

# Measurement of Attenuation in Low-Loss Optical Glass

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An improved spectrophotometer for measuring the bulk attenuation of optical quality bulk glass over the wavelength range 500 to 1000 nm is described. It is capable of measuring loss coefficients equivalent to an attenuation of a few dB/km. Results obtained with lead flint and barium crown glasses are described.

## 1. Introduction

Interest in the possibility of achieving an optical communications system using clad glass fibre transmission lines has led to the need for a better knowledge of the optical transmission properties of low-loss bulk glass. The principal losses encountered are those due to absorption and scattering. For various practical reasons the likely wavelength of operation of a fibre-optical communications system will be in the near infra-red region of the spectrum with the injection laser operating in the vicinity of 900 nm as a likely candidate for the source. At this wavelength the scattering loss in bulk material is small [1] but absorption is still a problem and techniques for its measurement are necessary in order to compare suitable starting materials for fibre manufacture.

The most direct method of measuring absorption is by noting the rise in temperature of a sample when a beam of appropriate radiation passes through it since in this way the effects on the measurement of reflection loss at the surfaces and of scattering are not serious. However, if a black-body source such as a tungsten-filament or arc lamp is used in order to make measurements over a range of wavelengths then with low-loss materials of extinction coefficients of the order  $10^{-4}$  to  $10^{-5}$  cm $^{-1}$  the temperature rise is very small and the technique is not an easy one although satisfactory results have been achieved [2].

Another method which makes use of the temperature rise of the absorbing medium has proved successful [3] with liquids having loss coefficients  $\sim 10^{-4}$  cm $^{-1}$ . The liquid sample is contained in a thin-walled quartz cell which is placed, at the Brewster angle, within a laser cavity. Radiation absorbed by the liquid causes a radial temperature variation about the beam axis and hence a corresponding refractive index variation, resulting in a lens effect. Insertion of the cell into the laser cavity thus modifies the optics of the resonator resulting in a change in the spot sizes at the mirrors. If the variation with temperature of the refractive index is known then the loss coefficient can be calculated from the measured changes in the spot sizes. This method has been attempted in our laboratories with glass samples but since the temperature dependence of refractive index in solids is much less than in liquids the sensitivity is too low for the loss coefficients involved.

A third method involves measuring the transmission loss of radiation passing through the sample. The difficulty in this case is that even with transmission paths as long as 30 cm the attenuation to be measured in glasses having losses  $\sim 5 \times 10^{-5}$  cm $^{-1}$  is 0.1% and occurs in the presence of reflection and scattering losses at the entry and exit faces of 8% or more. The latter must of course be corrected for in some way. As distinct from the two described above, this method measures total attenuation

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and does not discriminate between absorption and scattering. In the single-beam variant of the technique [4] two samples of the same material, but of different lengths, are inserted successively between a source and detector, and the difference in detector readings in the two cases is a measure of the difference in transmission losses of the two samples. If all four end faces have the same properties then the effects of surface losses are cancelled out. The method has several disadvantages. For example, it involves the measurement of a small change in a large standing current in the detector and the stability of both the source and the detector over the relatively long period required to insert and accurately align the two samples is also critical.

We have preferred to use a two-beam version of the technique [5] in which the probing radiation is switched alternately between the two samples so that sensitive AC methods of detection can be used and the restrictions on the stability of source, detector and amplifiers are greatly reduced. Since it seems to be a bad experimental technique to try to measure a small transmission loss in the presence of large and unknown end losses, an attempt was made to use a single sample with the ends cut at the Brewster angle since, in principle at least, the reflection losses can then be made zero. The reflection from a Brewster face is not a sensitive function of incident angle, but analysis shows that it is necessary to keep the angle of the tilt between the axis of the sample and the

plane of polarisation to not more than  $10^\circ$  for a measuring accuracy of 5%. For the same reason the amount of orthogonally, or randomly, polarised light must be kept to no more than  $6 \times 10^{-3}\%$ . While it may be possible, although not easy, to align the entry face sufficiently well, it is difficult to obtain the required degree of parallelism between the entrance and exit faces in long samples. In addition, scattering from the faces, particularly with lead glasses, remains a problem, and alignment at near-infra-red wavelengths is also difficult. It was decided therefore to resort to a two-sample, double-beam method with all end faces perpendicular to the axis. An improved version of this technique is therefore reported, together with detailed results for the variation of attenuation with wavelength over the range 500 to 1000 nm for four high-quality optical glasses.

## 2. Twin-beam, Double-sample, Spectrophotometer

### 2.1. Optical System

The arrangement of the optical part of the spectrophotometer is shown diagrammatically in fig. 1. The components are clamped magnetically onto a heavy plane steel table, with all light paths lying in a plane exactly 20 cm above the surface of the table, in order to simplify alignment. The table is enclosed by a laminar-flow clean-air cabinet to obviate the problem of dust settling on optical surfaces.

The light source is a 24 V, 150 W tungsten

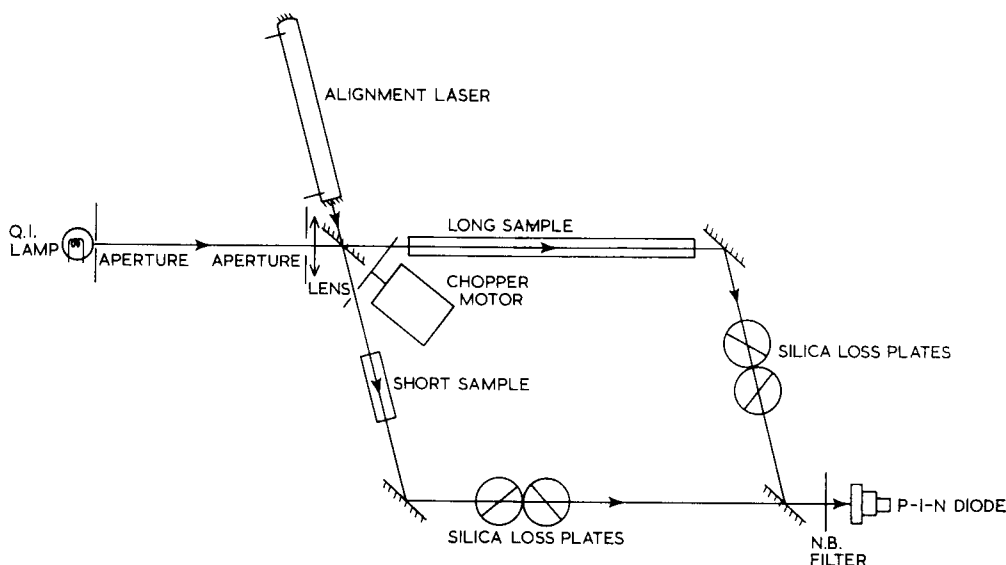


Figure 1 Optical arrangement (schematic) of twin-beam spectrophotometer.

iodine projector lamp, which has a close-wound, flat, spiral filament presenting a rectangular source  $2 \times 3$  mm. It is stopped down by a 2 mm diameter iris, and a 20 cm focal length lens, with a similar stop, is arranged to produce a full-size image of the source iris, 40 cm from the lens. In this manner the light beam travels entirely within a cylinder of 2 mm diameter between the lens and the image; the samples are always placed within the image distance.

The light beam is directed into two paths by a beam-splitting cube, and the two resulting beams are extinguished alternately by a three-bladed chopper disc driven by a synchronous motor. The pair of samples, glass rods 1 cm diameter with plane, parallel ends, are mounted one in each beam; the sample holders are so constructed that they may be removed from the light paths and accurately repositioned. The attitude of each rod may be adjusted using micrometer screws. The two beams are redirected by mirrors, the paths forming a parallelogram, and recombined at the point of intersection by a second beam-splitting cube. In each remaining arm of the parallelogram is placed a pair of contra-rotating silica flats [6] which are arranged to be at equal but opposite (small) angles to the beam axis. Precise control of this angle produces a known attenuation without any net lateral displacement of the beam. These attenuators are used to balance the beam intensities before the samples are inserted.

In an earlier version of this method [7] two photomultipliers were used as detectors in order to cover the wavelength range of interest. However, photomultiplier tubes suffer from the severe disadvantage, for this application, of a variation in sensitivity across the photocathode and this spatial variation changes with wavelength [8]. For accurate results it is necessary, therefore, for both beams to illuminate the same area of the photocathode and to have the same energy distribution. This is not possible to achieve with long samples because of alignment errors and the change in beam diameter. Jones and Kao [7] attempted to alleviate the problem somewhat by placing a scattering screen at distances of 5 cm or more in front of the photocathode. The effective detector sensitivity, which is already poor because of the low quantum efficiency of photo-

emissive surfaces at wavelengths approaching 1000 nm, was thereby greatly reduced.

We have chosen to use a large-area silicon p-i-n photodiode\*. As can be seen from fig. 2 the sensitivity is very uniform at roughly

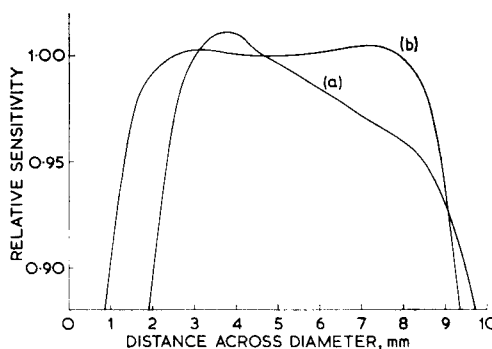


Figure 2 Variation in sensitivity of p-i-n 8-LC photodiode across a diameter which (a) includes the front contact. Curve (b) is for a diameter which is perpendicular to that in (a).

constant distances from the front contact and even in the worst direction, which is along a diameter through the contact, the variation is only 5% over a distance of 5 mm.† The spatial variation of sensitivity is therefore much smaller than with a photomultiplier and the severe restrictions on co-alignment of the two beams are eased. A scattering screen becomes unnecessary and this, combined with the higher quantum efficiency, improves the signal/noise ratio considerably over that given by a photomultiplier. The much smaller size of the detector is, additionally, a great convenience, as is the reduction in cost. The linearity is excellent.

Initial optical alignment of the system is greatly assisted by the use of a small helium/neon laser operating at 633 nm, the beam from which enters the spectrophotometer at the back of the first beam-splitting cube, and is coaxial with the white light beams throughout the rest of the system.

Because of its high brightness the laser beam, including reflections, can easily be seen in the spectrophotometer and once the laser axis has been accurately set the adjustment of all the components following the first beam splitter, including sample insertion, becomes straightforward. The datum level for the beams of 20 cm above the bench is maintained by checking

\*United Detector Technology type PIN-8-LC.

†Subsequent measurements indicate that the variation is less than this.

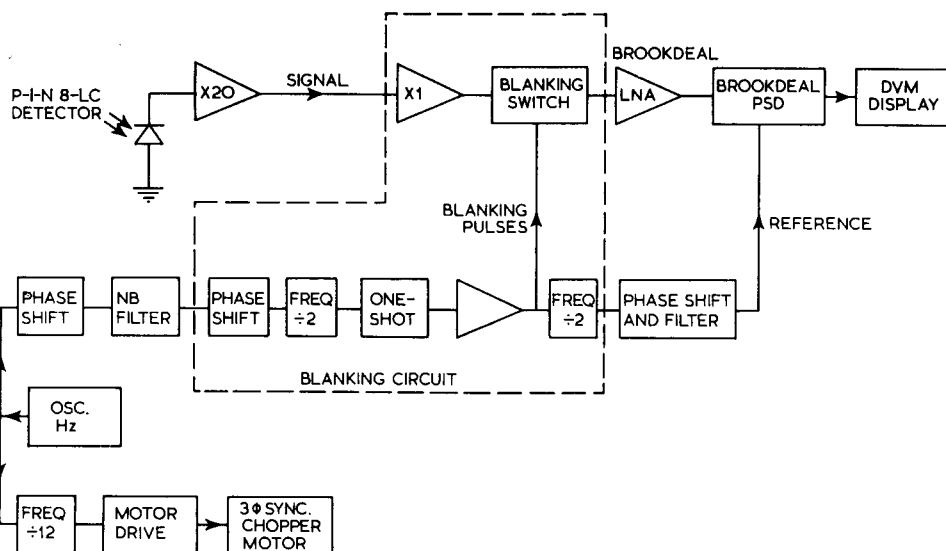


Figure 3 Block diagram of synchronous drive and detection circuits.

with moveable targets, while the final positioning of the beams at the detector is achieved using a white screen with cross accurately concentric with the detector sensitive area, mounted just in front of the detector. The samples are aligned so that no movement of the beam occurs at the detector when a sample is inserted or removed. It is estimated that a beam movement of 0.1 mm on the white screen can be visibly detected.

## 2.2. Electrical System

A block diagram of the electronic circuitry incorporated in the spectrophotometer is shown in fig. 3. The AC output from the silicon detector is a square wave, of which the amplitude represents the difference in intensity of the beams in the two arms of the spectrophotometer. A low-noise pre-amplifier with a gain of 20, located in the detector head, provides initial amplification of the a.c. component of the detector output. In order to remove any uncertainty in the changeover region from one beam to another (where, for example, large spikes may occur due to the brief passage of both beams simultaneously) blank periods are inserted in the waveform twice each cycle while the changeovers occur. Fig. 4 shows typical signals before and after the blanking stage. The processed signal is fed into a Brookdeal low-noise amplifier, type 450, and thence into a Brookdeal phase-sensitive detector, type

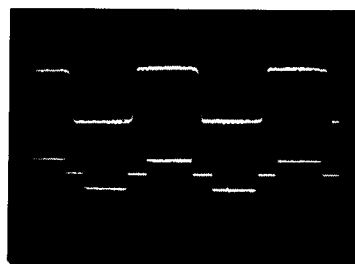


Figure 4 Detector output signal before (a) and after (b) blanking switch.

411, whose output is monitored on a digital voltmeter.

The chopper motor driving frequencies, signal blanking waveform and reference waveform for the PSD are all derived from a master oscillator running at 2335 Hz. The chopper motor is three-phase, and the output from the master oscillator is divided by twelve in a ring counter circuit, the correct phases being fed into three power amplifiers which drive the motor. Because the chopper wheel is three-bladed, the signal frequency is three times the motor drive frequency. The master oscillator output is also fed, via phase-shifting circuits and a frequency-halving circuit, to the blanking pulse generator the output of which drives a double-blanking switch in the signal circuit. The blanking frequency is also halved, and passed through a Brookdeal phase-shifter and

filter, type 421, to provide the reference signal for the PSD.

### 2.3. Calculated Sensitivity of the Spectrophotometer

From a knowledge of the dimensions of the optical system it is possible to estimate the ultimate sensitivity of the spectrophotometer. Assuming that the tungsten-iodine source is a grey body with emissivity  $E = 0.33$ , at a temperature  $T = 3000^\circ\text{K}$ , then the radiated power density may be calculated from Planck's black-body radiation law:

Power radiated,

$$W_\lambda = \frac{E c_1}{\lambda^5 [\exp(c_2/\lambda T) - 1]} \text{ W cm}^{-2} \mu\text{m}^{-1}$$

where the constants have the values

$c_1 = 3.74 \times 10^4$ ;  $c_2 = 1.439 \times 10^4$  for  $\lambda$  in  $\mu\text{m}$ .

For a wavelength,  $\lambda$ , of  $0.9 \mu\text{m}$  then

$$W_\lambda = 1.01 \times 10^2 \text{ W cm}^{-2} \mu\text{m}^{-1}.$$

The interference filters have bandwidths of the order of 20 nm and peak transmissions of the order of 50%. Thus with the stop sizes and beam geometry already discussed, the power in the filtered beam is  $2 \times 10^{-7} \text{ W}$ . At least 75% of this power is inevitably lost in chopping and splitting the beam. The sensitivity of the silicon detector at  $\lambda = 0.9 \mu\text{m}$  is 0.35 A/W, so that the DC signal current in the diode,  $I_s$ , is  $1.8 \times 10^{-8} \text{ A}$ . The measured diode dark current is  $2 \times 10^{-7} \text{ A}$ , so that the diode shot noise,  $\overline{i_{\text{sn}}^2} = 2 e I B$  where  $I$  is the total DC current and mainly comprises the dark current.  $B$ , the PSD bandwidth, is 0.03 Hz giving a mean-squared shot-noise current  $\overline{i_{\text{sn}}^2} = 2 \times 10^{-27} \text{ A}^2$ .

Let the amplitude of the differential-loss square-wave current be  $a \times I_s$  so that the RMS value of the fundamental component of this square wave,  $I_L = a \times 8 \times 10^{-9} \text{ A}$ . Hence the signal/noise ratio at the detector output is

$$\frac{I_L^2}{\overline{i_{\text{sn}}^2}} = 3.3 \times 10^{10} a^2$$

For a signal/noise ratio of 1, the detectable differential loss,  $a = 5.5 \times 10^{-6}$ . With a difference in sample pair lengths of 20 cm it follows that an attenuation of  $2.8 \times 10^{-7} \text{ cm}^{-1}$ , or 0.1 dB/km, is detectable. The theoretical ultimate sensitivity is therefore well within the range required and in practice the resolution and sensitivity achieved will be determined by

experimental factors such as unequal properties of the sample surfaces.

### 3. Experimental Procedure

The glass to be measured is obtained in the form of a rod of 1 cm diameter, from which samples are cut in lengths of approximately 30 cm and 5 cm. The cut ends of the samples are optically polished flat (to  $\lambda/10$ ) and parallel (to  $1'$  of arc). The samples are first examined for strain by placing each in turn in a helium/neon laser beam between crossed polarisers. If strain is present then the samples are annealed. The effect of strain in a 30 cm length of redrawn F7 glass was to produce a marked divergence of the light beam passing through the sample, rendering loss measurement impracticable. Before measurements commence, the sample end faces are washed carefully in spectroscopic grade ethanol. No mechanical cleaning technique is used, as some of the samples are very soft and prone to scratching.

Initial co-alignment of the white light beam and the laser beam involves adjustment of the positions of the laser and tungsten-iodine sources and the first beamsplitter. The two beams emerging from the beamsplitter are at an angle of about  $70^\circ$  to each other. Reflections from the beamsplitting cube faces are thus directed away from the main optical paths and a potential source of error is thereby removed. The chopper wheel intersects each beam just after emergence from the cube. The chopper motor is mounted on foam rubber to isolate it mechanically from other components and prevent vibration. The remaining optical components are aligned using the laser beam and targets, and the beam positions are checked from time to time throughout an experiment. In particular, the coincidence of both beams at the detector is set carefully. The samples are placed in their holders and lightly clamped, after which the attitude of each sample holder is adjusted so that the samples cause no beam deviation. Doubly-reflected beams from the sample ends are then seen to coincide with the main beam at the detector. After alignment, the laser is extinguished.

With the chopper motor synchronised to the master oscillator, phase adjustments are made to ensure that the detected signal is blanked at the correct time, and the reference phase to the PSD is adjusted for maximum PSD output. The photodiode is temporarily obscured, and

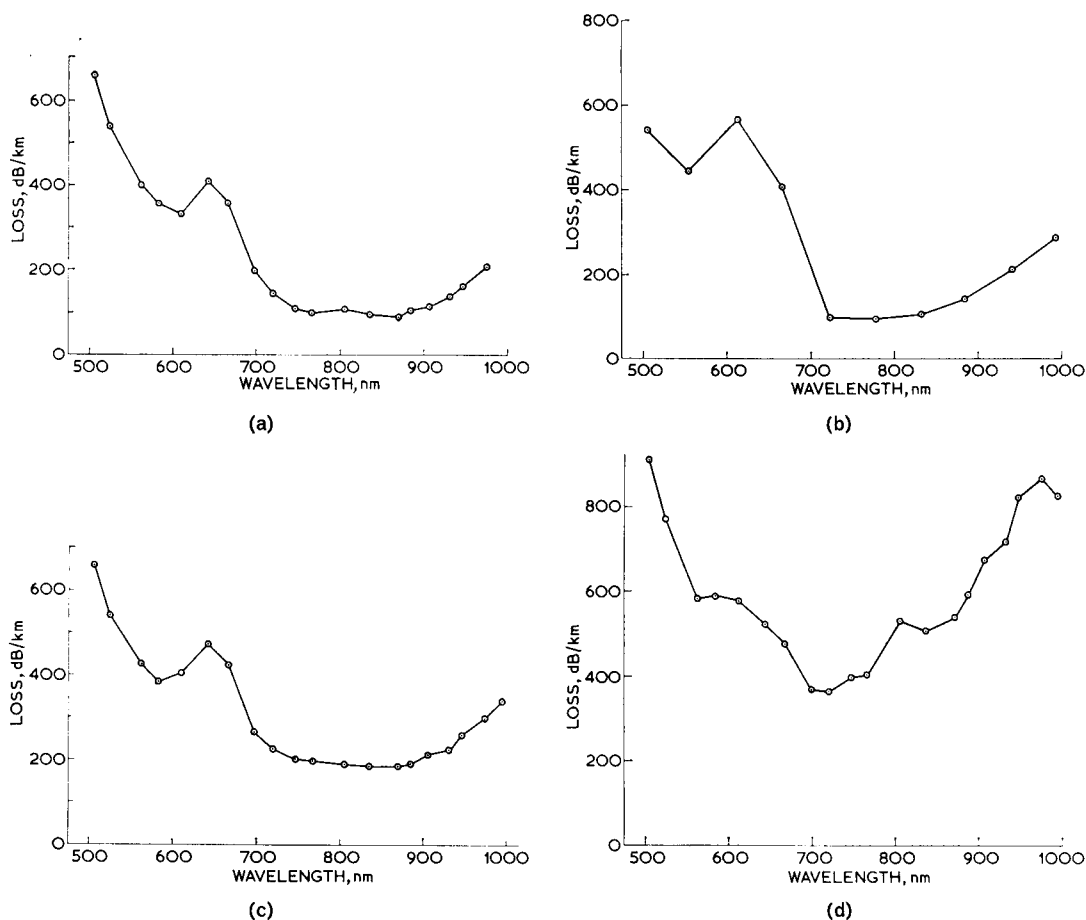


Figure 5 Bulk attenuation as a function of wavelength. (a) glass type F2. (b) glass type F7. (c) glass type F8. (d) glass type SK16.

an electrical zero is obtained for the detection system. With a narrow-band filter of appropriate wavelength in position in front of the photodiode, the two beams are optically balanced for zero output at the PSD by rotating one or other pair of attenuating silica flats. Insertion of the samples into their respective beams produces imbalance in the PSD output which is noted, and represents the differential optical loss. After removal of the samples one beam is blocked to give the relative output for 100% loss. Finally the optical balance of the two unimpeded beams is re-measured to check that no drift has occurred.

A cycle of measurements is performed at about twenty wavelengths through the spectrum from 500 nm to 1  $\mu\text{m}$ . From time to time the electrical zero of the detection system is checked, but after warming-up for about 2 h

adjustments to the detection circuits are rarely needed. Overall noise equivalent to 0.02% optical loss (attenuation equal to  $10^{-5} \text{ cm}^{-1}$  or 4 dB/km) occurs using a 10 s time constant to integrate the PSD output. This figure may be improved by using a longer integration time, but improved accuracy has not been required for the samples measured so far. Averaging over several measurement cycles improves the statistical accuracy, but some measurement uncertainty remains, particularly because of uncertain reflection and scattering losses at the samples' end faces. The method relies on the equality of end losses, which may be affected by thin surface layers formed during optical polishing [9, 10]. Sample-pair ends have been treated identically to reduce inequalities as far as possible. It has been noticed that exposure to the atmosphere of a pair of lead glass sam-

ples allowed visible blotchy surface films to develop making loss measurements impossible and necessitating the repolishing of the samples.

#### 4. Results

Attenuation measurements have been carried out on four types of optical glass, all produced by Schott and Co of Mainz. Three samples, namely F2, F7 and F8, are lead flint glasses, while the fourth, SK16, is a barium crown glass. The F2, F8 and SK16 samples were in the form of rod cut from optical quality solid glass, while the F7 samples were redrawn in rod form from molten glass. The redrawn rod required annealing to improve its optical homogeneity.

The total attenuation of each type of glass as a function of wavelength is shown in fig. 5. Each experimental point is the mean of about four readings. The repeatability of the readings when the sample is replaced, not necessarily in the same position or without rotation, is about  $\pm 10$  dB/km although the change of attenuation with wavelength can be relied on to a greater accuracy than this. The lowest attenuation is exhibited by the two lead flint glasses, types F2 and F7 at about 100 dB/km between 750 to 850 nm wavelength. The F8 lead flint glass shows a minimum attenuation of 190 dB/km at 850 nm, while the SK16 barium crown sample exhibits much higher losses, with a minimum of 360 dB/km at 720 nm.

The variation of attenuation with wavelength for the three lead flint glasses show very similar features. The contribution to attenuation by scattering has been measured [1] and found to be small. At a wavelength of 500 nm, the scattering loss of F7 glass is equivalent to 60 dB/km, reducing at longer wavelengths proportional to  $\lambda^{-4.5}$ . Thus at 800 nm the scattering loss is equivalent to 7 dB/km. Consequently the total attenuation curves presented here closely approximate to the absorption spectra. The features present in the attenuation spectra of the lead flint samples are: high absorption in the 500 nm region; an absorption peak at around 640 nm; low absorption between 700 and 900 nm; absorption increasing towards 1  $\mu\text{m}$ .

These results may be compared with absorption measurements made at a wavelength of 900 nm by the "thermal" technique with F2 and F7 glasses [11] which give values of 300 and 160 dB/km, respectively. The result for F7 is in good agreement with fig. 5b but fig. 5a

gives the much lower value of 115 dB/km for F2. The cause of this discrepancy is not clear but the higher value was obtained with a much earlier sample of F2 glass. It could also be due to different heat treatments of the two samples of glass since these are known to cause quite large changes in the loss coefficients. Results obtained for F2 at 633 nm with a rather simpler apparatus [12] using a helium/neon laser give 560 dB/km as compared with 390 dB/km from fig. 5a. However, full details of the measuring technique have not been published. Other results for F7 give 650 to 800 dB/km at 630 nm and  $100 \pm 30$  dB/km at 850 nm [13] compared with 500 and 115 dB/km, respectively, from fig. 5b. Part of these discrepancies also may be due to different treatments of the materials.

It is thought probable that much of the absorption loss is caused by transition metal ion impurities. Identification of the ion species responsible for the particular features shown in the loss curves is difficult. Earlier investigations [14, 15] have concentrated on the absorption spectra of transition metal ion complexes in silicate and borate glass, and any conclusions reached when comparing the present results with the earlier findings can only be tentative. A common impurity in glass is iron, in either divalent or trivalent state. Absorption by  $\text{Fe}^{2+}$  in silicate glass is at about 1.05  $\mu\text{m}$ , while absorption peaks at 440, 430 and 380  $\mu\text{m}$  are due to  $\text{Fe}^{3+}$  [15] ions. Thus iron as an impurity could account for the increased absorption at either end of the measured spectra.

Several possibilities exist for the ion responsible for the absorption peak at 640 nm [14]. By comparison with the spectra for a soda-lime-silica glass, the  $\text{Cr}^{3+}$  ion with three close absorption peaks at around 650 nm emerges as a strong contender. Absorption by  $\text{Co}^{2+}$  is another possibility. On the other hand,  $\text{Cu}^{2+}$  with an absorption band centred at 800 nm does not appear to contribute noticeably to the glass absorption loss.

#### 5. Conclusions

An improved twin-beam spectrophotometer has been developed to measure bulk glass attenuations of the order of 20 dB/km. The glass is required as the core material of a glass-fibre optical waveguide. Four types of commercially-available optical glass have been measured over the wavelength range 500 to 1000 nm. None of

the glasses measured show a total attenuation low enough for the fibre requirements, the two lead flint glass types, F2 and F7, already in common use as core materials, having the lowest attenuation of about 100 dB/km. Further improvement in glass technology is required to bring about the required decrease in bulk glass attenuation.

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