Measurements of frequency dependence of fused-silica capacitors

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Abstract

In order to improve the capacitance calibration services at the National Institute of Standards and Technology, we need to determine the frequency dependence of all the reference capacitance standards over the audio frequency range. The value of a standard capacitor may vary slightly with frequency because the imperfect medium between its electrodes has varying degrees of dielectric relaxation over the frequency range and the leads and electrodes of the capacitor have residual inductance. Using a combination of a 1 pF cross capacitor that has negligible frequency dependence due to electrode surface films, and a 10 pF nitrogen dielectric capacitor with a very small residual inductance as references, we have measured the frequency dependence of two 10 pF transportable fused-silica capacitors from 50 Hz to 20 kHz. The results and the associated uncertainty analysis are discussed.

<u>Keywords</u> – calibration, capacitance, farad, frequency dependence, fused-silica capacitor, impedance, measurement, uncertainty

1. Introduction

We have recently put a significant effort into determining the frequency dependence of capacitance standards in the audio frequency range. This effort at the National Institute of Standards and Technology (NIST) is in response to industrial needs. Recently, ultra-precision multi-frequency (from 50 Hz to 20 kHz) capacitance bridges have become commercially available and secondary calibration laboratories have started using this new type of bridge for their impedance calibrations; thus, NIST needs to improve calibration services for characterizing the frequency dependence of commercial fused-silica capacitance standards over the entire audio frequency range. The effort on frequency dependence is also aimed at addressing some concerns within the metrology community regarding two possible quantum representations of the farad. For the electron counting capacitance standard [1] which functions near DC, we need to characterize the frequency dependence of a cryogenic capacitor in order to use it for AC calibrations. For the farad representation via AC Quantum Hall Resistance (QHR), a comparison between a frequency-characterized capacitor and an AC QHR through a multi-frequency quad bridge may help to resolve the origin of the linear frequency dependence observed in QHR devices [2].

The primary maintenance standard for NIST capacitance calibrations consists of a bank of four 10 pF fused-silica standards (referred to as the Farad Bank) which are maintained in an oil bath at

2003 NCSL International Workshop and Symposium

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1

25 °C. The Farad Bank is very stable, drifting only fractionally 0.02×10^{-6} per year, and the standards are calibrated twice a year indirectly against the calculable capacitor [3] at NIST at a frequency of ≈ 1592 Hz ($\omega = 10^4$ rad/s), using a 10 pF transportable fused-silica capacitor, C_{112} . This frequency was chosen for a convenient link between the farad and the ohm.

The NIST calculable capacitor is based on the Thompson-Lampard theorem [4]. It is comprised of four parallel, uniform electrodes with very small gaps between them as shown in Fig. 1(a). The capacitances per unit length, C_a and C_b , between the opposing pairs of electrodes obey the relationship:

 $\exp(-\pi C_a / \varepsilon_o) + \exp(-\pi C_b / \varepsilon_o) = 1,$

where ε_o is the permittivity of vacuum. When $C_a = C_b = C$, this reduces to

$$C = \frac{\varepsilon_o \ln 2}{\pi},$$

which is a constant that directly links to the defined speed of light. The calculable capacitor provides an absolute determination of capacitance in terms of length only and is the ultimate reference for all impedance measurements in the U.S. However, the calculable capacitor system is optimized for operation at 1592 Hz; modifications and corrections required to operate at other frequencies are non-trivial.

Today, capacitance calibrations in the audio frequency range are available from NIST only at frequencies of 1000 Hz, 400 Hz, and 100 Hz [5]. In the 1960s, the frequency dependence of the Farad Bank between 1592 Hz and these lower frequencies was determined by comparison to a 10 pF guarded cylindrical capacitor filled with nitrogen, C_{10} . The frequency dependence of this



Fig. 1. (a) 4-rod cross capacitor. Dashed line represents a ground shield. (b) 9-rod cross capacitor.

2003 NCSL International Workshop and Symposium

nitrogen capacitor was estimated to be small; however, it had not been experimentally determined. Therefore, large relative uncertainties were assigned to the frequency dependent capacitance calibrations: 1.5×10^{-6} , 2.5×10^{-6} , and 4.0×10^{-6} (k=2) for 1000 Hz, 400 Hz, and 100 Hz, respectively.

Recently, Jeffery and Koffman [5] have re-characterized the frequency dependence of the Farad Bank from 1592 Hz to 1000 Hz using the calculable capacitor system. Thus, NIST was able to reduce the overall expanded uncertainty for 1000 Hz calibrations of 10 pF and 100 pF fused-silica capacitors from 1.5×10^{-6} to 0.5×10^{-6} . However, this approach is very tedious; calibration of the bridge transformer used for the calculable capacitor alone involves multiple steps at each frequency. The method cannot be simply copied to reduce the uncertainties at very low frequencies such as 100 Hz. The reported Type-A uncertainty of 0.05×10^{-6} at 1000 Hz is expected to increase by a factor of 100 at 100 Hz. The impedance of a capacitor increases with decreasing frequency and the operating voltage has to be reduced linearly with frequency below 1000 Hz to avoid saturating the ac bridge for the calculable capacitor system. These two effects lead to severe reduction in signal with no corresponding reduction in noise.

2. Mechanisms of Frequency Dependence

There are two dominant sources of capacitance change with frequency. Residual inductance tends to increase the apparent capacitance of a simple capacitor. Consider a three-terminal capacitor with the residual series inductance equal to L, the capacitance between the two active electrodes C_o , and the stray capacitance from leads and electrodes to the ground terminal C_g . A simplified equivalent circuit for the capacitor consists of L in series with $C_g + C_o$. The fractional increase in capacitance is approximately $\Delta C/C_o \approx \omega^2 L(C_g + C_o)$. This increase can be significant when the frequency is high, but as it will become clear later, it is negligible below 1592 Hz compared to other possible frequency dependences.

The other dominant source of frequency dependence results from dielectric relaxation, which tends to increase the capacitance at low frequencies. This can be understood by considering a set of non-interacting dipoles in an ac field. At low enough frequencies, the orientation of the dipoles can keep up with the instantaneous value of the electric field, therefore the resultant dielectric constant is at maximum. As the frequency increases, the dipoles start to lag behind the field, decreasing the effective dielectric constant.

Dielectric relaxation manifested in the audio frequency range may not be solely caused by bulk properties of the dielectric in a capacitor. Cutkosky and Lee [6] have shown that the relative frequency dependence of a set of 10 pF fused-silica capacitors, which were fabricated identically, changes greatly from one capacitor to another, suggesting that some defects may be responsible for the observed frequency dependence. When nitrogen gas fills the vacuum gap in a capacitor, the dielectric constant and therefore the capacitance increase; however, the increase is frequency-independent in the audio frequency range because the frequency associated with the dielectric relaxation time of the gas molecules is in the microwave region. Dielectric films on electrode surfaces are the main cause of the frequency dependence of gas dielectric and vacuum gap capacitors. Inglis [7] performed a thorough study of electrode surface film effects on the frequency dependence of parallel-plate capacitors. He modeled a parallel-plate capacitor as an ideal capacitor C_{o} , representing the vacuum gap, in series with a lossy capacitor C_s , lumping capacitances associated with the electrode surface films. The apparent capacitance is then $C \approx C_o (1 - C_o / C_s)$, and the component that causes frequency dependence is quadratically proportional to C_o : $\Delta C = -C_o^2 / C_s$. By putting two variable parallel-plate capacitors in parallel and varying the separations in such a way that the total capacitance was kept constant at a chosen frequency, he found that the frequency dependence from 50 Hz to 50 kHz due to the surface films on plain brass electrodes or Rh plated electrodes approximately follows the empirical function: $\Delta C / C = b \log \omega + c$, where b and c are constants.

3. Reference Standards

This work was started with a 9-rod, 1 pF nitrogen cross capacitor, C_1 , as shown in Fig. 1(b). This type of 9-rod cross capacitor can be considered as four conventional 4-rod cross capacitors in parallel. It was designed at NIST to increase the capacitance while keeping the capacitor within a manageable length. When rod 5 is powered, rods 1, 3, 7, and 9 are connected to the detector while all other rods are grounded. Conversely, when rods 2 and 8 are powered together, rods 4 and 6 are connected to the detector while all other rods are grounded. All detector rods have guard ends to reduce the fringe effects. This cross capacitor was first chosen for frequency-dependence study because it has been shown by Lampard and Cutkosky [8] that thin dielectric films uniformly deposited on the electrodes of a cross capacitor do not alter its individual cross capacitances to the first order. However, it was soon realized that the required lead corrections and the associated uncertainties at high frequencies could be significant, because extra wires were required to connect the capacitors in parallel. To determine the lead corrections, a comparison was made between C_{I} and C_{10} which has a very small residual inductance, using the 4-terminal pair bridge at NIST [10]. At each frequency, the transformer ratio of this bridge was evaluated using our usual method of the permutation of eleven 10 pF capacitors [9]. The measured ratio, C_{10}/C_1 , expressed as relative change from the ratio at 400 Hz is shown in Fig. 2, as a function of frequency from 400 Hz to 20 kHz. Also shown in the figure is a least-squares fit of the formula $a\omega^2 + b\log\omega + c$ to the experimental results, where the first term accounts for the inductance of the 1 pF cross capacitor, while the second term represents the surface film effects of the 10 pF nitrogen-filled cylindrical capacitor. The results indicate that the cross capacitor has small lead corrections at low frequencies, about 0.03×10⁻⁶ at 1592 Hz, and thus it can serve as a good reference for frequency dependence in the low-frequency region. The results also show that the frequency dependence due to the electrode surface films of the 10 pF nitrogen capacitor is about 0.2×10⁻⁶ per decade change in frequency, which is much smaller than the uncertainties previously assigned to this source.

4. Results and Uncertainty Analysis

As a first step to improve the capacitance calibrations at NIST, we decided to extend the frequency range previously explored for the fused-silica reference capacitors and reevaluate the uncertainties. Shown in Fig. 3 is the measured frequency dependence of the fused-silica transfer standard C_{112} , expressed as the fractional change from the capacitance at 1592 Hz, using C_{10} as the reference standard. The measurements were performed using the 10:1 4-terminal pair bridge from 400 Hz to 20 kHz and a conventional 10:1 2-terminal pair bridge [11] below 400 Hz; the conductance injection scheme for the latter has been described by Jeffery and Koffman [5]. For the entire frequency range from 50 Hz to 20 kHz, an auxiliary temperature-stabilized 100 pF fused-silica





capacitor, C_{272} , was used on the bridge low potential arm, and a simple substitution method for the two 10 pF capacitors on the high potential arm was employed to minimize the sources of uncertainties.

The main sources of uncertainties for the measurements shown in Fig. 3 are listed on Table 1 for five representative frequencies. All measurements are relative to C_{112} at 1592 Hz, where it is linked to the calculable capacitor with a relative standard uncertainty of 0.019×10^{-6} . At other frequencies, three sources of uncertainties dominate. The Type-A uncertainty, which is directly linked to the signal-to-noise ratio of the ac bridge systems, is the largest at 50 Hz, about 0.5×10^{-6} . It decreases quadratically with increasing frequency and reaches a minimum at a few kHz; it then increases slightly at the highest audio frequencies owing to a lower output voltage from the source and a bandwidth roll-off of the pre-amplifier. The inductances of the leads and electrodes of C_{10} were estimated using a resonance method similar to the one described by Free and Jones [12]. The uncertainty due to the residual inductances and stray capacitances is the largest at 20 kHz, about 0.27×10^{-6} ; this uncertainty decreases quadratically with decreasing frequency. The fitting results in Fig. 2 indicate that the frequency dependence due to electrode surface films of C_{10} follows the equation $x \equiv b \log \omega + c$. Here 2x is taken as the upper bound to estimate the uncertainty due to the surface film effects, leading to a standard deviation of 0.23×10^{-6} in the capacitance per decade

2003 NCSL International Workshop and Symposium

5



Fig. 3. Frequency dependence of C_{112} expressed as fractional change from the capacitance at 1592 Hz, with 1 σ uncertainty bars.

Table 1.	Contribution of	component uncertainties to the tota	l uncertainties at six re	presentative f	frequencies f	for C	2112
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	Relative standard uncertainty (×10 ⁻⁶)							
Source of uncertainty	50 Hz	100 Hz	400 Hz	1000 Hz	4 kHz	20 kHz		
Туре-А	0.5	0.13	0.01	0.001	0.001	0.01		
Lead inductance effects of C_{10}	0	0	0	0.002	0.012	0.27		
Surface film effects of C_{10}	0.34	0.28	0.14	0.05	0.09	0.25		
Stability of C_{10}	0.03	0.03	0.03	0.03	0.03	0.03		
Stability of C_{112}	0.002	0.002	0.002	0.002	0.002	0.002		
Stability of C_{272}	0.01	0.01	0.01	0.01	0.01	0.01		
Bridge linearity errors	0.05	0.05	0.005	0.005	0.005	0.005		
Relative combined standard uncertainty	0.61	0.32	0.15	0.06	0.1	0.37		

2003 NCSL International Workshop and Symposium

6

change in frequency from 1592 Hz. All other sources of uncertainties, including the stabilities of the capacitors during the experiments and the bridge linearity errors, make very small contributions to the final relative combined standard uncertainties which are shown in Fig. 3, together with the frequency dependence data.

The new measurements of the frequency dependence of C_{112} are consistent with all the previous frequency dependence data released from NIST. The present result of 0.05×10^{-6} at 1000 Hz relative to the capacitance at 1592 Hz agrees well with the result (0.04×10^{-6}) determined by Jeffery and Koffman [5], using the calculable capacitor chain directly. It is also interesting to note that the relative standard uncertainty (0.06×10^{-6}) at 1000 Hz determined using the present method agrees with the uncertainty (0.055×10^{-6}) assigned earlier for the calculable capacitor measurement of C_{112} at 1000 Hz; however, this agreement may be fortuitous because the dominant source of uncertainties is the electrode surface films of C_{10} for the former method while it is the Type-A uncertainty for the latter. In addition, the present data overlap, within the experimental uncertainties, with the frequency dependence data obtained earlier for 159 Hz and 15900 Hz by Cutkosky and Lee [6].



Fig. 4. (a). NIST measurements of frequency dependence of a 10 pF Andeen-Hagerling capacitor expressed as fractional change from the capacitance at 1592 Hz, with associated 1 σ uncertainty bars. (b). Relative change of the same capacitor as a function of frequency measured by an Andeen-Hagerling model AH2700A bridge with uncertainties specified by the manufacturer.

It is straightforward to transfer the frequency dependence data of C_{112} to other 10 pF capacitance standards. Shown in Fig. 4 (a) are measurements of frequency dependence of a 10 pF fused-silica

2003 NCSL International Workshop and Symposium

capacitor (S/N 01234) made by Andeen-Hagerling [13]. Also shown in Fig.4 (b) for comparison are the apparent fractional changes of its capacitance as a function of frequency measured by an Andeen-Hagerling model AH2700A bridge.

5. Conclusion

We have measured the frequency dependence of two transportable fused-silica capacitance standards from 50 Hz to 20 kHz. The relative standard uncertainties determined at 400 Hz and 100 Hz $(0.15 \times 10^{-6} \text{ and } 0.32 \times 10^{-6}, \text{ respectively})$ are smaller than the uncertainties previously assigned to frequency dependence at these frequencies by a factor of five. This will lead to improvements in the capacitance calibration services at NIST by a factor three or four. Next, we plan to transfer the frequency dependence data of C_{112} to the Farad Bank and other reference capacitance standards, so that improved capacitance calibrations will be available for the entire audio frequency range from NIST in the near future.

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