsilon_f(\omega)$ and $\varepsilon_b(\omega)$ given above.

To complete the analysis it is necessary to examine the limits of small and large ω. Below 10 MHz, $\varepsilon'(\omega) \approx$ constant and $\varepsilon''(\omega)$ is given by constant/ω where the ionic conductivity of liquid H_2O dominates [3]. Above 1 GHz, $\varepsilon(\omega) \approx \varepsilon_f(\omega)$. That is, the frequency response is independent of the dry material properties above 1 GHz and only depends upon the free water present. The behavior expected of the loss tangent over the entire range may be summarized as

$$\varepsilon''(\omega)/\varepsilon'(\omega) \approx \text{constant}/\omega \qquad \omega \leq 10 \text{ MHz}$$

$$\approx \varepsilon''_m/\varepsilon'_m + \varepsilon''_f/\varepsilon'_f + \varepsilon''_b/\varepsilon'_b \qquad 10 \text{ MHz} \leq \omega \leq 1\text{GHz}$$

$$\approx \varepsilon''_f(\omega)/\varepsilon'_f(\omega) \qquad \omega \geq 1\text{GHz}$$

Two practical issues may be considered: the moisture determination in the sample and the differentiation of the moisture into free and bound contributions. Specifically, the experimental limitations on these quantities may be addressed. First, the resolution needed to evaluate the moisture content of the material follows from the approximate expression for $\varepsilon_f(\omega)$ above since the free water dominates all contributions over a large portion of the frequency domain. That is, $\varepsilon''_f/\sqrt{\varepsilon'_f} \approx 0.3$ while $\varepsilon''_m/\sqrt{\varepsilon'_m} \approx 0.01$ for 100 MHz $< \omega <$ 1 GHz so that an error of 0.05 in the loss tangent measurement will not affect the qualitative observation that moisture is present. On the other hand, differentiating free and bound water is more difficult. The region in the frequency domain which has the most pronounced difference between free and bound water is 100 MHz $\leq \omega \leq$ 1 GHz with a maximum at about 500 MHz. Even at this frequency, the expressions for ε_b above yield $\varepsilon''_b/\sqrt{\varepsilon'_b} \approx .02$ which is essentially indistinguishable from the dry material contribution in this frequency range. The loss tangent measurement must be about a factor of 10 more accurate to distinguish the bound water from the background, especially if $M_b/M_f \ll 1$.

The brief analysis above indicates that the presence of moisture in the sample should be observable over most of the frequency domain. The separation of free and bound components presents more stringent experimental requirements.

EXPERIMENTAL

The explosives used in this study were triaminotrinitrobenzene (TATB) and a plastic-bonded derivative LX-17 which consists of 92.7% TATB with 7.3% Kel-F 800 binder. (See Table 1 for a comparison of the material properties.

Table 1. Material Properties of TATB and LX-17

	TATB (Lot B474)	LX-17-0 (Lot B789)
TATB, Wt. %	100	92.7
Kel-F 800, Wt. %	0	7.3
Particle size	See Fig. 2	See Fig. 2
Surface area, m^2/g	0.513	0.530
density, g/cm^3	1.888	1.909
density, % theoretical maximum	97.2	98.2

The partial size distribution for each lot of TATB is given in Figure 2. The hollow cylindrical samples of each were machined from billets pressed to high densities. Figure 3 shows the dimensions of the machined samples which had to fit snugly in the coaxial sample holder, and also the schematic diagram of the experimental setup. After machining, a cylinder of each material was vacuum dried for 48 hrs. at 80°C, weighed and the dielectric properties measured. Next, each cylinder was exposed for 144 hrs. to 85% humidity. The TATB sample gained 1.27% moisture while the LX-17 gained only 0.01%. Subsequently, the TATB cylinder was redried, reweighed and exposed to various humidities for varying lengths of time to give a range of moisture contents. The final composition of moisture was determined by weight difference in each case.

We used an Automated Network Analyzer (ANA) System to measure the complex scattering parameters when placed in a precision coaxial air line. The ANA system consists of a Hewlett-Packard (HP) 8505A Network Analyzer, HP 8503A S-Parameter Test Set, and HP 7245 Printer-Plotter, all controlled by an HP 9825 computer. The block diagram is shown in Figure 3.

The measurement procedure includes the following steps:

1. Specification of the frequency range and interval

2. The calibration of the network analyzer using precision standards. We determine values for each term in an error correction model from these measurements.

3. Measurement of the raw scattering parameters

4. Application of the error correction model to obtain corrected scattering parameters for the sample and holder.

5. Inversion of the scattering parametric data to obtain the dielectric properties.

The whole process is computer controlled including the printing and plotting of the data.

The objective of the calibration procedure is to produce a perfect measurement system having infinite dynamic range, isolation, and directivity characteristics, no impedance mismatches in any part of the test setup, and a flat frequency response. In practice, the "perfect" network analyzer is achieved by measuring the magnitude and phase response of known standards, using this data in conjunction with a model of the measurement system to determine error contributions, then measuring a test device and using vector mathematics to compute the actual test device response by removing the error terms. The dynamic range and accuracy of the measurement is then limited by system noise and the accuracy to which the characteristics of the calibration standards are known.

The error model described above can correct for systematic errors, those repeatable errors which the system can measure; not random errors such as those due to noise, temperature drift, and other physical changes to the test setup. Fortunately, in our measurement system the systematic category of repeatable, measurable error terms is the most significant source of measurement uncertainty. For the purpose of our error correction model, these uncertainties are quantified as effective directivity, source match, load match, isolation, and frequency response.

Consider, for example, the effective directivity. It is caused by the inability of the signal separating device within the network analyzer to absolutely separate incident and reflected waves, as well as residual reflection effects of test cables and adapters between the signal separation device and the measurement plane. This uncertainty usually produces the major ambiguity in reflection measurements. The interested reader may wish to pursue this subject in more detail [5].

The error correction scheme employed is an extension of the Hewlett Packard Accuracy Improved Measurement (AIM) method. We have changed the error correction model so that it includes 10 terms instead of 8. This improves accuracy when the reflection coefficient is low (in the low frequency range) when the sample length is a small fraction of the wave length.

To check the effectiveness of the error correction procedure, we measured the S parameters and calculated the dielectric properties for a standard sample of fused silica from the National Bureau of Standards (NBS) in Boulder, Colo. This material is well characterized and has a relative permittivity of 3.825 ± 0.67% with a loss tangent of $(5 \pm 5) \times 10^{-4}$. Comparison of our measured permittivity values for this standard with the NBS values shows acceptable agreement except at a few discrete frequencies. These are the frequencies for which the connecting cable and test set create resonances. It is impossible to remove these errors with our calibration procedure since perfect standards are required. In practice, these systematic errors may be shifted in frequency by using connecting cables of other lengths. Thus, it is possible to obtain permittivity values within the NBS limits by combining two data sets taken with different length cables.

Our measured values for the loss tangent of fused silica lie in the range $(1 \pm 1) \times 10^{-2}$ which is the lower limit of detectability for our ANA system. In order to get accurate values for very low loss materials like fused silica, one must use other methods (e.g., cavity resonators).

The deviations (+ and -) from the known value of the dielectric constant of fused silica were added to or subtracted from the measured values for TATB at each frequency. This procedure seems justified since the dielectric constants of fused silica and TATB are nearly identical (3.83 and 4.00), and the values for fused silica are well known. By doing this, we were able to remove the small but obvious errors which cannot be removed using AIM.

We calculated the relative permittivity and loss tangent of partially saturated and dry high explosive (H.E.) samples using the relationships derived by Nicolson and Ross [6] together with the error-corrected values of sample scattering parameters. The scattering parameters are just a generalized form of the familiar reflection and transmission coefficients[7].

Results and Discussion

The values of the dielectric constant for dry TATB and LX-17 are shown as a function of frequency in Figure 4. The dielectric constant is flat over the whole frequency range for both materials; $\varepsilon'_{TATB} = 4.00 \pm 0.01$ and $\varepsilon'_{LX-17} = 3.95 \pm 0.01$. There is no evidence for either bound or free moisture in the dried samples since the response at both low and high frequencies is flat. The precision shown is excellent after the data were corrected for systematic errors. The dielectric properties of TATB and its plastic bonded derivatives have not been measured previously [8].

Figure 5 contains all of the data measured for LX-17. The lower line shows the frequency behavior for the dry material. The dry LX-17 was exposed to 85% humidity for six days and only picked up 0.01% moisture. The crosses represent the total dielectric constant for the host material plus 0.01% water. The ε' for this barely moist material is 3.97 ± 0.02 which is statistically indistinguishable from the dry material. The top set of points (diamonds) represents the moisture (0.1%) picked up by LX-17 after being submerged in water for 34 days. Based on our experience with this one pressed and machined cylinder, this material is not very hygroscopic. From this data, we can infer that the detection limit of moisture in LX-17 is about 0.02 to 0.03% H_2O.

The characteristic low frequency effect on the combined dielectric constant for host plus water is illustrated for TATB in Figure 6. The curves appear to belong to a family, having the same approximate shape. Note that there is no evidence of a "bump" in the curve in the midpart of the frequency range as Meyer and Schilz [3] saw in a variety of organic materials and thus, no evidence for bound moisture.

Figure 7 illustrates the ε' versus frequency curves at the lower percentages of moisture. The lower limit of 0.04% moisture is clearly seen. Again, there is no evidence for a bump which would suggest the presence of bound moisture in this material. To show the utility of using this method for analyzing the free moisture in TATB, we selected two frequencies in the flat region of the curves and plotted ε' versus % H_2O. The resulting linear relationships are shown in Figure 8.

We conclude that this method can be used to measure the total moisture in TATB and LX-17 down to a limit of 0.04%. Its use is cumbersome, however, since the method requires precisely machined samples of the explosives.

We have looked at constructing an apparatus which would extend our measurements to lower moisture contents. In our assessment, the required precision can only be achieved with a very high Q resonator. The required accuracies of the order of 1-2% are only possible for resonators having Q-factors of the order of 10^5. Such high-Q factors are possible at the higher frequencies using an open spherical or hemispherical resonator and a source which is stable to one part on 10^9 in frequency.[9] However, such open resonators become prohibitively large, as does also the test sample size, in the 0.1 to 1 GHz range. The use of closed resonators to achieve higher sensitivity in either frequency regime seems to be out of the question since their Q is inherently only of the order of 10^3.

One of us [10] is developing an open-ended microwave resonator which shows promise of extending the measurement of total moisture down to the order of 0.005%. This method has the added advantage of being able to measure moisture in solid materials in situ.

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- Water in solids
  - Chemically bound water (water of crystallization)
    - Bonded water
      - Coordinated water – $MgCl_2 \cdot 6H_2O$
      - Lattice water – $CuSO_4 \cdot 5H_2O$
      - H_3O^+ water – $Ca(H_3O)_2(UO_2)(SiO_4)_2 \cdot 3H_2O$
      - Hydrogen-bonded water – $Na_2SO_4 \cdot 10H_2O$
      - Decomposition water – $Ca(OH)_2$
    - Zeolite water – $Be_2Al_2(SiO_3)_6 \cdot nH_2O$ (Beryl)
  - Physically bound water
    - Adsorption (surface only)
    - Liquid inclusion – bubble during crystallization
    - Absorption (structural uptake) – silica gel

Fig. 1. Structural classification of water in solids. Some examples are shown.

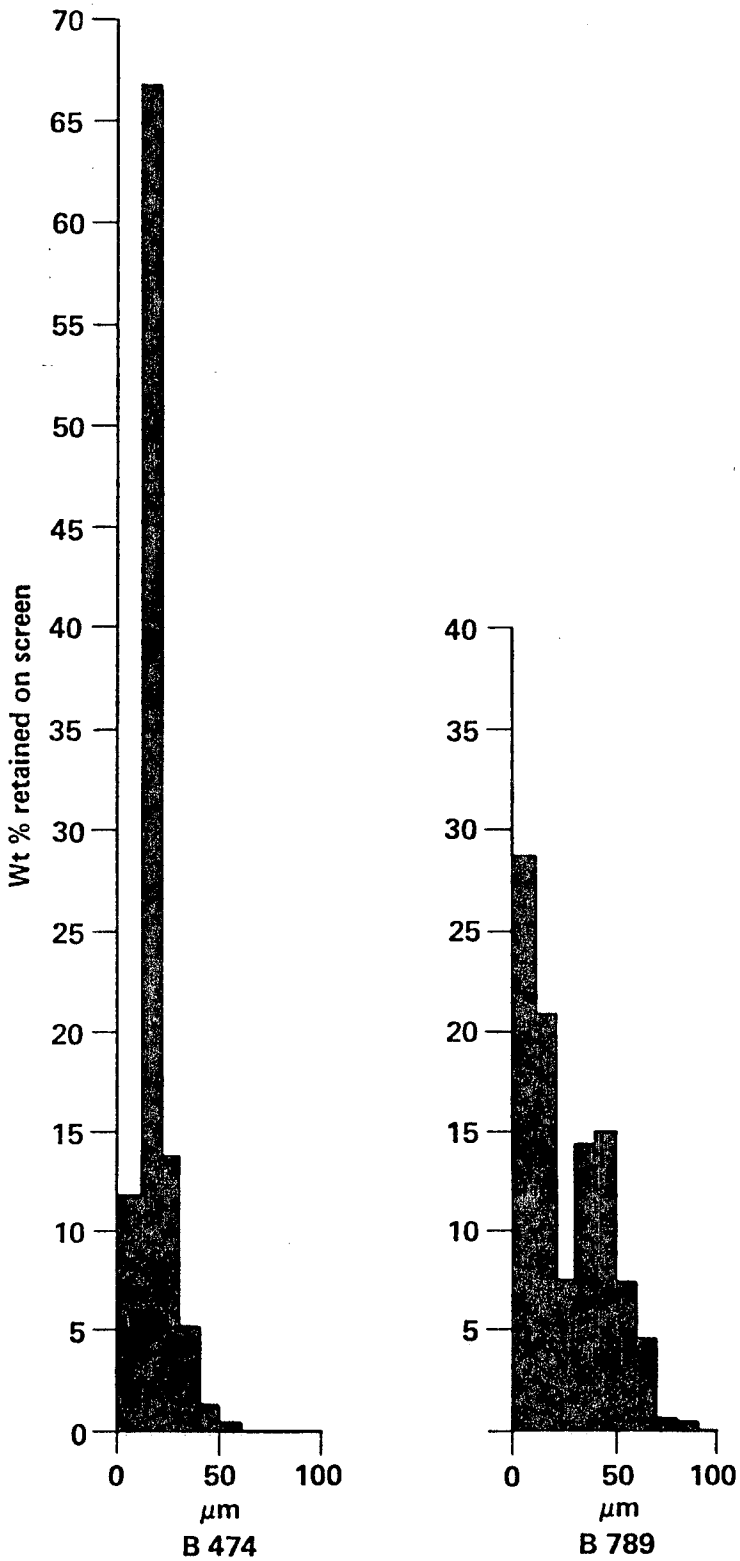

Figure 2. TATB particle size distributions

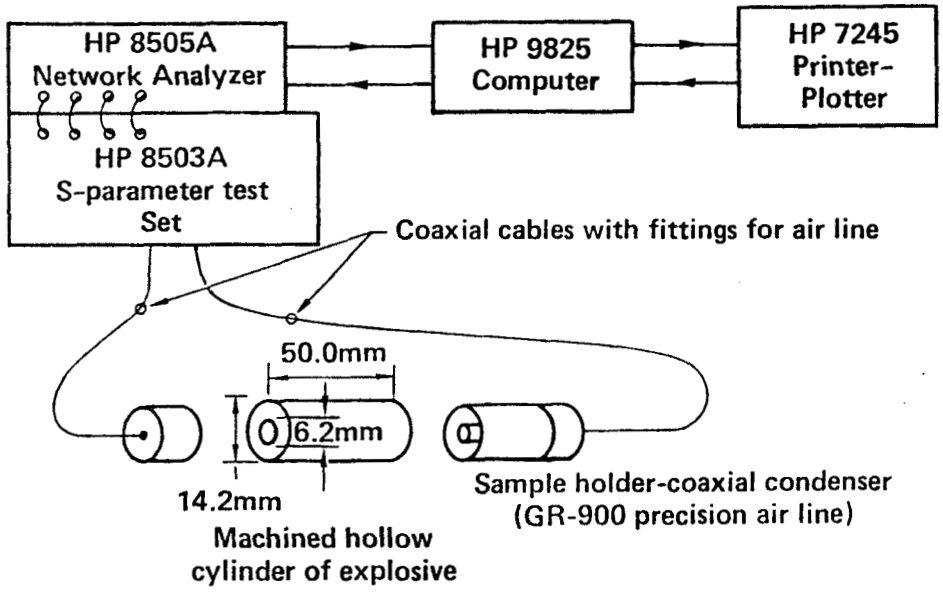

Figure 3. Experimental apparatus

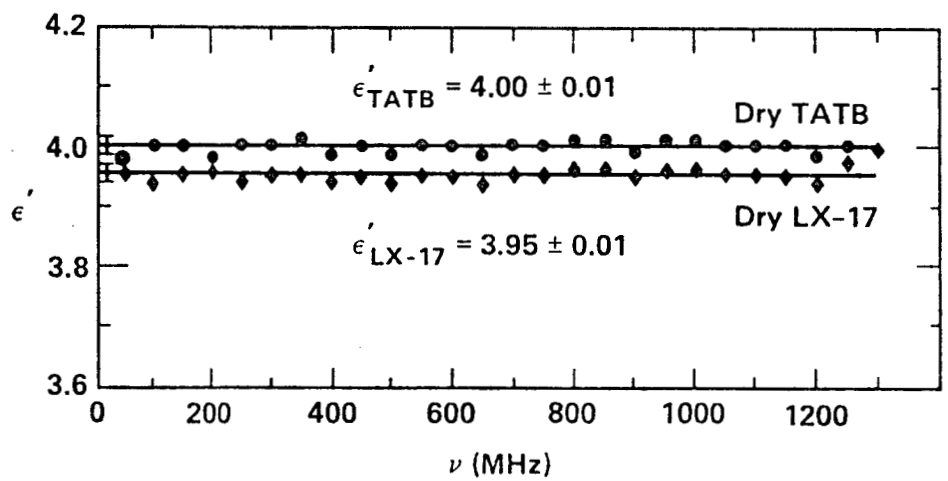

Figure 4. Plot of dielectric constant versus frequency for dry TATB and dry LX-17.

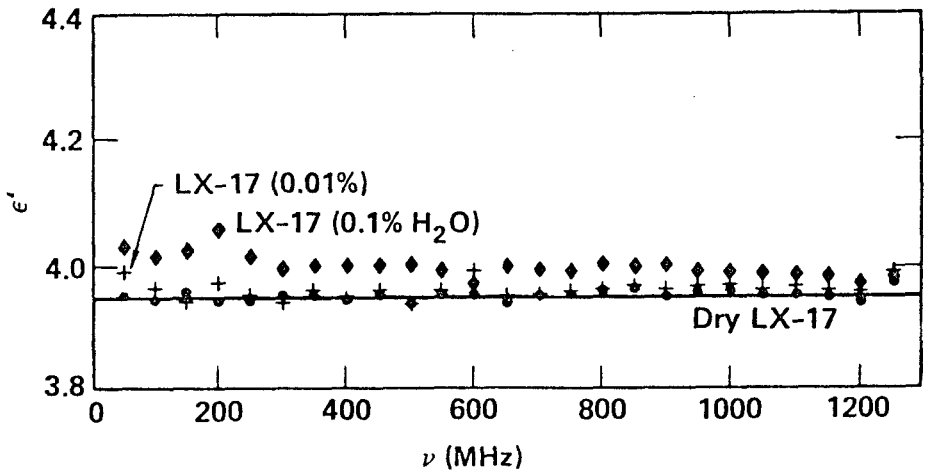

Figure 5. Plots of ϵ' versus frequency for 0.0%, 0.01%, and 0.1% H_2O in LX-17.

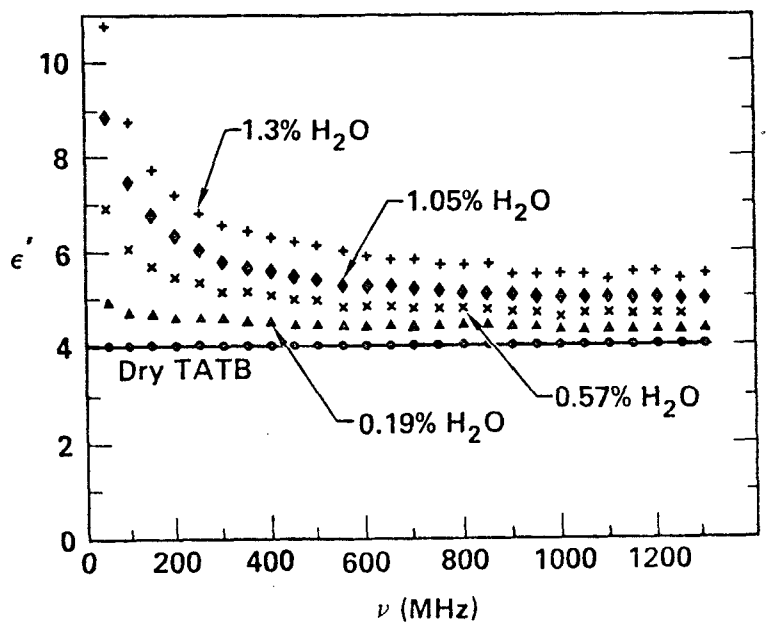

Figure 6. Plots of ϵ_{TATB} vs. frequency at higher % H_2O showing low frequency effect

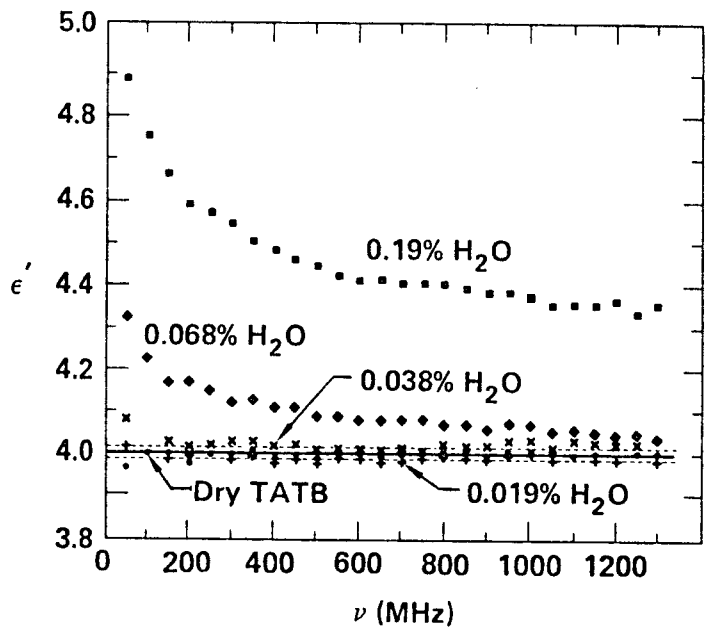

Figure 7. Plot showing detection limit of 0.04% H_2O in TATB

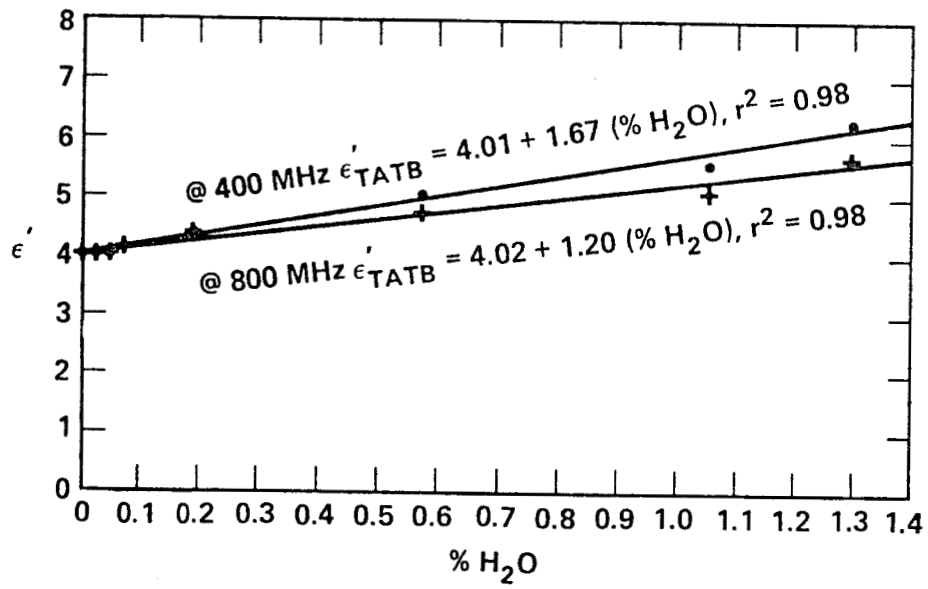

Figure 8. Linear regression plots of dielectric constant vs. % H_2O in TATB at 400 MHz and 800 MHz