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Spectrophotometric studies of ultra low loss optical glasses II: double beam method

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Abstract A double beam spectrophotometer is described, suitable for the measurements of very low attenuation coefficients in glass in the wavelength range 500–1000 nm. The instrument is shown to have a balance stability of $\pm 1 \times 10^{-5}$. The systematic errors involved in the use of the long samples required for these measurements are studied. The instrument is shown to be satisfactory in making measurements of attenuation coefficients of 0·0001 cm^{-1} with an accuracy to $\pm 0{\cdot}000\,01$ cm^{-1} when sample pairs of 20 cm length difference are used. Results of measurements on very low loss silica, which illustrate the performance of the instrument, are given.

1 Introduction

The use of cladded glass fibres as a possible transmission medium for optical signals has stimulated this study of attenuation in glasses. For this purpose glasses possessing attenuation coefficients of less than 0·0001 cm^{-1} are required, and the measurement of such low attenuation coefficients is beyond the range of normal spectrophotometers. A single beam instrument for such measurements has been described (Kao and Davies 1968) and forms part 1 of this study. This was shown to be suitable in making measurements to a limit of $\pm 0{\cdot}000\,05$ cm^{-1} when sample pairs of 20 cm length difference are used.

A double beam instrument capable of more accurate measurements is described in this paper. The basic balance stability of the instrument is examined, and the problems of systematic errors on measurements with long samples considered. Results are given of measurements on samples of silica having very low attenuation coefficients and these illustrate the performance of the instrument.

2 Description of the instrument

A schematic diagram of the double beam spectrophotometer is shown in figure 1. The instrument differs essentially from conventional types in providing a large light path (~70 cm) for the insertion of samples and attenuators. This is necessary to accommodate the long glass specimens, up to 25 cm, which are required in accurate measurements of attenuation coefficients of less than 0·0001 cm^{-1}. In designing the system an arrangement with a small number of optical components was sought, and similarity of the paths of the two beams was required. Particular care was taken to obtain a mechanically stable set-up which was free from vibrations. For this reason a fairly massive base plate is used in the chopper assembly, and high quality bearings are employed in the chopper mounting. Flexibility was maintained in the positioning of the lenses and mirrors, and individual components were designed to be adjusted in their mountings and locked in position.

The light source is a 12 v, 50 w tungsten–iodine filament lamp. Good electrical contacts to the tungsten pins of the lamp are necessary for a stable output; spot-welding of nickel tape to these was found convenient and satisfactory. The current for the lamp is supplied by a transistorized power pack

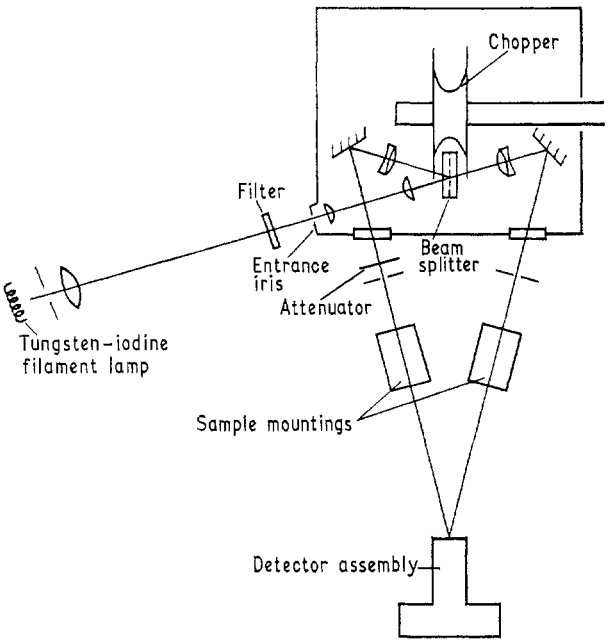

Figure 1 Schematic diagram of double beam spectrophotometer

in parallel with a large capacity 12 v lead–acid accumulator. A resistor in series with the lamp reduces the voltage across its terminals to 11 v, increasing the lamp life without seriously affecting its output. The potential difference across the lamp terminals was measured with a digital voltmeter and showed no fluctuation, indicating a voltage stability of better than 1×10^{-4}.

The lamp filament is imaged on an iris diaphragm by a condenser lens. Adjustment of this diaphragm and that adjacent to the condenser lens allows variation of beam diameter from 3 to 10 mm. These are adjusted together with the other lenses to give a well-collimated beam to the detector.

The selection of light of the required wavelength is done by the use of filters placed in the beam after the condensing

lens. A set of filters for the wavelength range 500–1000 nm having a half-bandwidth of 10 nm is used for this purpose. Since absorption bands in glasses are broad at these wavelengths this coverage is adequate.

The beam splitter consists of a partially reflecting aluminium film sandwiched between optical flats 6 mm thick. Although the flats are bloomed to reduce reflected images, significant secondary beams exist. These are removed by iris diaphragms in the light path. The relative intensities of the reflected and transmitted beams change with wavelength; this means that there is a slightly different spectral distribution of light in each of the beams for a particular band of light. The difference in reflection loss due to this effect is negligible with glass samples. Care must be taken, however, in interpreting measurements of loss at wavelengths near absorption edges.

The beams transmitted and reflected by the beam splitter are cut sequentially by a precisely constructed double chopper. This is driven by a $\frac{1}{2}$ h.p. motor run from a Servomex M.C.47 controller. The speed stability of the motor was measured by a counter to record the frequency of a harmonic of the reference signal. Variations of $\pm 2 \times 10^{-4}$ were noted in a period of several minutes, after the motor had been run for some time. This is within the value specified by the manufacturer. A small light source is placed between the blades of the chopper and photodiodes fixed on opposite sides. The positions of the photodiodes are adjusted to provide reference signals in phase and 90° out of phase with the chopped beams.

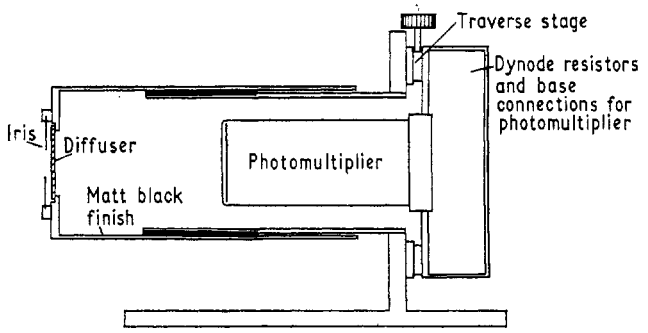

Figure 2 Detector assembly

The samples are clamped in V-blocks which are placed on adjustable mountings. Location points are provided to minimize the change in V-block position with repeated replacement. Evidence from measurements showed that this is satisfactory. Attenuators are required to balance the two beams. A simple system of neutral density filters was found to be satisfactory in practice. These are placed in the beam at a slight angle so that reflected light from the filter is directed away from the detector. Alteration of this angle gives some small variation in the transmitted light and this provides a means for fine adjustment. These filters are very thin so that the beam displacement caused can be ignored.

Reflected light can easily give rise to measurement errors in the instrument. In particular care must be taken to ensure that the introduction of samples into the two beams does not direct unwanted light back to the detector. For this reason, for example, the detector is not symmetrically located with respect to the two beams, but angled slightly so that light from one beam reflected off the front face of the diffuser is not reflected back when a sample is put in the other beam.

The mounting for the photomultiplier used as a detector is shown in figure 2. This allows movement of the photomultiplier in the detector head of ± 10 mm in two directions perpendicular to its axis. Adjustment of the photomultiplier is made for a maximum signal with one beam incident on the diffuser. The beam spot is therefore on the 'effective' axis of the photomultiplier, and systematic errors due to beam diameter change and displacement are minimized. In addition the diffuser–photomultiplier distance is variable from 5 to 15 cm. The mounting accommodates an EMI 9558B photomultiplier (S-20 response) or a Mullard 150 CVP photomultiplier (S-1 response).

The out-of-balance signal is detected by a phase-sensitive system (Aim Electronics system 5-1). In practice some difficulty is caused by the 'spikes' present in the balance signal at crossover point. These give rise to inconvenient overloading of the phase-sensitive detector, and limit the balance stability. A spike suppression circuit is used to overcome this problem, in which the spike is replaced by a constant level signal bounded by constant amplitude spikes of very narrow width. The output from the photomultiplier load is then led successively from the spike suppression circuit through the phase-sensitive detection system and to a pen recorder.

In order to reduce the possibility of sample contamination during measurements a clean-air tent is mounted over the spectrophotometer on the steel supporting table.

3 Balance stability

A fundamental limit to the balance stability is imposed by the discrete nature of the emission of electrons from the photocathode, the so-called shot noise. In order to examine other factors influencing the balance, these fluctuations may be reduced to 1×10^{-5} by use of a high light intensity and suitable choice of circuit time constant in the phase-sensitive detector output. A recorder trace of the balance stability is then taken, sufficient time having been allowed for the various components of the instrument to be stabilized. The figures of balance stability quoted here are estimated r.m.s. fluctuations, taken as one quarter of the deviation containing most of the trace, when drift was absent. The detailed observations were made with an EMI 9558B photomultiplier with an S-20 response. Similar results were obtained for a Mullard 150 CVP photomultiplier with an S-1 response.

Experiments showed that in the absence of a spike-suppression circuit a factor limiting the balance stability is the speed variation of the motor driving the chopper. Fluctuations in the motor speed cause changes in the reference frequency. The phase-shift amplifier is frequency dependent, and thus variations in the phase of the reference signal are produced. The 'spike' signal at the crossover point is 90° out of phase with the reference signal. Variations of the phase of the reference signal cause varying components of this to be seen at balance. It was shown that a motor speed change of $\pm 2 \times 10^{-4}$ (the short term speed stability of the motor) would give balance changes of $\pm 6 \times 10^{-5}$.

The balance stability was measured with different beam intensities. An estimate of the expected shot noise fluctuation was made from the signal strength and the known photomultiplier response. The two values agreed within the experimental error for stabilities up to $\pm 1 \times 10^{-5}$ (time constant $T = 1$ s) over a period of several minutes. However, at the highest stabilities the balance was found to be best characterized by two parameters: a rapid fluctuation due to shot noise, and a slow fluctuation describing the balance change over longer periods of up to one hour. For example, in two experiments over periods of 15 minutes, the r.m.s. rapid fluctuations were estimated to be $\pm 1 \times 10^{-5}$ ($T = 1$ s) and less than $\pm 1 \times 10^{-5}$ ($T = 5$ s). The balance changed slowly in these experiments up to $\pm 5 \times 10^{-5}$ ($T = 1$ s) and $\pm 4 \times 10^{-5}$ ($T = 5$ s), the range indicating the maximum deviations from balance.

The balance stability of interest so far as absorption measurements are concerned is that over the time required for sample insertion and measurements, i.e. several minutes. The results above therefore, show that the intrinsic balance stability of the instrument is satisfactory for measurements to $\pm 0{\cdot}000\,01$ cm^{-1} with sample pairs of 20 cm length difference. The cause of the long-term slow fluctuations in balance, observed to be of the order of $\pm 5 \times 10^{-5}$ in 1 hour, has not been found. The fact that they are slow makes experimental measurements difficult, but the results suggest that the fluctuation is random about the mean. A possible cause is slow variation in the light source, since only a small part of the filament is imaged on the entrance iris diaphragm.

4 Systematic errors

The introduction of samples into the light beams causes signal changes due to systematic errors in addition to those due to loss. These errors have been studied in some detail with the object of reducing their magnitude to less than 2×10^{-4} in a typical measurement.

The two important systematic errors are recognized: (i) beam displacement and (ii) beam diameter change. The former arises, in the case of samples with parallel end faces, because of alignment errors in the beam. The latter is a consequence of a change in focus position of $b(n-1)/n$ for paraxial rays, when a sample of length b and refractive index n is placed in the beam. It is obviously more important in measurements with long samples. In order to minimize the change due to these two effects, a detector arrangement is required so that small changes in beam spot position cause little change in signal. The detector assembly (figure 2) has a positional response which depends on the distance d between the diffuser and the photocathode. If it is assumed that the diffuser is Lambertian, the positional response may be calculated for the arrangement shown. The signal I_0 will change by $-2I_0r^2/d^2$ for a displacement r from the 'axis' position (for small values of r/d).

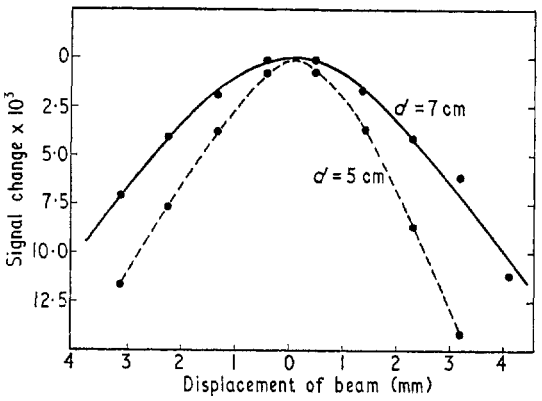

Figure 3 Detector response with displacement for plane diffuser. Photocathode–diffuser distance d, beam diameter 3 mm

The response of the detector with respect to position of beam spot on the diffuser can be examined by displacing the photomultiplier in the detector mounting and noting the change in signal. It is difficult to measure small changes in this way, however, since only a single beam is used. A more satisfactory method made use of the adjustment of a good quality long glass sample in one of the beams. The iris diaphragms were adjusted to give a beam spot of 3 mm diameter on the diffuser, and a displacement up to $\pm 4{\cdot}5$ mm obtained by sample adjustment. In this manner use is made of the better stability of the double beam system. The results obtained are shown in figure 3 for distances between the photocathode and diffuser of 5 and 7 cm. These show a response of the parabolic form anticipated by theory, but signal changes are larger than predicted by the formula above. The difference is due in part to the finite diameter of the beam which gives a larger 'effective' displacement, and in part to the approximation made in the simple theory.

The beam displacement caused by the insertion of a sample with parallel end faces in the beam at an angle α is $\alpha b(n-1)/n$ (notation as above). This means that the alignment for long samples (~ 20 cm) must be very accurate, to better than 3 minutes of arc, if displacements of less than 0·1 mm are to be caused. Also, the sample has to be removed from the beam and replaced without significant changes in alignment. The beam spot was observed on a graduated screen placed in front of the detector. Repeated insertion of samples and realignment suggested that the beam displacement produced was not more than 0·2 mm. This method has the advantage that the parameter of interest is measured directly, but accuracy is limited by the blurred edges of the beam, and also the beam diameter changes for long samples. In the case of good samples with parallel end faces it was found more convenient to align by means of the light reflected from the end faces. The apertures in the beams are stopped down to 1 mm and the samples adjusted for coincidence of the reflected beams and the apertures. This gives an estimated alignment accuracy to a few minutes, since the distance between aperture and farther reflecting sample face is about 30 cm. All these observations taken in conjunction with the response of the detector with position of beam spot (figure 3) indicate that the systematic error due to sample alignment should be less than 1×10^{-4}. Experiments carried out with repeated insertion and realignment of samples confirmed this, showing changes of not more than 2×10^{-4}.

In order to minimize the change in diameter of the beam spot on the diffuser when a sample is placed in the beam, the beam collimation needs to be optimum in the region near the detector assembly. Adjustment of the optical system is made and a long sample placed in the beam. The change in beam spot diameter on a screen in front of the detector is readily seen, but blurring of the edges makes exact measurement of the change difficult. This does, however, provide a satisfactory means of optimizing the beam collimation. In order to examine the beam intensity distribution in more detail further experiments were carried out. A 1 mm diameter aperture was traversed across the beam diameter close to the detector and the change in signal with position noted. A long sample was placed in the beam and the process repeated.

Results for a sample of length 23·9 cm ($n = 1{\cdot}67$) are shown in figure 4. Correction has been made here for attenuation and reflection losses, so the curves are normalized. Measurements of this kind suggested that a simple observation of the change in beam intensity in the central region provided a quick and simple method of estimating the beam spot diameter change on sample insertion.

A knowledge of the positional response of the detector and the beam diameter change enables a calculation of the systematic error to be made. Figure 5 shows how this varies with beam diameter for distances between the photocathode and diffuser of 5 and 7 cm and a beam diameter change of 10% on sample insertion. In addition curves are drawn showing the shot noise balance stability (time constant 1 s) at 545 and 825 nm. These are extrapolated in the form expected from single measurements at a beam diameter of 5 mm and a diffuser–photocathode distance of 7 cm. A photomultiplier with an S-1 response is used here. It can thus be seen how the compromise between balance stability and systematic errors is achieved.

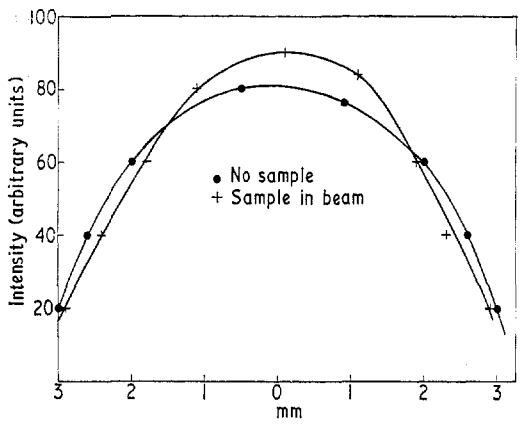

Figure 4 Change in intensity distribution across beam on insertion of sample of length 23·9 cm ($n = 1{\cdot}67$)

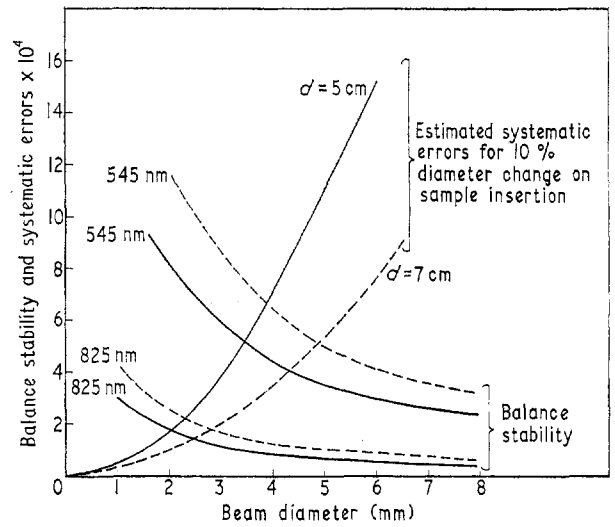

Figure 5 Variation of balance stability (time constant 1 s) with beam diameter (at 545 nm and 825 nm) and estimated systematic errors for different photomultiplier distances d (5 cm, 7 cm)

Some further experimental confirmation was sought to verify estimates of the systematic error due to beam diameter change. Measurements were made using some very low loss silica samples, and the systematic error was altered by changing beam diameter and divergence. It was considered important to try to make observations with the normally used detector arrangement where the systematic error is expected to be very small. The results obtained are shown in figure 6 which shows reasonable correlation between theoretical and experimental changes in spite of their small size.

5 Sample requirements

In order to make measurements of attenuation in glass at the resolution of the double beam spectrophotometer, stringent conditions are imposed upon the samples. A pair of samples must have end faces parallel, preferably within one minute of arc. These samples are cut from a rod of the specimen glass and polished under identical conditions. This is important since the measurements assume equality of reflection losses at the sample surfaces. Confirmation of the similarity of the surfaces is obtained by ellipsometry studies (Wright and Kao 1969). This technique is very sensitive to changes in the

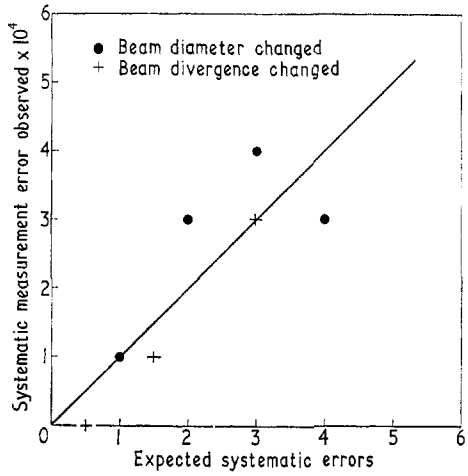

Figure 6 Correlation between observed measurement changes and expected systematic errors

surface and shows that a surface film of lower refractive index than the bulk is characteristic of glasses. An exception is the case of fused silica for which a film of higher refractive index is observed. Measurements on well-polished samples generally gave no difference within the experimental error of the instrument.

Typically a pair of samples of lengths 5 and 20 cm with a cross section of 2 cm (square or circular) has been found convenient for attenuation measurements. The standard preparation procedure adopted is to clean the sample with Teepol and water, scrubbing the end faces gently with a tissue, rinse in deionized water and vapour-degrease for an hour in acetone. The samples are stored in a desiccator prior to measurements.

Care in sampling handling is important, to avoid damage to the end surfaces. Any slight scratches will cause measurement inaccuracies. Contamination of the sample by dust and fragments of cleaning tissue is also a problem. In spite of these difficulties it appeared that visual observation of the surface was a reliable guide to the cleanliness. Changes of up to 1×10^{-3} in measurements were seen with marked dust contamination. The surface after thorough cleaning showed no tendency to collect dust provided it was untouched after the vapour-degreasing. Dust particles can be removed by a fine camel-hair brush, but recontamination is then rapid.

6 Measurement procedure

The samples are cleaned and placed in the V-block holders. Alignment of the samples in the beams is then carried out. The samples are removed, a filter is selected and the attenuator adjusted to balance the two beams. The large variation in the beam signal with wavelength makes it necessary to alter the voltage across the dynode resistor chain of the photomultiplier. This is done to give a signal level of 0·2 v for one of the beams. Fine adjustment of the amplifier gain allows the output signal (i.e. 100%) to be set to a conventional level. The amplifier gain steps are used in conjunction with a pen recorder to give a sensitivity suitable for the loss difference to be measured. The time constant of the output circuit in the phase-sensitive detector is adjusted as necessary.

The balanced beam output is recorded for about 1 minute when steady, the recorder is then switched off and the input to the phase-sensitive detection system shorted. The two samples are placed in the beams, the circuit is reconnected, and the change in signal level recorded for 1 minute. The samples are then removed and the balance level again

recorded. This process, which takes about 5 minutes, is repeated if there is any significant balance drift. The loss measurement is accurate to 5%. The whole procedure is repeated for a series of wavelengths over the range 500–1000 nm, taking up to 2 hours. This procedure is satisfactory in measurement of attenuation differences down to 2×10^{-3}, but with smaller changes some further precautions are taken.

A set of measurements is made in the manner described above, care being taken, by repeating some measurements, to ensure that no changes in the sample occur in the process. It is assumed that although this set of results may suffer from a systematic error, differences in attenuation at different wavelengths are correct. Detailed study is then made of the loss at the wavelength of minimum loss. Sample re-cleaning and re-alignment are carried out until reproducible results are obtained. The systematic errors are estimated and correction made for them. The end faces of the samples are examined by ellipsometry to check similarity of surfaces.

7 Performance of instrument

Some measurements of attenuation which illustrate the resolution of the instrument are shown in figure 7. These are for samples of fused silica of different types (obtained from the manufacturers, Schott and Corning). The difference in length between the long and short samples was 15 cm.

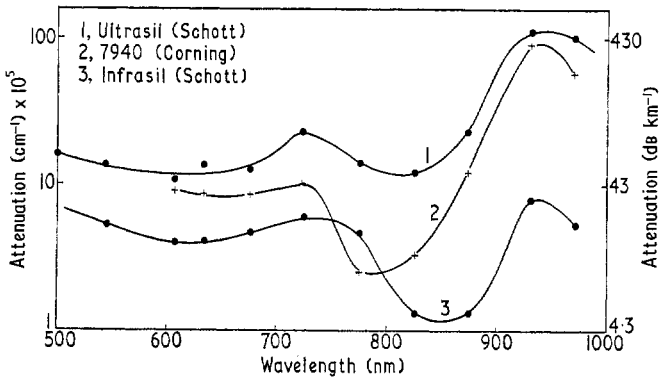

Figure 7 Attenuation in silica samples

It is of interest to compare the curves 1 and 2 as the silica here has a high transmission in the ultra-violet region. This type of silica usually has a high 'water' content and gives characteristic absorption bands in the infra-red (Adams and Douglas 1959).

Attenuation peaks are shown on the curves at 930 nm and 730 nm. These, it is suggested, are due to the third and fourth 'overtones' of the fundamental absorption at 2730 nm due to OH bonds in the silica.

Curve 3 shows the attenuation in Infrasil (Schott) which is designed to have a high transmission in the infra-red, and hence has a low 'water' content. In spite of this, attenuation peaks which we ascribe to OH bonds are indicated. The extremely low attenuation measured in the region of 950 nm was the subject of some detailed study. The loss difference observed on insertion of samples was less than 1×10^{-4}. A significant result was obtained only after the correction for systematic errors was applied, giving a positive loss of 2×10^{-4}.

The above measurements show that the instrument can be used to determine attenuation coefficients of 0·0001 cm^{-1} with an accuracy to $\pm 0{\cdot}000\,01$ cm^{-1}, providing suitable samples having a 20 cm length difference are available.

Acknowledgments

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References

Adams R V and Douglas R W 1959 *J. Soc. Glass Tech.* **43** 147T–158T

Kao K C and Davies T W 1968 *J. Sci. Instrum. (J. Phys.* E) **1** (Ser. 2) 1063–8

Wright C R and Kao K C 1969 *J. Sci. Instrum. (J. Phys.* E) **2** (Ser. 2) in the press