

Fig. 10. Calibration curve of comparator.

principally due to the nonzero effective input noise temperature of the comparator.

Calibration Curve

A calibration curve for the comparator is shown in Fig. 10. On the semilog plot, the curve is nearly linear above 500°K and becomes curved where the internal noise becomes influential. From this curve, the net effective internal noise temperature T_i of the comparator appears to be approximately 40°K.

CONCLUSION

The prototype 3-MHz noise-power comparator has been shown to have operational characteristics as predicted by theory and can intercompare noise generators having effective noise temperatures from below 75°K to greater than 30,000°K. The accuracy of comparison is better than 1 per cent at 75°K and improves to better than 0.2 per cent above 500°K. These results indicate that a high-quality comparator can be built for use in a calibration service for noise power.

ACKNOWLEDGMENT

The authors gratefully acknowledge the contributions to this work by L. D. Driver, R. L. Martin, E. E. Baldwin, W. H. Long, and J. J. Norton.

International Comparison of Dielectric Measurements

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Abstract—Three materials, i.e., fused silica, glass, and alumina, were selected for comparison based on known or expected homogeneity, isotropy, and stability. Measurements were made by the three Government laboratories (U. K., U. S. A., and Canada), both in the radio-frequency range, using capacitor-type holders either with or without an air gap, and at microwave frequencies, using either cavity-resonance methods or transmission-line impedance methods. The range among the laboratories on the real part of the permittivity is 0.4, 0.8, and 0.2 per cent for the three materials, respectively. The agreement on loss tangent is of the order of 0.0001 in many cases, but larger discrepancies exist. An introductory statistical analysis for systematic differences between laboratories is given.

Manuscript received August 3, 1964.

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I. INTRODUCTION

THE PRESENT ENDEAVOR is a cooperative effort by three national standards laboratories to check or improve their measuring systems for complex permittivity. The original proposal for such a comparison came to the laboratories through URSI channels.

An important difficulty with such an intercomparison is the fact that different laboratories, in general, require differently shaped specimens. For valid results, these samples should 1) be obtained from a stock of material that is homogeneous, 2) be stable in space and time, and 3) be preferably isotropic. An example of a material that would probably satisfy these requirements to a high degree is optical glass; it was not used, however, because other materials of more interest to radio applications were expected to have sufficient homogeneity and isotropy.

The standardization of the permittivity of a material and, concomitantly, the comparison of the measuring accuracy at separate laboratories have the interesting feature that the standard for the work is supposedly identical for all laboratories, being ϵ_0 to the dielectric constant of the vacuum. The constitutive electrical parameters of materials are measured by replacing the vacuum, or the air, with a specimen of known dimensions somewhere in an electrical circuit and measuring appropriate changes in the circuit. The sample holder is usually a lumped circuit reactance or a section of transmission line, and the observation may be of resonance or of impedance.

II. MATERIAL UNIFORMITY

The materials used were an alumina, an electrical glass, and a fused silica assigned the numbers 995, 1723, and 7940, respectively. The checks on homogeneity and isotropy will be described.

The 995 alumina for the different laboratories¹ was formed into all the shapes required during one production run. The homogeneity was checked by comparing four specimens from the NBS pieces. Results for the real part of the permittivity were $k' = 9.407, 9.402, 9.413, 9.407$, and loss tangent $\times 10^4 = 1.94, 1.96, 1.92, 2.04$, respectively. These measurements were on 0.25-inch diameter rods in the H_{011} resonator, which will be referred to later. Also, previous experience with ten specimens of a similar alumina production had given $k' = 9.559$ and a standard deviation $s = 0.0027$, and loss tangent $\times 10^4 = 1.74$ and $s = 0.012$.

The 7940 samples were cut from a plate 1.1 inches thick. Homogeneity was deduced from four rod specimens cut from near the top and bottom of the thickness at each of two locations eight inches apart. The range of k' from top to bottom locations was 0.05 per cent, and the range for the locations separated by eight inches was 0.15 per cent. The range of $\tan \delta$ was 0.00002.

Previous measurements [1] of the 1723 material on samples from three corners of a glass sheet having dimensions of the order of 12 inches had revealed a range of 0.1 per cent in k' and of 0.00004 in the loss tangent of 0.0053.

The observed variations may not be due entirely to inhomogeneity. Measuring errors also contribute. It may, therefore, be assumed that a reasonable estimate of the range of inhomogeneity is 0.1 per cent of k' and 0.00004 or less in the loss tangent for the 1723 and 7940 materials. These limits probably apply to the 995, also, judging from the final results of the three laboratories. The results from the four previously mentioned specimens were in close agreement but are not considered definitive because the specimens were not scattered through all the different pieces.

¹ Ten laboratories in all were involved in the comparison; the seven not named here are comparing measurements at elevated temperatures.

The isotropy of the 995 and 1723 materials was checked by making specimens $0.4 \times 0.4 \times 0.9$ inch that could be turned by 90° in the usual rectangular waveguide at 9 GHz. Thus the electric field was either perpendicular or parallel to the axis of the original cylindrical 995 stock material and to the plane of the original glass plate. The isotropy of k' was within 0.05 per cent for the 995 material as checked by NBS, and within 0.1 per cent for the 1723 material, as checked by NRC. For the 7940 material, two cylindrical specimens for the TE_{11} line were cut with axes respectively perpendicular and parallel to the plane of the plate. Within the standard deviation of the instrument for this test, 0.14 per cent, there was no anisotropy.

III. MEASUREMENT METHODS

A. National Research Council Description

Measurements of the dielectric constant and loss tangent of two samples of each of the three materials were made at frequencies of 1, 10, and 100 kHz using a capacitance-conductance bridge [2] and a three-terminal sample holder. Equipment of the Hartshorn-Ward type [3] using a two-terminal sample holder was used for measurements at 100 kHz, 1 MHz and 10 MHz. In addition, some measurements were made at 100 MHz using this equipment. However, there is evidence of a systematic error at this frequency, and the results have not been included here.

All measurements at 1 kHz to 10 MHz were made using an air gap between the upper surface of the sample and the upper electrode of the sample holder. For each sample at least three measurements of the dielectric constant and loss tangent were made. In each measurement a different air gap was employed. The range of air gaps used was from 0.0015 cm to 0.025 cm.

There were slight discrepancies between the values of dielectric constant obtained by the two methods at 100 kHz. The largest discrepancy occurred for the glass samples, for which the difference in dielectric constant was 0.5 per cent. It was felt that the results obtained using the three-terminal sample holder and the bridge were more accurate than those obtained using the two-terminal sample holder. Therefore, the difference between the average of the dielectric constant results obtained by the two methods at 100 kHz was used to correct the 1-MHz and 10-MHz data.

The air-gap technique has the advantage that no secondary electrodes have to be applied to the samples. It has the disadvantage that the accuracy of determination of the dielectric constant is critically dependent on the accuracy of measurement of the sample thickness. The percentage error in dielectric constant k' can be shown to be $(k' - 1)$ times the percentage error in determination of the average sample thickness. For the very flat samples used in this intercomparison the maxi-

imum percentage error in determination of sample thickness was estimated to be ± 0.05 per cent. For each sample this gives rise to an uncertainty in the true value of the dielectric constant of up to ± 0.05 ($k' - 1$) per cent. This amounts to ± 0.14 per cent for the silica, ± 0.26 per cent for the glass, and ± 0.42 per cent for the alumina. In practice, the average values of dielectric constant obtained for two samples of the same material at the same frequency never differed by more than 0.3 per cent; therefore, the actual uncertainties in dielectric constant were probably smaller than the maximum values just given. Some estimate of the random error can be obtained from the agreement between measurements on the same sample with different air gaps. The average range was ± 0.05 per cent for glass and silica and ± 0.12 per cent for alumina; the maximum range was ± 0.2 per cent. The estimated limits of error in the average loss tangents given are ± 5 per cent for loss tangents greater than 1×10^{-4} . For smaller loss tangents the limits of error are considerably greater.

All the NRC measurements at microwave frequencies were made using the short-circuited waveguide method. At 8.5 and 8.6 GHz, a circular waveguide slotted section excited in the TE_{11} mode was used for the measurement of disk samples. A rectangular waveguide slotted section was used at 9.2 GHz to check the isotropy of a sample of 1723 glass. A rectangular slotted section was used at 23 GHz for measurements on fused silica and alumina samples. The results for the alumina at 23 GHz have not been included because the relatively large clearances between the samples and the waveguide walls gave rise to an uncertainty of several per cent in the dielectric constant, even after a correction for sample fit had been applied [1].

In the measurements at 8.5 GHz it was found that the shorter samples gave rather inconsistent results, and there was some evidence that more than one mode was propagating in the region of the dielectric sample. Changing the frequency to 8.6 GHz resulted in somewhat less evidence of multiple mode propagation, but the consistency of the results was still poor. With the longer samples more consistent results were obtained. Consequently, the average dielectric properties were calculated from the results obtained with the longer samples only. For these samples the reproducibility was such that the maximum variation in measured dielectric constant was ± 0.3 per cent, and the maximum variation in measured loss tangent was ± 3 per cent or $\pm 0.6 \times 10^{-4}$, whichever was larger.

In the case of the glass sample tested for isotropy at 9.2 GHz, reproducible results were obtained. However, the correction for sample fit was approximately 3 per cent, and there is some evidence [1] that when the sample fit correction is as large as this the corrected dielectric constant may still be in error by several tenths of a per cent. For the circular-disk samples the sample

fit corrections were about 0.4 per cent for the fused silica and glass and 1.1 per cent for the alumina.

Since all measurements were made in air, the measured dielectric constant values were corrected for the dielectric constant of air. This was taken as 1.00053.

Preparation of the Samples and Conditions of Measurement: The fused-silica and glass samples were cleaned by washing them with soap and water. They were then rinsed in distilled water and dried. The alumina samples as received were very clean and were not treated in any way before measurement. Samples were kept in the measuring laboratory for at least twelve hours before any measurements were made. This laboratory had temperature control but not humidity control. During the measurements the ambient temperature was $22.0 \pm 1.5^\circ\text{C}$. The relative humidity varied between the limits 28 per cent and 44 per cent.

B. National Physical Laboratory Description

Apparatus:

1) *Audio-frequency measurement:* The measurements at 1 kHz and 10 kHz were made on a Wagner earthed Schering bridge by the substitution method with the customary substitution for loss in the opposite arm of the network. A guard-ring system with mercury electrodes [4] was employed.

2) *Radio frequency measurements:* The measurements at 100 kHz and 1 MHz were again made by the substitution method on a Wagner earthed Schering bridge after Dye and Jones [5]. For capacitance (permittivity) measurement the mercury clamp with guard-ring electrode system was again employed, while for loss measurement the electrode system from a dielectric test set [3] was employed with silver electrodes evaporated onto the sample.

The measurements at 1 MHz, 10 MHz and 100 MHz were made on the Dielectric Test Set of Hartshorn and Ward [3].

3) *Microwave measurement:* The measurements at 9 GHz were made by placing the two-inch diameter disk sample in an H_{01} cavity resonator after Horner, et al. [6], with coupling to the cavity by the method of Bleaney and Penrose [7] and using a Pound [8] stabilized klystron. The output from the crystal detector was fed directly into a galvanometer; the square-law response was checked by making measurements at the $\theta/2$ and $4\theta/5$ points.

Sample preparation: After lapping, the samples were washed in carbon tetrachloride and petroleum spirit and heated to over 100°C for two to three days. They were then stored until measurement in a desiccator over silica gel.

Sample dimensions: The NPL apparatus has been designed to enable measurements to be made on the

TABLE I
MEASUREMENT RESULTS

Permittivity						
Frequency in Hz	k'			Loss Tangent $\times 10^4$		
	NRC	NPL	NBS	NRC	NPL	NBS
7940 Fused Silica						
10^3	3.82 _†	3.83 _*	3.83 _†	2 _§	1.8 [*]	—
10^4	3.82 _†	3.83 _*	3.83 _†	0.6 _†	1.5 [*]	—
10^5	3.82 _†	3.83 _†	3.82 _*	0.2 [*]	0.4 [*]	0.4 [*]
10^6	3.82 _†	3.83 _§	3.82 _*	0.1 [*]	0.5 [*]	0.4 [*]
10^6	—	3.83 _§	—	—	0.4 _§	—
10^7	3.82 _†	3.83 _§	3.82 _*	0.1 [*]	0.2 _§	0.3 [*]
10^8	—	3.83 _§	—	—	1 _§	—
8.6×10^8	3.83 _†	—	—	4 ^{**}	—	—
$9-9.2 \times 10^8$	—	3.83 _§	3.83 _†	—	0.8 _§	1.2 [*]
$23-30 \times 10^8$	3.84 _§	—	3.81	—	—	4
1723 Glass						
10^3	6.28 _†	6.318 [*]	6.32 _‡	8.3 [*]	8.8 [*]	—
10^4	6.27 _†	6.308 [*]	6.30 _†	8.8 [*]	9.2 [*]	—
10^5	6.26 _†	6.29 _†	6.30 _§	9.8 [*]	9.1 [*]	11.2 [*]
10^6	6.25 _§	6.29 _†	6.27 _†	11.7 [*]	10.7 [*]	12.7 [*]
10^6	—	6.29 _§	—	—	10.9 _†	—
10^7	6.24 _§	6.28 _§	6.27 _†	15 _§	15 _†	14.0 _†
10^8	—	6.28 _§	—	—	20 _†	—
8.6×10^8	6.19 _†	—	—	54	—	—
$9-9.2 \times 10^8$	6.22 _§	6.25 ^{**}	6.20 _†	59	53 ^{**}	53.6 _§
$23-30 \times 10^8$	—	—	6.16	—	—	77
995 Alumina						
10^3	9.44 _†	9.43 [*]	—	5 ^{**}	14 _§	—
10^4	9.43 _†	9.41 _†	9.48 _†	3.6 _†	7 _§	—
10^5	9.43 _†	9.43 _†	9.44 _†	2.0 _†	4 _§	2.6 _†
10^6	9.42 _§	9.43 _†	9.44 _†	1.2 _†	1.6 _†	2.5 _†
10^6	—	9.43 _§	—	—	2.2 _§	—
10^7	9.42 _§	9.43 _§	9.43 _†	1 _§	1.8 _§	2.6 _†
10^8	—	9.43 _§	—	—	0.4 _§	—
8.6×10^8	9.43 _†	—	—	4	—	—
$9-9.2 \times 10^8$	—	9.41 _§	9.41	—	1	2.0 [*]
$23-30 \times 10^8$	—	—	9.37	—	—	3.7

Note: Superscripts * to ** indicate the estimated usual limits of error of the apparatus used.
 * = ± 0.2 per cent in k' or $\pm 2 \times 10^{-4}$ in $\tan \delta$.
 † = ± 0.3 per cent in k' or $\pm 3 \times 10^{-4}$ in $\tan \delta$.
 ‡ = ± 0.4 per cent in k' or $\pm 4 \times 10^{-4}$ in $\tan \delta$.
 § = ± 0.5 per cent in k' or $\pm 5 \times 10^{-4}$ in $\tan \delta$.
 ** = ± 1 per cent in k' or $\pm 10^{-4}$ in $\tan \delta$.

same sample from 40 Hz to 9 GHz, although for optimum results a thicker sample is sometimes required for 9 GHz.

The samples supplied were all two inches in diameter and after lightly lapping them in our workshop to improve their accuracy the thicknesses were as follows: fused silica 2.0485 mm, glass 2.2294 mm, alumina 2.5517; the variation in thickness still being +0.0076 and -0.0031 mm. Any further lapping would have made the samples too thin for accurate measurement.

The results of the first series of measurements are given in Table I.

The fused silica was too thin for measurements at 9 GHz with optimum accuracy; the results given are for a thicker sample. The alumina was affected by moisture. The values given at 1 and 10 kHz are for the sample taken straight from the desiccator. Higher values of permittivity were obtained later in the day, and they were even higher after being left in the laboratory overnight under the mercury clamps. The accuracies quoted represent the measurement accuracy and do not include any humidity effect. The daytime relative humidity was about 55 per cent and the temperature throughout the measurement was 20 ± 1°C. The errors shown are those normally given for the apparatus. The error of 0.5 per cent for the Dielectric Test Set results from errors in estimating the head reading and also because an inductance effect in the head gives a higher than true value at 100 MHz. A tan δ = 0.00002 measured on this instrument would normally be quoted as <0.00005.

C. National Bureau of Standards Description

The radio-frequency measurements were made with a two-terminal capacitor sample holder of the Hartshorn-Ward type [3]. The samples were washed, then dried at 75°C. Measurements were obtained 1) in a dry condition, by enclosing the holder in plastic and 2) when the specimens were in equilibrium with the humidity of the room. The real part k' was obtained with tinfoil secondary electrodes attached with petrolatum. The loss was obtained on the bare specimen.

The capacitance at 10 and 100 kHz was obtained with a ratio-arm capacitance bridge having a resolution of 10⁻⁴ pF. The loss tangent was obtained separately from a Schering-type bridge having higher conductance resolution.

At 1 MHz a high-precision conductance-capacitance bridge was used. This same bridge was used at 10 MHz, at which frequency the capacitance resolution was still good though the conductance resolution was poor. Therefore, at 10 MHz additional observations were made, especially of the loss, by resonating the holder on a Q meter.

The measurements at 9.2 and 30 GHz were obtained from H₀₁₁ resonators that were approximately identical, except in size. The sample diameters were usually 0.25

and 0.075 inch at the two frequencies. The method of splitting the degeneracy of the E₁₁₁ mode is new and better than previously reported [9] in that no coupling interaction is detectable. A shallow groove was cut around the cylindrical wall approximately at the location of the coupling irises, thus enhancing the splitting by the irises. The calculation of the real part of the permittivity k' takes into account the changes of the perturbations as the sample is inserted. These perturbations are of the irises, the groove, and the sample hole.

The microwave loss as reported in Table I was obtained from the change in transmission of the resonator, taking into account the change in coupling produced by the sample [9]. The loss could also be obtained from Q width measured by frequency and by length variations [9]. A comparison of the three methods of obtaining the loss of the glass gave the following results:

53.6, by ΔT 53.4, by Δf 53.7, by ΔL

where T, f, and L designate the transmission, frequency, and length measurements, respectively.

The assigned uncertainties (uncertainty in this connection means the estimated maximum error due to both random and systematic errors) of the NBS measurements (see Table I) were estimated as follows. For k' at RF frequencies the measured change of capacitance, by varying the gap of the holder, contributes 0.05 per cent. The sample thickness uncertainty contributes 0.05 per cent. The repeatability of applying foil and contacting it is 0.1 per cent or less. Another 0.1 per cent is added for systematic errors. At 9 GHz, the uncertainty of 10⁻⁴-inch in rod diameter contributes ±0.15 per cent in k', and the changing perturbations contribute ±0.1 per cent. The uncertainty of the loss tangent is obtained from the uncertainty of the conductance balance for bridge measurements and at microwaves from the uncertainty of the Q and transmission changes.

The temperature and humidity were approximately 23°C and 40 per cent. k' is relative to vacuum.

IV. RESULTS

Table I gives the results obtained by each laboratory, usually based on the average of two specimens. No large variations are obscured, however, by such averaging, as may be seen from previous discussions of individual variations and homogeneity checks. The errors shown in Table I are the errors of the apparatus and the measuring operations and do not include variability of samples as caused, for example, by humidity differences between laboratories.

Figures 1 and 2 are plots of the results vs. frequency for each laboratory. The general range of differences between the laboratories for k' is shown by the band. The differences on loss are not so easily described and may possibly arise from the samples themselves. Further study of the loss differences is needed.

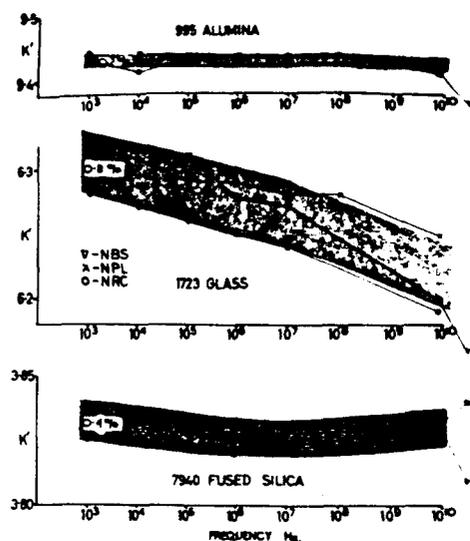
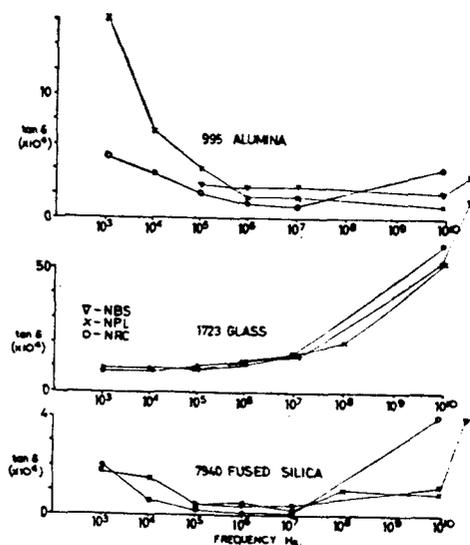
Fig. 1. Plots of k' vs. frequency.

Fig. 2. Plots of loss tangent vs. frequency.

V. STATISTICAL CONSIDERATIONS

The present work was not designed beforehand for a statistical treatment of inter-laboratory comparisons. We can, however, give an approximate and limited treatment, partly as a reminder for future comparisons. The central question is to decide whether systematic differences between laboratories exist. Student's t test, and more generally the F test, gives such a decision [10]. Results from two laboratories only will be compared.

As previously indicated, inhomogeneity of the material from which the specimens are cut may contribute to an apparent systematic difference if it is not taken into account. No inhomogeneity was confirmed in the analysis of the disk specimens (again analyzed by the t test). Actually none was to be expected inasmuch as the disk samples of the two laboratories came from locations which were close together.

TABLE II

t TEST OF DIFFERENCES BETWEEN LABORATORIES AT 100 kHz

Material, Quantity	s_{x1} and s_{x2} NRC NBS	$\bar{x}_{NRC} - \bar{x}_{NBS}$	t'	Significant at 5 per cent level
Silica: k'	0.003, 0.001	± 0.000	0	No
$10^4 \times \tan \delta'$	0.2 0.1	-0.2	1	No
Glass: k'	0.004 0.002	-0.046	13	Yes
$10^4 \times \tan \delta'$	0.2 0.3	-1.4	4	No
Alumina: k'	0.002 0.001	-0.010	6	No
$10^4 \times \tan \delta'$	0.4 0.2	-0.6	1	No

The result \bar{x} of NRC is based on two samples. The result \bar{x} of NBS is based on one sample of silica and glass and two samples of alumina. The dispersion of NBS was obtained by measuring the two NRC samples, but the absolute measurements could not be compared with each other because the larger disks of NRC had a small fringing-field error in the NBS holder.

We compare two determinations of x by the statistic

$$t' = (\bar{x}_1 - \bar{x}_2) / (s_1^2 + s_2^2)^{1/2}$$

where \bar{x}_1 and \bar{x}_2 are means of the quantities x_1 and x_2 , and s_1^2 and s_2^2 are the variances of these means, e.g., $s_1^2 = s_{x1}^2 / n_1$ where s_{x1}^2 is the variance of determining x_1 and n_1 is the number of specimens entering into the mean \bar{x}_1 . Then t' is compared with a tabulated t value to test for significance. (Our t test must be considered to be somewhat approximate because we have not checked whether necessary conditions, e.g., a normal distribution, are satisfied.)

Some of the disk specimens came from locations one above the other, separated by very little distance, and were assumed to display no inhomogeneity. Measurements on these were used to estimate the standard deviation of the measuring operation s_x . Table II gives the standard deviation of measurement so obtained, the difference between laboratories, and t' . The number of samples is three of silica or glass and four of alumina. Only the difference between laboratories of k' of the glass material is significant. This difference on k' of glass may exist in all of the low-frequency measurements, judging from the plots in Fig. 1. Paragraph 3 of the NRC description has already remarked on this problem with glass. A similar analysis of the microwave work showed that differences in $\tan \delta$ of the two laboratories are significant but that differences in k' are not.

Future comparisons should be statistically designed specifically to find inhomogeneity by mixing up the locations of specimens furnished to the various laboratories. Also all factors of the measurement should be varied in a way that easily furnishes the overall variance. A difficulty with the present analysis was that some factors, e.g., thickness, stayed constant during repetitions.

VI. DISCUSSION AND CONCLUSIONS

The various methods used have salient features which may be of interest. The air-gap method of NRC is experimentally convenient because the somewhat diffi-

cult task of applying electrodes is avoided. At the same time, more precision is required in measuring the thickness and the electrode spacing. The NPL system is certainly convenient because it requires only one sample to cover the radio and microwave ranges. The H_{011} resonator method of NBS has proved to be accurate and convenient at 9 GHz and reasonably so at 30 GHz.

It may be concluded that the comparison has been worthwhile, especially because it gives a good indication of the actual limits of accuracy, whereas the errors in Table I are those which the respective laboratories would have assigned in the absence of a comparison. The measurements of k' agree within 0.2 per cent, 0.4 per cent, and 0.8 per cent for alumina, silica, and glass, respectively, for all frequencies. The loss tangents agree to 1×10^{-4} for silica and 2×10^{-4} for alumina between 10^6 and 10^8 Hz. The microwave losses obtained by cavity methods at NPL and NBS are in reasonable agreement. The loss from the slotted line of NRC is systematically higher according to the t test. The agreement between laboratories may be influenced by the fact that we have not all measured the same samples under the same ambient conditions. More work is required. Statistical design should be used in future work.

ACKNOWLEDGMENT

The authors wish to thank Dr. E. L. Crow for his suggestions concerning the statistical discussion, the Corning Glass Works, N. Y., and the Coors Porcelain Company, Golden, Colo., for furnishing various samples.

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Added in Proof

The largest discrepancy in permittivity values between the three laboratories was found for the 1723 glass. Recently (March, 1965) measurements were made at frequencies of 1, 10, and 100 kHz by NRC using a different method, the two-fluid method [11]. This method should give more accurate results than the air-gap method used previously. Values of the permittivity of the 7940 fused silica and the 995 alumina agreed to within 0.1 per cent of the values previously obtained by NRC. However, the values for the 1723 glass obtained by the new method were consistently 0.4 per cent higher than the values obtained previously, resulting in better agreement with NPL and NBS. At present the reason for this change is not known.

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