

Obtaining Accurate Reflectance and Transmittance Measurements for Optical Coatings

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ABSTRACT

The accuracy of the derivation of index of refraction values, n and k , for optical thin film coating materials depends heavily on the accuracy of the spectrophotometric measurement data supporting the derivation. Any reflectance and/or transmittance errors will lead to errors in the resulting n - and k -values, which will in turn cause errors in the associated designs, and the actual coatings will have errors and depart from that which was intended. These derivations are an important part of the production of optical thin films, and need to be fully understood and carefully exercised. Possible causes of measurement errors, calibration procedures, and working standards are discussed. Tests for photometric accuracy and linearity are suggested. Techniques for calibrating spectrophotometers and obtaining accurate reflectance and transmittance data are reviewed.

MEASURING TRANSMITTANCE

There are occasional papers in the journals and sections in texts on the fine points of the measurement of optical coatings such as Arndt et al.[1], but there is seldom a review of "common" practice for a person new to the field. This work attempts to help with that problem of "how to measure" before one embarks on what to do with those measurements.

Different spectrophotometers have different f -number beams in their sample compartments and different minimum sample sizes in their beams. Older instruments may have been $f/10$ while some newer ones may be $f/4$. This beam convergence/divergence may need to be taken into account where coatings are angle sensitive. For example, a 100 GHz fiber optics communication (DWDM) filter will shift a significant portion of its bandwidth (0.4 nm) with a one degree tilt. This means that such a filter cannot be properly measured with an ordinary spectrophotometer because the beam divergence will distort the band shape significantly. A typical beam in a grating instrument is also a millimeter or more wide and about a centimeter high at its smallest point. Again, a DWDM filter of about 1.5 mm square could not be measured properly in such a beam. A new family of instruments has appeared for the DWDM field such as Optical Spectrum Analyzers (OSAs). These are built around the tunable lasers, Gradient Index Lenses (GRINs), and the fiber optics of the field that they serve. They usually measure in Decibels (dB) rather than %T. Although an OSA is very different in detail, in the final result, it is a spectrophotometer.

Infrared instruments are subject to effects of the absorption of the water vapor and CO_2 in the atmosphere. These are most noticeable at about 2.7, 3.2, and 6.2 μm for H_2O , and at 2.7, 4.3, and 15 μm for CO_2 . If the atmosphere within the instrument changes from when the 100%T calibration is run to when the sample is run, there may be spectral artifacts at some of these wavelengths. If this is a problem, the instrument can be purged with dry nitrogen to eliminate these effects. Similar things can be said for regions of the ultraviolet (UV, 200-400 nm). The region from 40 to 200 nm in the UV is referred to as the vacuum ultraviolet (VUV) because the atmosphere absorbs at those short wavelengths and instruments must be evacuated to be effective. It is understood that a dry nitrogen purge might be usable for some cases in the UV.

The typical spectrophotometer is designed to measure transmittance (%T). Ways to measure reflectance are discussed below, but this section deals with transmittance only. Figure 1a illustrates a typical sample beam in the sample compartment of a grating spectrophotometer as seen from above, and Fig. 1b shows the beam as seen from the side. The slit of the monochromator is typically imaged in the

sample compartment; often near the center as shown, but sometimes at one side of the compartment. The reference beam is usually identical and toward the back of the compartment. The slit image is typically much higher than it is wide. It is assumed here that the light travels from left to right in these figures.

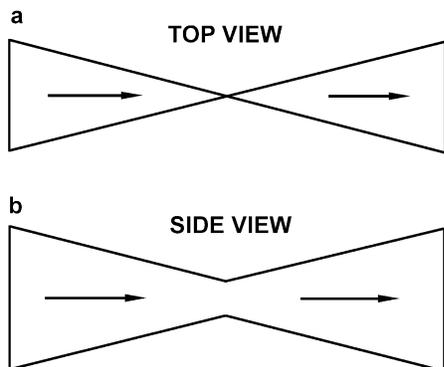


Figure 1. Sample compartment of a typical spectrophotometer as seen from above (a) and the side (b).

A normal practice would be to run a 100%T calibration over the spectral range of interest with no sample in the beams, and then run a 0%T with an opaque sample in the sample beam, while being careful not to interfere with the reference beam in the same sample compartment. The sample of interest is then placed in the sample beam and its transmittance spectrum scanned. Barring errors and instrumental non-linearity, the displayed result is the actual transmittance of the sample.

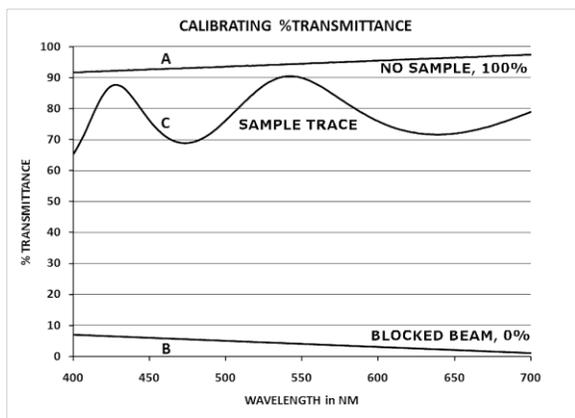


Figure 2. Plot of calibration for transmittance with 100% (A), 0% (B), and then the sample %T (C).

The 100% and 0% scans are not usually perfect, as is illustrated in Fig. 2. The corrected value for the sample %T at each wavelength would be $100 \cdot (C-B)/(A-B)$. This assumes that the instrumental response is linear between curves A and B; but this might need to be tested using polished glass plates of known indices. In the case of dispersive instruments, the assumption may be more nearly valid than in FTIR instruments. Figure 3 shows the calibration of a typical dispersive spectrophotometer with a Schott N-BK7 plane-parallel piece of two-sides polished glass with “known” index versus wavelength. Such a piece might be purchased from Edmund Optics, etc. This shows the accuracy to be about 0.2%T over the spectral range at about 92%. The linearity of a UV-VIS-NIR spectrophotometer might be assessed by using additional “standards” of Fused Silica and Sapphire (which can also be obtained from Edmund Optics, etc.) as lower and higher indices materials than N-BK7.

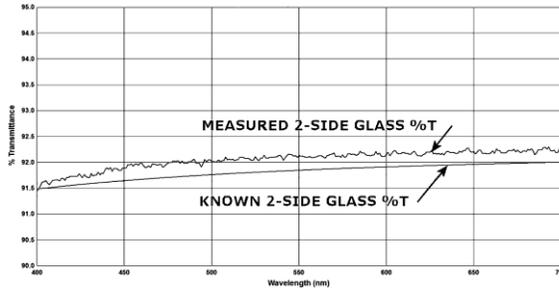


Figure 3. Calibration of a typical spectrophotometer with a sample of Schott N-BK7 of know index and therefore %T.

POTENTIAL MEASUREMENTS PROBLEMS

There are a few problems to be aware of and to avoid. Figure 4 shows what can happen if a sample has some wedge in it. The beam is deflected and some of the transmitted light may be vignetted or not reach the detector. Different instruments have different sensitivities to wedge effects depending on the geometry of the optics between the sample and the detector. It is useful to rotate the sample about the horizontal beam axis to see if that influences the readings; this may be an indication of wedge in the sample. Similar problems can occur with thick samples like plates and prisms.

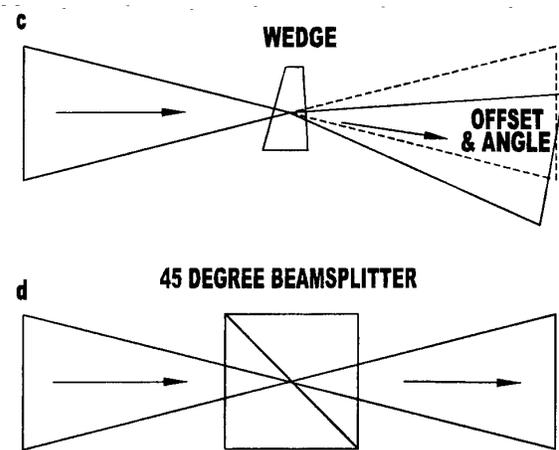


Figure 4. Effect of sample wedge in deflecting the beam.

As seen in Fig. 5e, a tilted thick plate can offset the beam and cause some of the beam to not reach the detector. Figure 5f shows how a thick plate or prism also would cause a focus shift which might be a problem for some instruments. Instruments have been seen which use a small integrating sphere at the detector to minimize these effects.

It may be possible to overcome some of these effects by reducing the beam diameter (f /number) on the left (input) side as it enters the compartment. This narrower beam may allow the transmitted beam to have some offset without causing vignetting or further loss of signal. The 100%T, of course, needs to be recalibrated with such a reducing aperture in place, and some signal-to-noise ratio (S/N) is lost. If a sample is smaller than the normal sample beam at its smallest cross section, one can insert an aperture slightly smaller than the sample to be measured at the sample position and run a 100%T scan with no sample in place. The sample can then be added over the aperture and its transmittance spectrum run. This will reduce the S/N ratio in proportion to the area reduction of the sample beam; but this can be overcome by slower/longer data gathering.

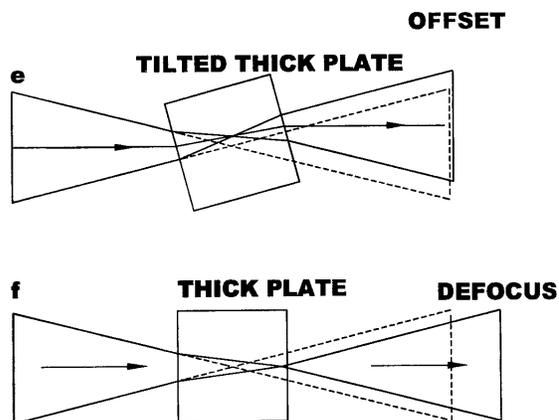


Figure 5. Thick and tilted plates can offset and defocus a beam to a point that some of the beam does not reach the detector.

Coatings on beamsplitters that are on/in prisms need to be measured. A beamsplitter coated on a 45° prism can be measured by putting it together with an uncoated prism using uncured cement or an index matching fluid. One such fluid is "oil of wintergreen" (index ~ 1.536) which can be purchased at a pharmacy; another might be glycerin (index ~ 1.473). The transmittance of the assembly can then be measured as in Fig. 4d. The above-mentioned effects of offset and defocus need to be considered. Another problem is polarization. All spectrophotometers have some complex polarization effects in the beams. Coatings at anything other than near normal incidence will have polarization effects. One practice is to measure the assembly such as that in Fig. 4d once and then rotate it 90° about the horizontal beam axis and measure it again. The average of the two scans should represent the effect of random polarization on the coating.

It is also possible to insert linear polarizers in *both* sample and reference beams (typically on the left side where the beams enter the compartment) to only pass the desired polarization through the sample. There are also depolarizers which are crystalline in nature and change the state of polarization rapidly across the aperture of the beam so as to homogenize the polarization of the beams.

MEASURING REFLECTANCE

Since most spectrophotometers have been made for transmittance measurements, it is necessary to use attachments in the sample beam to accommodate the desired reflectance measurements. In some cases, the instrument manufacturers have attachments available; and in other cases there are a wide variety of attachments available from companies like Harrick[2] and Labsphere[3].

The simplest and first essential reflection attachment is illustrated in Fig. 6. The normally horizontal transmittance beam is reflected upward by a mirror (M1) to the sample, the beam is then reflected from the sample, and then is reflected by another mirror (M2) again into the optical path. The angle of incidence on the sample mirror should be as small as practical to avoid the changes in reflectance due to angle; 8° to 12° is typical and satisfactory. Error studies have shown this to be the best type of fixture for measuring antireflection and other low reflecting coatings.

The mirrors M1 and M2 should actually be of a conic section shape to focus what would have been the slit image at the center of the sample compartment onto the reflectance sample and then back into the normal beam. Toroidal approximations of these mirrors are usually adequate.

There are several alternatives to calibrate this attachment. First, a 0%R line can be run with no sample in position. Then, a thin glass slide of known index such as N-BK7 can be placed in the sample position and the spectrum measured. If the glass is thin and of index about 1.52 with no absorption in the region of interest, then the reflection should be about 8%. All subsequent samples can be compared with

this calibration of the 8% reflectance. The glass slide can be verified by measuring its %T (which should be about 92%), if the absorption can be neglected.

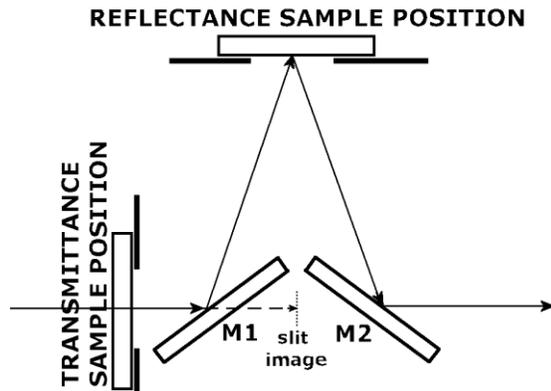


Figure 6. A near-normal reflectance attachment.

Like the procedure above for %T, the corrected value for the sample %R at each wavelength would be $D*(C-B)/(A-B)$, where the 100% is replaced by the known %R (D) of the reference glass as in Fig. 7.

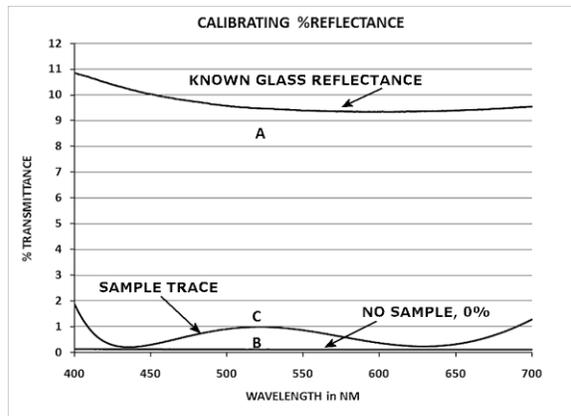


Figure 7. Plot of calibration for reflectance with a known piece of glass (A), 0% (B), and then the sample %R (C).

Another alternative is to measure only the one-side reflectance of a known glass. This can be done several ways. As illustrated in Fig. 8, one approach is to coarse grind the back side of a sample so that the light reflected from the back is widely scattered, and hopefully very little of the light from the rear surface gets back into the beam that reaches the detector. The suitability of this approach will be somewhat dependent on the f/number of the instrument. Some people further blacken the ground surface with a black marking pen to reduce the reflection from the ground side. It is recommended however to paint the **unground** back side of the flat witness with what amounts to an index matching fluid filled with carbon black (black paint). In this case, all the light reaching the rear surface enters the vehicle (which has an index near that of the glass, thus producing very little reflection at the interface) of the black lacquer (such as black KrylonTM spray paint) and is absorbed by the carbon black. Thus only the front surface reflection is seen and measured. It can be useful to use the last case illustrated in Fig. 8, where a single witness piece is painted black on one area for one-side %R measurements and the rest is left clear for %T and two-side %R measurements.

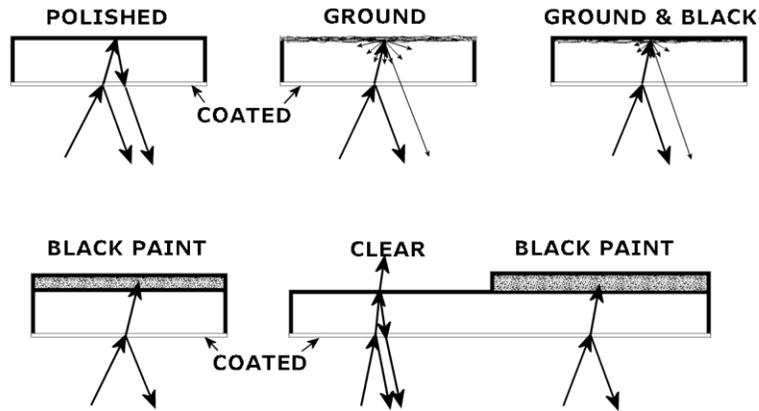


Figure 8. Several ways to treat %R (and %T in the last case) witness samples for accurate measurement.

For calibration of one-side reflectance, there are a few additional geometries that can be used. Some organizations use a two-side polished witness glass of known index with a significant wedge between the bottom and top surfaces. The back surface should then be at such an angle that none of the light reflected from it gets back into the sample beam that reaches the detector. This wedge would then reflect about 4% depending on the exact index of the glass. A variant of this wedge technique is to have a coating sample on a thin plane parallel glass witness piece which is then oiled to such a wedge with index matching fluid such as oil of wintergreen or glycerin to create the same effect as the wedge alone.

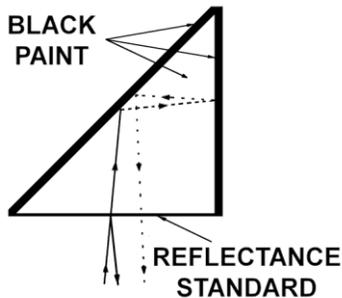


Figure 9. A light trap possibility for a one-side calibration standard or an oil-on second-side reflection remover.

A recommended example of eliminating the effects of the second side reflectance of a Reflectance Standard is shown in Fig. 9. Here a prism is used which has had all sides except the reference surface painted with black paint to capture the transmitted light through the first surface. This double light trap should be very effective as a replacement for the oiled wedge mentioned above. Prisms of N-BK7 and Fused Silica (without any coating) can be obtained from Edmund Optics, etc., and then painted with Black Krylon spray paint to be reflectance “standards.” However, simply painting the back of plane-parallel witnesses of N-BK7 might be adequate for this type of work in most facilities.

CHECKING LINEARITY OF %R AND %T MEASUREMENTS

It is appropriate to check the linearity of the %R measurements also, since erroneous measurements have caused extensive problems in the AR coating industry. Figure 10 illustrates the procedure where a known glass such as N-BK7 is used in both its two-side and one-side reflectances. The two-side

reflection is used to calibrate as described in Fig. 7; then the one-side can be used to check linearity against its known value. In the case of Fig. 10, we see an error of about 0.4%R.

In Fig. 11, further checking is done using Fused Silica in two-side and one-side %R after the N-BK7 calibration. This shows about 0.3%R error for one-side and a “crossing” from plus to minus errors on the two-side reflectance. Such tests indicate the confidence level which can be expected from any given measurements.

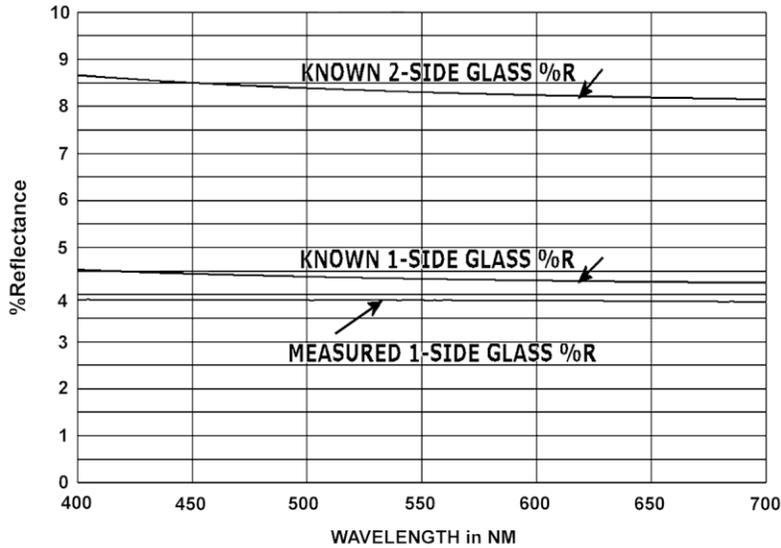


Fig. 10. Linearity check using a “reference standard” like N-BK7 glass for two-side calibration and the one-side reflection for linearity check.

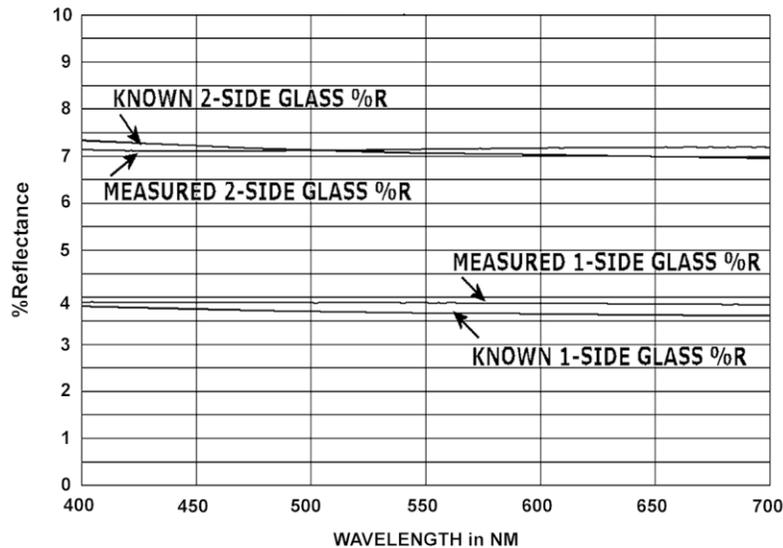


Fig. 11. Linearity check using a “reference standard” like N-BK7 glass for two-side calibration and then both two-side and one-side reflection of a sample like Fused Silica for linearity check.

Table 2.1 Best available data for %R and %T to use in spectrophotometer calibrations.

WL in nm	N-BK7		N-BK7	Sapphire		Sapphire		Silica		Silica
	Two-Side	Two-Side	One-Side	Two-Side	Two-Side	One-Side	Two-Side	Two-Side	Two-Side	One-Side
	%R	%T	%R	%R	%T	%R	%R	%T	%R	%R
400	8.43	91.57	4.40	14.77	85.23	7.97	6.99	93.01	3.62	
410	8.40	91.60	4.38	14.71	85.29	7.94	6.97	93.03	3.61	
420	8.37	91.63	4.37	14.67	85.33	7.92	6.95	93.05	3.60	
430	8.34	91.66	4.35	14.64	85.36	7.90	6.92	93.08	3.59	
440	8.32	91.68	4.34	14.61	85.39	7.88	6.90	93.10	3.58	
450	8.30	91.70	4.33	14.58	85.42	7.87	6.89	93.11	3.57	
460	8.27	91.73	4.32	14.57	85.43	7.86	6.87	93.13	3.56	
470	8.26	91.74	4.31	14.54	85.46	7.84	6.85	93.15	3.55	
480	8.24	91.76	4.30	14.51	85.49	7.82	6.84	93.16	3.54	
490	8.22	91.78	4.29	14.49	85.51	7.81	6.82	93.18	3.53	
500	8.20	91.80	4.28	14.46	85.54	7.79	6.81	93.19	3.53	
510	8.19	91.81	4.27	14.44	85.56	7.78	6.80	93.20	3.52	
520	8.17	91.83	4.26	14.41	85.59	7.77	6.79	93.21	3.51	
530	8.16	91.84	4.25	14.39	85.61	7.75	6.78	93.22	3.51	
540	8.15	91.85	4.25	14.37	85.63	7.74	6.77	93.23	3.50	
550	8.13	91.87	4.24	14.36	85.64	7.74	6.76	93.24	3.50	
560	8.12	91.88	4.23	14.35	85.65	7.73	6.75	93.25	3.49	
570	8.11	91.89	4.23	14.33	85.67	7.72	6.74	93.26	3.49	
580	8.10	91.90	4.22	14.32	85.68	7.71	6.73	93.27	3.48	
590	8.09	91.91	4.22	14.30	85.70	7.70	6.72	93.28	3.48	
600	8.08	91.92	4.21	14.29	85.71	7.69	6.71	93.29	3.47	
610	8.07	91.93	4.21	14.28	85.72	7.69	6.71	93.29	3.47	
620	8.06	91.94	4.20	14.27	85.73	7.68	6.70	93.30	3.47	
630	8.05	91.95	4.20	14.26	85.74	7.67	6.69	93.31	3.46	
640	8.05	91.95	4.19	14.24	85.76	7.67	6.68	93.32	3.46	
650	8.04	91.96	4.19	14.23	85.77	7.66	6.68	93.32	3.45	
660	8.03	91.97	4.18	14.22	85.78	7.65	6.67	93.33	3.45	
670	8.02	91.98	4.18	14.21	85.79	7.65	6.67	93.33	3.45	
680	8.02	91.98	4.18	14.20	85.80	7.64	6.66	93.34	3.44	
690	8.01	91.99	4.17	14.19	85.81	7.64	6.65	93.35	3.44	
700	8.00	92.00	4.17	14.18	85.82	7.63	6.65	93.35	3.44	
710	8.00	92.00	4.17	14.17	85.83	7.63	6.64	93.36	3.44	
720	7.99	92.01	4.16	14.16	85.84	7.62	6.64	93.36	3.43	
730	7.99	92.01	4.16	14.15	85.85	7.61	6.64	93.36	3.43	
740	7.99	92.01	4.16	14.14	85.86	7.61	6.63	93.37	3.43	
750	7.98	92.02	4.16	14.13	85.87	7.60	6.63	93.37	3.43	
760	7.98	92.02	4.15	14.13	85.87	7.60	6.62	93.38	3.42	
770	7.97	92.03	4.15	14.12	85.88	7.60	6.62	93.38	3.42	
780	7.97	92.03	4.15	14.12	85.88	7.59	6.61	93.39	3.42	
790	7.96	92.04	4.15	14.11	85.89	7.59	6.61	93.39	3.42	
800	7.96	92.04	4.15	14.10	85.90	7.58	6.61	93.39	3.42	

Table 2.1 gives the calculated values for N-BK7, Sapphire (Al_2O_3), and Fused Silica of two-side %T, two-side %R, and one-side %R. These may be used as calibration “Standards” with some caveats. The N-BK7 data is expected to be the most reliable because Schott Glass⁴ quotes the variation of index to be less than ± 0.0005 . This would cause a variation in the %R of only $\pm 0.006\%$, which would be in the noise of most spectrophotometers. The values for Fused Silica (SiO_2) and Sapphire (Al_2O_3) are taken from the Palik^{5,6} books. This data might not be as close a representation of the actual material which might be obtained from Edmund Optics (or others) as is the N-BK7 data supplied by Schott⁴.

Figures 12 and 13 plot the values of Table 2.1 to show the ranges over which linearity and calibrations can be checked with these materials. Percent transmittance can be checked over about 85% to 94%, %R reflectance from about 3% to 15%.

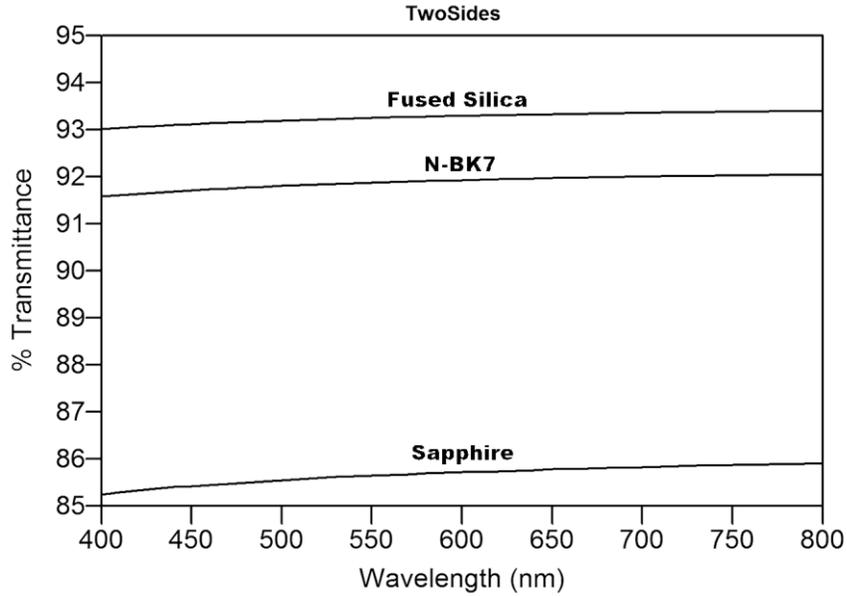


Fig. 12. Linearity check possibilities using “reference standard” from Table 2.1 for two-side %T.

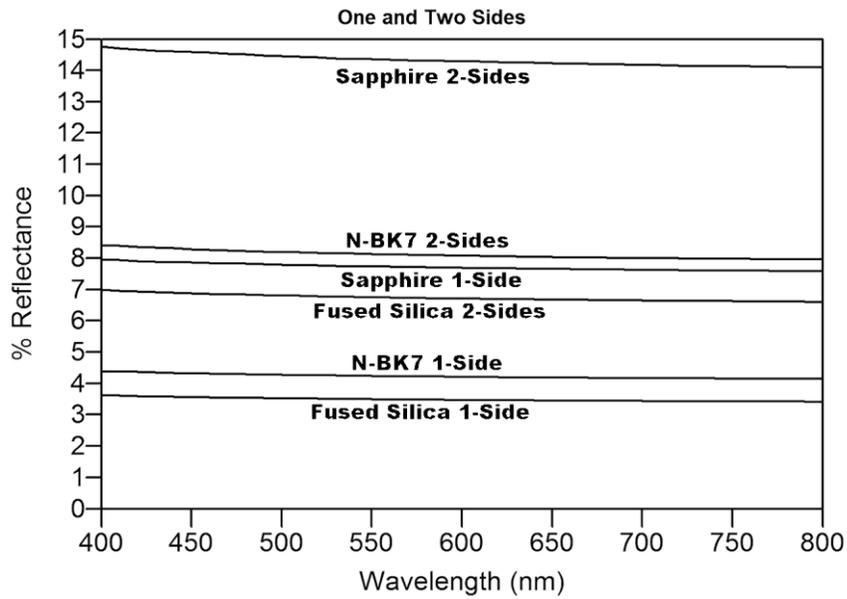


Fig. 13. Linearity check possibilities using a “reference standard” from Table 2.1 for two-side %R and one-side %R.

CONCLUSIONS

The accuracy of the derivation of n- and k-values for a material depends heavily on the accuracy of the spectrophotometric measurements discussed above. Any %R and/or %T errors will lead to errors in the resulting n- and k-values, which will in turn cause errors in designs, and the actual coatings will have errors and depart from that performance which was desired. This is an important part of the production of optical thin films, and it should be carefully understood.

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