# Recent Developments in the Capacitance Calibration Services at the National Institute of Standards and Technology

#### Presenter

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### **ABSTRACT**

Since 1994 a bank of four 10 pF oil-bath type fused-silica dielectric capacitors has been maintained in the NIST Impedance Calibration Laboratory for use as reference standards to assign values to the secondary reference and check standards used for performing calibrations on customer's standards. The values of the reference standards in this bank are determined via comparisons with a 10 pF air-bath type fused-silica capacitor from the NIST primary capacitance laboratory. This laboratory maintains the U. S. capacitance unit in a primary bank of 10 pF oil-bath type, fused-silica dielectric capacitors, traceable to the NIST calculable capacitor. The database for these reference standards is now established and the algorithms for maintaining their values with statistical prediction methods are developed. Also discussed is a preliminary investigation of the frequency dependence of the transfer standard, which significantly affects the present capacitance calibration uncertainties.

### INTRODUCTION

The major capacitance calibration system in the NIST Impedance Calibration Laboratory (ICL) is a transformer-ratio bridge, known as the "Type-2" capacitance bridge (1), which uses both external reference standards and internal capacitors to perform measurements of unknown capacitors. The reference standard normally is a capacitor whose value is well established, and the internal capacitors

<sup>\*</sup> Technology Administration, Department of Commerce.

are a set of eight air capacitors with values of 100 pF, 10 pF,....., and 10<sup>-5</sup> pF, which are used to balance the bridge during measurements at 1:1, 1:10, and 10:1 ratios. The Type-2 bridge is utilized to measure capacitors with nominal values in the range between 0.001 pF and 10 000 pF, at frequencies of 100 Hz, 400 Hz, and 1 kHz. A detailed description of the capacitance calibration facility at NIST, which includes the Type-2 bridge as well as other measuring systems and standards being used, is given in the NIST Special Publication 250-47 (2).

### REFERENCE STANDARDS

The reference standards of the ICL are a group of NIST-fabricated, 10 pF oil-bath type, fused-silica dielectric capacitors <sup>(3)</sup> that are maintained in an oil-bath at a temperature of 25°C. The values of these reference standards are determined via comparisons made with another NIST-fabricated, 10 pF air-bath type, fused-silica dielectric capacitor, used as a transfer standard. This transfer standard is obtained from the NIST primary capacitance laboratory, which maintains the U. S. representation of the farad, realized by the NIST calculable capacitor. Since each of these reference standards, including the transfer standard, has a large temperature coefficient, the capacitance measurement for each of them must be corrected to a reference temperature to eliminate the effect of temperature on the capacitance value. Therefore, besides the capacitance value, the temperature of an individual capacitor during the measurement is also obtained in order to correct the temperature to that at its reference temperature. The temperature is measured by a resistive temperature sensor which is mounted around the capacitor's fused-silica element. The ICL reference standards are then used to assign values to the secondary and check standards that are used for performing calibrations on customer's standards.

### ALGORITHM FOR MAINTAINING THE REFERENCE

There are a total of five fused-silica capacitors (designated as #121, #181, #182, #183, and #184) used in the oil bath, whose values are determined by the transfer standard. Figure 1 is an example to illustrate the data history, shown in parts per million (ppm) from the nominal value, including the control limit interval at the 95% confidence level, of a typical capacitor (#121) at its reference temperature. According to the analysis of the data histories of all capacitors over the past four years, except for one (#182), the capacitors have drift rates of less than 0.02 ppm/year and standard deviations of less than 0.03 ppm. The average value of these four capacitors has a drift rate of less than 0.005 ppm/year and a standard deviation of less than 0.01 ppm, as shown in Fig. 2. Therefore, the database for the average value of the four capacitors is maintained in the ICL and used as the reference for the calibrations of customer's standards.

Recently, an algorithm has been developed to maintain the database for each of the four capacitance standards by using the prediction of the average value of the four capacitors and the measurements performed with the fifth reference bank capacitor (#182). Normally, the value of each capacitance standard is determined by the transfer standard during the transfer of the farad; so is the average value of the four capacitors. During the period between the farad transfers, the value of each of these four capacitors is obtained by using the predicted average value and the measurements made by

performing left and right direct comparisons (i.e. to perform two measurements at the 1:1 ratio by interchanging the positions of the reference and the unknown) with the fifth capacitor (#182). Capacitor #182 is utilized as a dummy capacitor in the direct comparison. Its absolute value can be unknown, but must be stable during direct comparison which takes between 10 to 15 minutes to complete. From the analysis of the data, the stability (drift rate) of capacitor #182 was found to be within 0.2 ppm/year, with a standard deviation of less than 0.02 ppm, and therefore, it can be used to perform direct comparisons to obtain the values of the other capacitors in the oil bath. Since the measurement of direct comparison is easy to perform, the value of each capacitor can be obtained monthly to ensure that its stability is in statistical control. However, the average value of the four capacitors is updated only after the transfer of the farad is completed, which occurs every three to six months. Figure 3 shows the additional data obtained for capacitor #121 from direct comparisons with capacitor #182 and the predicted average value of the four capacitors, together with the data obtained using the transfer standard from the primary capacitance laboratory.

### **CALIBRATION UNCERTAINTIES**

The process of performing capacitance calibrations at NIST involves assigning values to the secondary reference standards, then to the check standards, and then to the customer's standards, as shown in Fig. 4. A detailed description of the calibration procedures is included in the Reference  $^{(2)}$ . The calibration uncertainties for fused-silica- and nitrogen-dielectric capacitors have been analyzed by Chang  $^{(4)}$  and summarized in Table 1. The term calibration uncertainty as used in this paper refers to the relative expanded uncertainty, (expressed in ppm), which is the combined uncertainty of Type A and Type B standard uncertainties using a coverage factor of k = 2  $^{(5)}$ .

According to the Reference <sup>(4)</sup>, the Type B standard uncertainty during the farad transfer contains several components, including the frequency dependence, voltage dependence, and uncertainty of the transfer standard, and errors in dial corrections and transformer ratios of the measurement system. The major source of error that contributes to the Type B standard uncertainty is the frequency dependence of the transfer standard, due to the fact that the primary capacitance laboratory maintains its reference standards at a frequency of 1592 Hz while the calibration requirement in the ICL is mostly at 1 kHz. Therefore, the frequency dependence of the transfer standard, especially between 1592 Hz and 1 kHz, is an important element for the calibration uncertainties assigned to the customer's standards.

# FREQUENCY DEPENDENCE OF TRANSFER STANDARD

The frequency dependence of a group of ten NIST-made 10 pF fused-silica capacitors was initially investigated by Cutkosky and Lee, comparing them with two 10 pF air capacitors at frequencies of 159 Hz, 1592 Hz, and 15900 Hz <sup>(3)</sup>. Among these ten capacitors, two were the air-bath type and eight were the oil-bath type. The frequency dependence of the two air-bath type capacitors, #112 and #113, between 1592 Hz and 159 Hz, was found to be +0.2 ppm and +1.9 ppm, respectively. The overall average of the frequency dependence of these ten capacitors between these two frequencies was found to be +1.12 ppm. For the past ten years, a third air-bath type 10 pF fused-silica capacitor

(#125) has been used as the transfer standard, which is also NIST-made and of similar design. Due to the lack of hard information concerning the frequency dependence of this transfer standard, a conservative value, +1.10 ppm, had been chosen to use as the frequency dependence uncertainty in going between 1592 Hz and 1 kHz for capacitor #125, since it was approximately the average of the two air-type capacitors (#112 and #113) and the overall average of the ten capacitors, between the larger range of 1592 Hz and 159 Hz.

In October, 1997, another measurement was performed in the primary capacitance laboratory to observe the frequency dependence at 1592 Hz, 1 kHz, 400 Hz, and 100 Hz on the three air-bath type capacitors, #112, #113, and #125, using an oil-bath type 10 pF fused-silica capacitor (#114) as the reference. According to the previous results, capacitor #114 has a frequency dependence of +0.1 ppm between 1592 Hz and 159 Hz; therefore, it could be used as a reference with the assumption of negligible frequency dependence, especially between 1592 Hz and 1 kHz.

Results from the measurements show that capacitor #112 has a relatively flat frequency response from 400 Hz to 1592 Hz, (measurements at 100 Hz were made but found unreliable), which is consistent with the previous investigation. The frequency dependence of capacitor #125 was found to be less than +0.1 ppm, between 1592 Hz and 1 kHz, which implied that the previously assigned frequency dependence (+1.1 ppm) might be a much larger value than necessary. The frequency dependence of capacitor #113 was found to be approximately +0.32 ppm, between 1592 Hz and 1 kHz, which indicated that this capacitor has the largest frequency dependence as compared with the other air-bath type fused-silica capacitors being used.

An attempt has been made to observe the frequency dependence of capacitor #113 from 1592 Hz to 1 kHz, to 400 Hz, to 159 Hz, and to 100 Hz, by combining the data from both measurements. Figure 5a is a plot to show the difference in capacitance values of capacitor #113 as a function of frequency, and the logarithmic least square regression line of the data using the equation:

$$y = c \ln x + b$$
, the set of each transfer and that the set of the

where y is the difference in capacitance values from the value at 1592 Hz (in ppm), x is the frequency of measurement in Hz, c and b are constants, and ln is the natural logarithm function.

Figure 5b is a similar plot to that of Fig. 5a by using the logarithm scale for the frequency, i.e. y vs.  $\ln(x)$ ; thus, the regression line becomes linear. The constants c and b in eq. (1), together with the standard error of the prediction,  $1\sigma$ , are determined and shown in both Fig. 5a and Fig. 5b. The correlation coefficient is found to be R = -0.98, which indicates that the frequency dependence of this capacitor is quite linearly correlated to the logarithm function of the frequency.

After the completion of the data analysis, the error components due to the frequency dependence of capacitor #113 are estimated to be +0.7 ppm at 1 kHz, +1.6 ppm at 400 Hz, and +3.2 ppm at 100 Hz. When capacitor #125 is being used as the transfer standard, its error components due to frequency dependence are much less than that of using capacitor #113 as the transfer standard, as shown in Fig. 6. Therefore, the present calibration uncertainties for the fused-silica and nitrogen dielectric

capacitors, as shown in Table 1, use the error components due to the frequency dependence of capacitor #113; these will be reevaluated after additional data are available, the adjustments for corrections are applied, and an absolute determination of the frequency dependence is done by relating the capacitors back to the calculable capacitor at each frequency.

### CONCLUSION

Some of the recent developments in the ICL at NIST are described including the establishment of oil-bath capacitors as the reference standards, the procedures for the transfer of the farad, and the methodology to maintain the reference standards. Also included is the analysis of the preliminary data from the measurements of the frequency dependence of the transfer standard, which represents a more accurate estimation in the calibration uncertainties of the fused-silica and nitrogen dielectric capacitors. Further investigations in the areas of frequency dependence of the transfer standards are needed such as to perform additional measurements, to apply corrections to it, and to relate it back to the calculable capacitor at each frequency.

### **ACKNOWLEDGMENTS**

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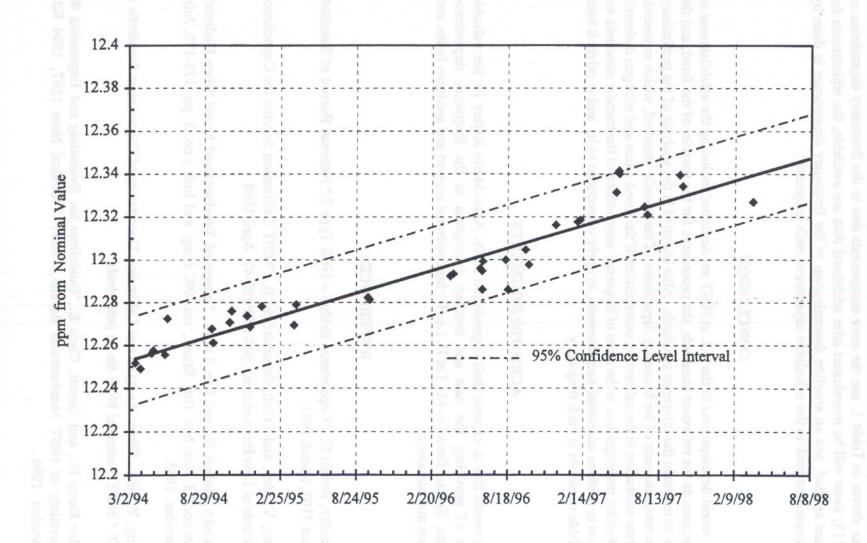


Figure 1. Reference Values of NIST Capacitance Standard #121 (10 pF Oil-Bath Type Fused-Silica Dielectric Capacitor).

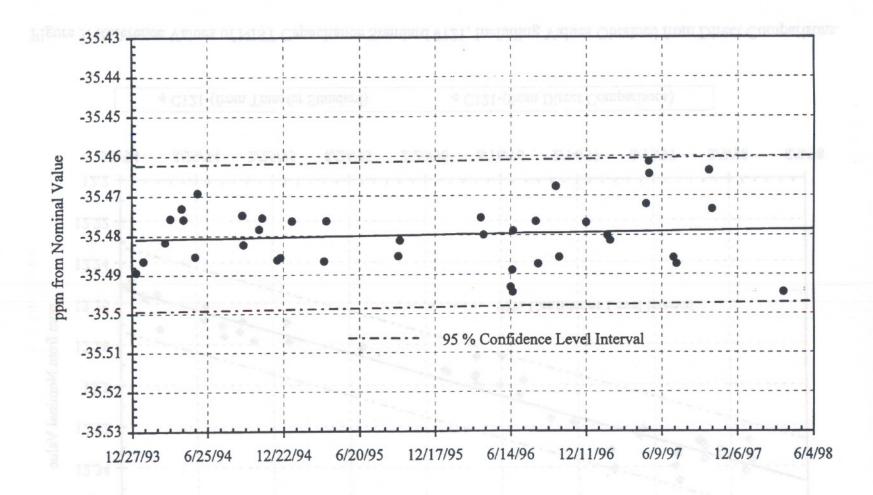


Figure 2. Average Values of Four NIST Capacitance Standards (10 pF Oil-Bath Type Fused-Silica Capacitors).

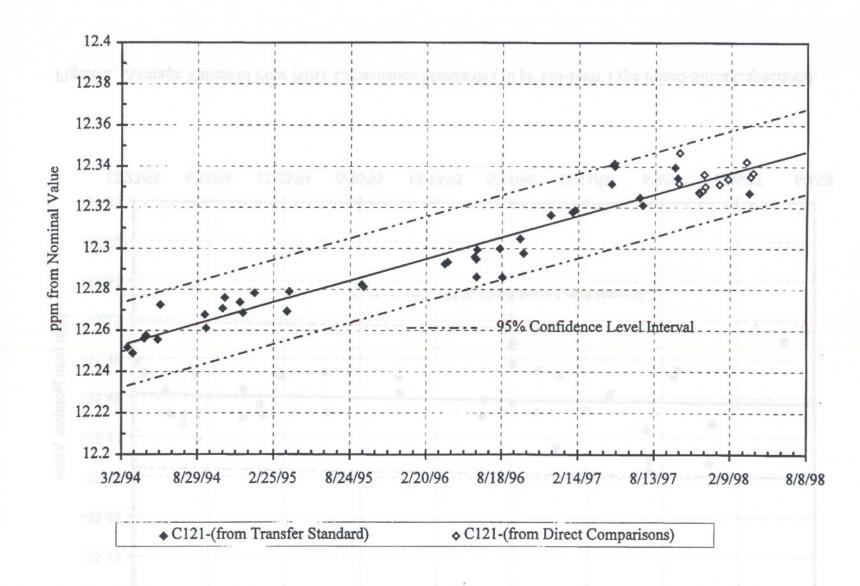


Figure 3. Reference Values of NIST Capacitance Standard #121, Including Values Obtained from Direct Comparisons.

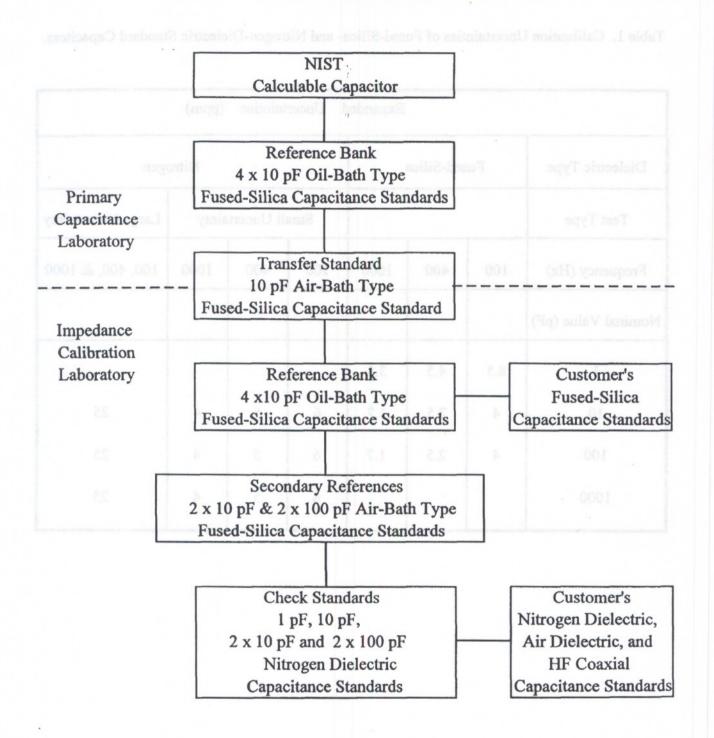


Figure 4. Block Diagram of the Farad Transfer and Calibration Process in the NIST Impedance Calibration Laboratory.

Table 1. Calibration Uncertainties of Fused-Silica- and Nitrogen-Dielectric Standard Capacitors.

| Expanded Uncertainties (ppm) |              |       |                     |                   |           |      |                           |
|------------------------------|--------------|-------|---------------------|-------------------|-----------|------|---------------------------|
| Dielectric Type              | Fused-Silica |       |                     | Nitrogen          |           |      |                           |
| Test Type                    |              |       |                     | Small Uncertainty |           |      | Large Uncertainty         |
| Frequency (Hz)               | 100          | 400   | 1000                | 100               | 400       | 1000 | 100, 400, & 1000          |
| Nominal Value (pF)           |              | bash  | nce Star            | Capacia           | eoiliR-be | Fus  | Impedance                 |
| Custdmer's                   | 8.5          | 4.5   | 3.8                 | lerence l         | Re        |      | Calibration<br>Laboratory |
| 10                           | 4            | 2.5   | 1.7                 | 6                 | 5         | 4    | 25                        |
| 100                          | 4            | 2.5   | 1.7                 | 6                 | 5         | 4    | 25                        |
| 1000                         |              | sqvTr | forences<br>Air-Bal | 6                 | 5         | 4    | 25                        |

4.5

3.5

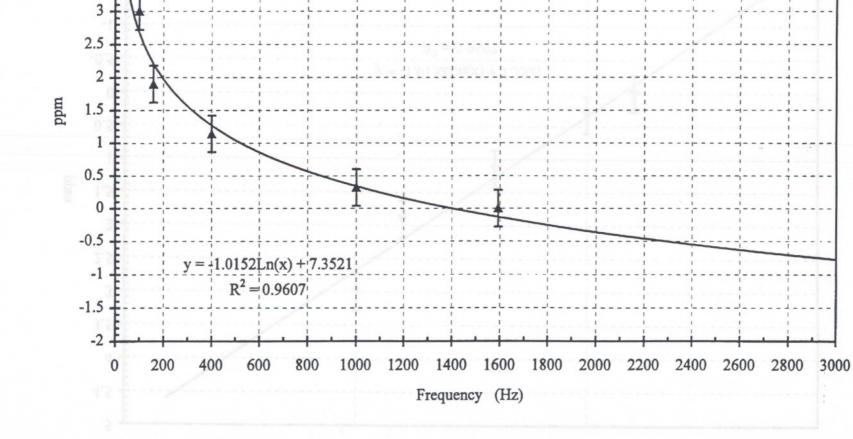


Figure 5a. Difference in Capacitance Values of Capacitor #113, from its Value at 1592 Hz, with the Logarithmic Regression Fit.

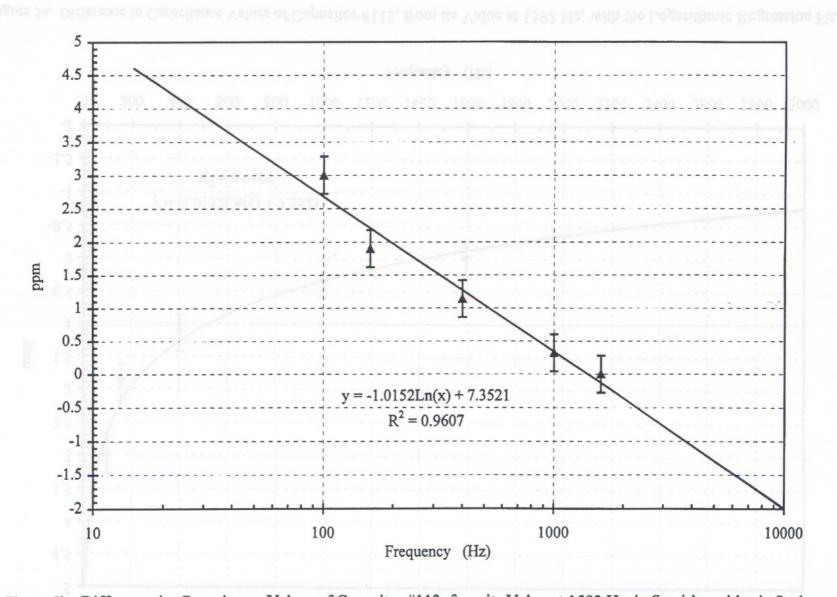


Figure 5b. Difference in Capacitance Values of Capacitor #113, from its Value at 1592 Hz, in Semi-logarithmic Scales.

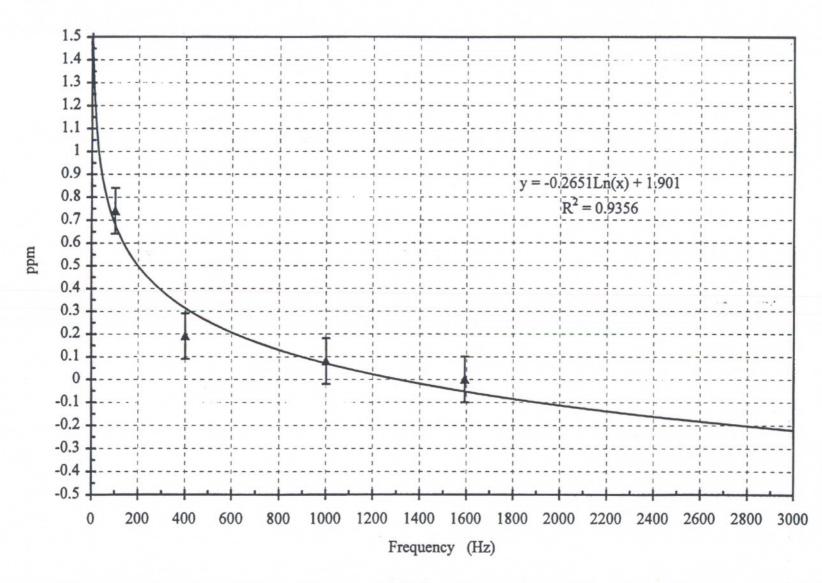
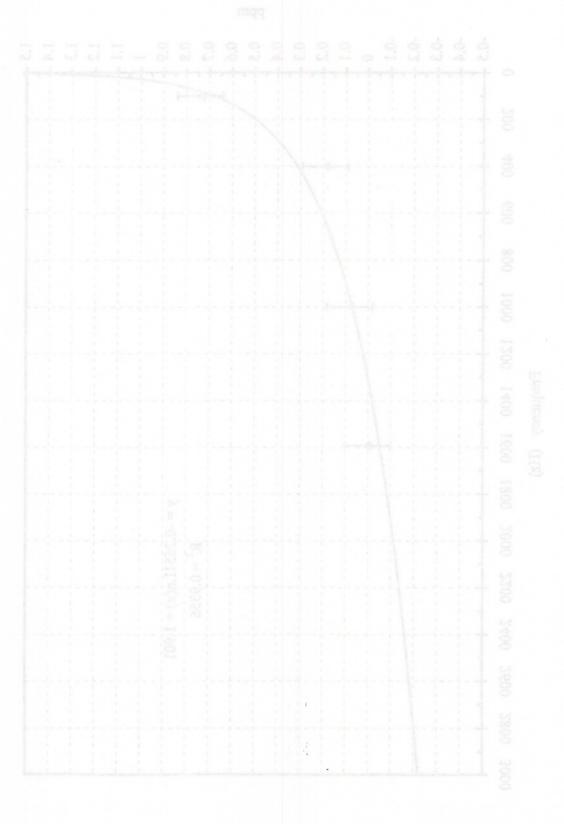


Figure 6. Difference in Capacitance Values of Capacitor #125, from its Value at 1592 Hz, with Logarithmic Regression Fit.



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