Metrologia 27, 33–38 (1990)

metrologia

International Intercomparison of Regular Transmittance Scales

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Received: July 13, 1989 and in revised form August 18, 1989

Abstract

An intercomparison of the regular spectral transmittance scales of NIST, Gaithersburg, MD. (USA); PTB, Braunschweig (FRG); NPL, Teddington, Middlesex (UK); and OMH, Budapest (H) was accomplished using three sets of neutral glass filters with transmittances ranging from approximately 0.92 to 0.001. The difference between the results from the reference spectrophotometers of the laboratories was generally smaller than the total uncertainty of the interchange. The relative total uncertainty ranges from 0.05% to 0.75% for transmittances from 0.92 to 0.001. The sample-induced error was large – contributing 40% or more of the total except in a few cases.

1. Introduction

Commercial instrumentation for measuring regular spectral transmittance has become more precise. Therefore it has become necessary for national standardizing laboratories to build reference instruments that are well characterized, accurate and precise. The uncertainties in modern regular transmittance measurements using reference instruments are generally better known than the characteristics of the specimens used to transfer measurements to other laboratories. This paper illustrates how well four national standardizing laboratories agree using high-quality samples.

2. Organization of Intercomparison

E. Sutter (PTB), G. H. C. Freeman (NPL), and K. L. Eckerle (NIST) agreed to intercompare transmittance measurements using two sets of Measurement Assurance Program (MAP) neutral filters from the NIST [1]. The measurement sequence would be NIST-PTB-NIST and NIST-NPL-NIST. Since the NIST would measure the filters before and after the other two laboratories, an estimate could be made of the possible change in the filters. The NIST would also set bounds on the transmittance variation due to non-uniformity of the filters. The measured non-uniformity of the NIST filters was generally less than 0.05% except for the more dense filters where the non-uniformity was as large as 0.35%.

About the same time, G. Andor (OMH) expressed interest in using some neutral filters, which are issued as Standard Reference Materials (SRM) [2] from OMH, for an intercomparison. The same sequence, NIST-OMH-NIST, was used for testing and estimates were also made for variation due to non-uniformity and possible changes. The measured non-uniformity for the OMH filters 1 and 2 was less than 0.1%, and for filters 3 and 4 was less than 0.25%. Since a smaller spot size was used for the OMH filters, a slightly larger non-uniformity than that measured for the NIST filters was expected.

A total of eighteen different filters was used for the intercomparison, 14 from NIST and 4 from OMH. A total time of approximately 18 months elapsed for all three interchanges to be completed. Approximately, six months elapsed for the PTB interchange, five months for the NPL interchange and eight months for the OMH interchange.

3. Instrumentation

The reference spectrophotometers [3–7] used in this intercomparison are well characterized with estimates of systematic and statistical uncertainties. Their important features are given in Table 1. A number of features are common to the measurement systems: they all use off-axis reflecting optics to eliminate interreflections between the optics and the sample, and collimate the radiation incident on the sample to minimize obliquity effects. System linearity is measured and corrections made. Systematic uncertainties are either negligible or are eliminated by

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Table 1. Instrument characteristics

Lab	Band-pass (nm)	Diameter of the illuminating area (mm)	Temp (°C)	Lamp	Collimating optics	Collimation (rad)	Monochromator	High order rejection	Detection system	Amplification
NIST	1.5	15 (for NIST filters) 8 (for OMH filters)	23 ± 0.5	tungsten ribbon	off-axis mirrors	0.0025	grating	prism pre-disperser	averaging sphere and photomultiplier	dc
PTB	1.5	15	23 ± 0.5	quartz-halogen	off-axis mirrors	0.0018	double-grating	filter	ground glass and photomultiplier	ac
NPL	0.5	15	23 ± 0.1	tungsten ribbon	off-axis mirrors	0.004	double-grating	filter	averaging sphere and photomultiplier	dc
OMH	1.5	8	23 ± 0.5	tungsten ribbon	off-axis mirrors	0.005	double-grating	filter glass	averaging sphere and photomultiplier	dc

Table 2. Results of interchange with PTB

λ (nm)	NIST τ (before)	NIST τ (after)	NIST τ (ave)	PTB τ	U_{NIST}	U_{PTB}	U_{SAMPLE}	U_{TOTAL}	$\Delta\tau$
Filter 2-1									
539.5	0.9160	0.9160	0.9160	0.9164	0.00068	0.00031	0.00012	0.0008	0.0004
542.5	0.9161	0.9160	0.9161	0.9162	0.00026	0.00066	0.00013	0.0007	0.0001
548.5	0.9162	0.9161	0.9162	0.9164	0.00021	0.00063	0.00013	0.0007	0.0002
554.5	0.9162	0.9161	0.9162	0.9165	0.00018	0.00051	0.00013	0.0006	0.0003
557.5	0.9163	0.9163	0.9163	0.9167	0.00022	0.00031	0.00012	0.0004	0.0004
Filter 2-2									
539.5	0.6922	0.6922	0.6922	0.6924	0.00035	0.00028	0.00017	0.0005	0.0002
542.5	0.6926	0.6924	0.6925	0.6927	0.00017	0.00031	0.00018	0.0004	0.0002
548.5	0.6928	0.6926	0.6927	0.6928	0.00017	0.00034	0.00019	0.0004	0.0001
554.5	0.6924	0.6922	0.6923	0.6923	0.00022	0.00014	0.00020	0.0003	0.0000
557.5	0.6919	0.6919	0.6919	0.6921	0.00012	0.00019	0.00017	0.0003	0.0002
Filter 2-3									
539.5	0.5160	0.5182	0.5171	0.5173	0.00023	0.00159	0.00106	0.0019	0.0002
542.5	0.5166	0.5186	0.5176	0.5178	0.00014	0.00075	0.00100	0.0013	0.0002
548.5	0.5171	0.5189	0.5180	0.5182	0.00015	0.00063	0.00095	0.0012	0.0002
554.5	0.5165	0.5183	0.5174	0.5179	0.00014	0.00037	0.00093	0.0010	0.0005
557.5	0.5158	0.5177	0.5168	0.5172	0.00013	0.00156	0.00098	0.0019	0.0004
Filter 2-4									
539.5	0.2360	0.2359	0.2360	0.2364	0.00018	0.00020	0.00024	0.0004	0.0004
542.5	0.2367	0.2366	0.2367	0.2370	0.00011	0.00034	0.00024	0.0004	0.0003
548.5	0.2374	0.2373	0.2374	0.2378	0.00012	0.00023	0.00024	0.0004	0.0004
554.5	0.2369	0.2368	0.2369	0.2374	0.00012	0.00009	0.00025	0.0003	0.0005
557.5	0.2362	0.2361	0.2362	0.2366	0.00011	0.00011	0.00024	0.0003	0.0004
Filter 2-5									
539.5	0.09595	0.09594	0.09595	0.09611	0.00013	0.00006	0.00003	0.00014	0.00016
542.5	0.09644	0.09639	0.09642	0.09658	0.00012	0.00006	0.00004	0.00014	0.00016
548.5	0.09689	0.09689	0.09689	0.09705	0.00012	0.00006	0.00003	0.00013	0.00016
554.5	0.09660	0.09656	0.09658	0.09675	0.00012	0.00007	0.00004	0.00014	0.00017
557.5	0.09607	0.09608	0.09608	0.09626	0.00011	0.00008	0.00003	0.00014	0.00018
Filter 2-6									
539.5	0.009157	0.009157	0.009157	0.009173	0.000024	0.000023	0.000003	0.000034	0.000016
542.5	0.009207	0.009202	0.009205	0.009224	0.000027	0.000035	0.000004	0.000044	0.000019
548.5	0.009244	0.009245	0.009245	0.009264	0.000022	0.000032	0.000003	0.000039	0.000019
554.5	0.009221	0.009201	0.009211	0.009223	0.000027	0.000031	0.000010	0.000042	0.000012
557.5	0.009148	0.009143	0.009146	0.009172	0.000024	0.000026	0.000004	0.000036	0.000026
Filter 2-7									
539.5	0.0009319	0.0009336	0.0009328	0.0009334	0.0000110	0.0000027	0.0000012	0.0000114	0.0000006
542.5	0.0009396	0.0009378	0.0009387	0.0009418	0.0000064	0.0000033	0.0000013	0.0000074	0.0000031
548.5	0.0009443	0.0009436	0.0009440	0.0009478	0.0000047	0.0000031	0.0000010	0.0000057	0.0000038
554.5	0.0009362	0.0009372	0.0009367	0.0009415	0.0000048	0.0000028	0.0000010	0.0000056	0.0000048
557.5	0.0009280	0.0009279	0.0009280	0.0009338	0.0000043	0.0000024	0.0000009	0.0000050	0.0000058

Table 3. Results of interchange with NPL

λ (nm)	NIST τ (before)	NIST τ (after)	NIST τ (ave)	NPL τ	U_{NIST}	U_{NPL}	U_{SAMPLE}	U_{TOTAL}	$\Delta\tau$
Filter 3-1									
539.5	0.9151	0.9157	0.9154	0.9163	0.00015	0.00017	0.00045	0.0005	0.0009
542.5	0.9152	0.9158	0.9155	0.9163	0.00025	0.00013	0.00044	0.0005	0.0008
548.5	0.9152	0.9158	0.9155	0.9164	0.00026	0.00013	0.00046	0.0006	0.0009
554.5	0.9154	0.9160	0.9157	0.9165	0.00027	0.00010	0.00042	0.0005	0.0008
557.5	0.9154	0.9161	0.9158	0.9166	0.00015	0.00017	0.00046	0.0005	0.0008
Filter 3-2									
539.5	0.6908	0.6913	0.6911	0.6913	0.00015	0.00017	0.00035	0.0004	0.0002
542.5	0.6912	0.6917	0.6915	0.6916	0.00019	0.00013	0.00037	0.0004	0.0001
548.5	0.6914	0.6920	0.6917	0.6918	0.00020	0.00013	0.00037	0.0004	0.0001
554.5	0.6911	0.6916	0.6914	0.6914	0.00020	0.00010	0.00034	0.0004	0.0000
557.5	0.6906	0.6910	0.6908	0.6910	0.00018	0.00017	0.00031	0.0004	0.0002
Filter 3-3									
539.5	0.5162	0.5179	0.5171	0.5172	0.00020	0.00020	0.00093	0.0010	0.0001
542.5	0.5167	0.5185	0.5176	0.5178	0.00015	0.00017	0.00093	0.0010	0.0002
548.5	0.5171	0.5187	0.5179	0.5181	0.00015	0.00017	0.00088	0.0009	0.0002
554.5	0.5166	0.5183	0.5175	0.5175	0.00021	0.00017	0.00089	0.0009	0.0000
557.5	0.5159	0.5176	0.5168	0.5168	0.00013	0.00020	0.00090	0.0009	0.0000
Filter 3-4									
539.5	0.2360	0.2361	0.2361	0.2358	0.00017	0.00020	0.00020	0.0003	−0.0003
542.5	0.2367	0.2369	0.2368	0.2366	0.00012	0.00017	0.00021	0.0003	−0.0002
548.5	0.2374	0.2375	0.2375	0.2372	0.00012	0.00013	0.00021	0.0003	−0.0003
554.5	0.2370	0.2371	0.2371	0.2367	0.00014	0.00006	0.00020	0.0003	−0.0004
557.5	0.2362	0.2363	0.2363	0.2360	0.00013	0.00006	0.00020	0.0003	−0.0003
Filter 3-5									
539.5	0.09571	0.09576	0.09573	0.09559	0.00012	0.00001	0.00004	0.00013	−0.00014
542.5	0.09620	0.09627	0.09624	0.09609	0.00011	0.00004	0.00005	0.00013	−0.00015
548.5	0.09669	0.09673	0.09671	0.09652	0.00011	0.00001	0.00004	0.00012	−0.00019
554.5	0.09635	0.09643	0.09639	0.09618	0.00011	0.00002	0.00005	0.00012	−0.00021
557.5	0.09587	0.09595	0.09591	0.09569	0.00010	0.00004	0.00005	0.00012	−0.00022
Filter 3-6									
539.5	0.009437	0.009456	0.009446	0.009445	0.000023	0.000013	0.000029	0.000039	−0.000001
542.5	0.009498	0.009491	0.009495	0.009496	0.000022	0.000018	0.000027	0.000039	0.000001
548.5	0.009533	0.009530	0.009532	0.009530	0.000023	0.000008	0.000027	0.000036	−0.000002
554.5	0.009496	0.009499	0.009498	0.009482	0.000021	0.000001	0.000027	0.000034	−0.000016
557.5	0.009433	0.009442	0.009437	0.009428	0.000024	0.000013	0.000027	0.000039	−0.000009
Filter 3-7									
539.5	0.0009354	0.0009411	0.0009383	0.0009332	0.0000050	0.0000013	0.0000043	0.0000067	−0.0000051
542.5	0.0009443	0.0009448	0.0009446	0.0009402	0.0000055	0.0000030	0.0000032	0.0000071	−0.0000044
548.5	0.0009479	0.0009489	0.0009484	0.0009474	0.0000043	0.0000044	0.0000032	0.0000069	−0.0000010
554.5	0.0009446	0.0009487	0.0009467	0.0009402	0.0000077	0.0000030	0.0000038	0.0000091	−0.0000065
557.5	0.0009327	0.0009362	0.0009345	0.0009317	0.0000042	0.0000030	0.0000036	0.0000063	−0.0000028

numerical corrections. The common sources of systematic uncertainty are: beam displacement and defocusing effects, interreflections, obliquity, polarization, linearity, wavelength bias and stray light. Estimates of other sources of systematic uncertainty have been made for each instrument. A treatment of the error analysis using current statistical practice may be found in Ref. [4].

The instruments are automated so that it is straightforward to repeat measurements and evaluate statistical uncertainties. The uncertainties, U_{LAB} (LAB = NIST, NPL, PTB or OMH), in Tables 2–4 are the combination in quadrature of an upper bound for the systematic uncertainty, ΔT_S, and the statistical uncertainty expressed as three times the standard deviation of the mean, $3\Delta T_R$:

$$U_{LAB} = [(\Delta T_S)^2 + (3\Delta T_R)^2]^{1/2} \qquad (1)$$

where:

$$\Delta T_R = \left[\sum_{i=1}^{n} (\tau_i - \bar{\tau})^2 / n(n-1)\right]^{1/2}.$$

Here τ_i is the measured transmittance, $\bar{\tau}$ the mean of the set of measurements and n is the number of measurements in the set. The NIST calculated U_{LAB} after putting ΔT_S from the four participants on a 3σ basis. Sample induced errors were evaluated for the specimens to be measured.

Table 4. Results of interchange with OMH

λ (nm)	NIST τ (before)	NIST τ (after)	NIST τ (ave)	OMH τ	U_{NIST}	U_{OMH}	U_{SAMPLE}	U_{TOTAL}	$\Delta\tau$
Filter 1									
530	0.8646	0.8645	0.8646	0.8645	0.00028	0.00105	0.00037	0.0012	−0.0001
540	0.8672	0.8666	0.8669	0.8669	0.00058	0.00031	0.00079	0.0010	0.0000
550	0.8621	0.8717	0.8719	0.8719	0.00034	0.00035	0.00058	0.0008	0.0000
560	0.8724	0.8720	0.8722	0.8725	0.00029	0.00071	0.00045	0.0009	0.0003
570	0.8694	0.8690	0.8692	0.8693	0.00061	0.00059	0.00059	0.0010	0.0001
Filter 2									
530	0.2508	0.2510	0.2509	0.2514	0.00016	0.00025	0.00017	0.0003	0.0005
540	0.2555	0.2555	0.2555	0.2560	0.00019	0.00026	0.00021	0.0004	0.0005
550	0.2564	0.2566	0.2565	0.2570	0.00030	0.00031	0.00018	0.0005	0.0005
560	0.2532	0.2534	0.2533	0.2537	0.00012	0.00020	0.00028	0.0004	0.0004
570	0.2471	0.2473	0.2472	0.2476	0.00012	0.00027	0.00021	0.0004	0.0004
Filter 3									
530	0.1363	0.1365	0.1364	0.1366	0.00011	0.00026	0.00026	0.0004	0.0002
540	0.1398	0.1402	0.1400	0.1402	0.00013	0.00017	0.00036	0.0004	0.0002
550	0.1408	0.1410	0.1409	0.1411	0.00042	0.00019	0.00034	0.0006	0.0002
560	0.1382	0.1384	0.1383	0.1385	0.00011	0.00019	0.00033	0.0004	0.0002
570	0.1333	0.1335	0.1334	0.1335	0.00019	0.00016	0.00031	0.0004	0.0001
Filter 4									
530	0.0825	0.0825	0.0825	0.0827	0.00013	0.00020	0.00008	0.0003	0.0002
540	0.0842	0.0842	0.0842	0.0844	0.00010	0.00023	0.00012	0.0003	0.0002
550	0.0845	0.0845	0.0845	0.0846	0.00024	0.00021	0.00012	0.0003	0.0001
560	0.0830	0.0832	0.0831	0.0833	0.00019	0.00015	0.00019	0.0003	0.0002
570	0.0801	0.0803	0.0802	0.0804	0.00012	0.00020	0.00017	0.0003	0.0002

4. Results

Tables 2–4 show the results of the intercomparisons. The wavelengths, λ, chosen for the MAP filters were those that the NIST normally uses for the MAP service. The wavelengths chosen for the OMH filters were in the same wavelength region, but OMH was interested in different wavelengths. Four significant figures are given in each case except for OMH filter 4 where only three significant figures were supplied by OHM. An estimate of the uncertainty, U_{sample}, is given for each specific filter. It is an addition in quadrature of the observed change in the filter and the non-uniformity of the filter (3σ estimates). Each individual filter change was estimated from the first and last NIST measurements. It was estimated that each laboratory could position the filters to 1 mm accuracy using their normal procedure. For the NIST instrument, some of the errors that can contribute to this 1 mm uncertainty are:

(a) The alignment laser and the tungsten lamp used for the measurement do not usually follow exactly the same path. It is estimated they can separate by an angle of 0.004 rad. Since the limiting aperture and the filter are separated by 50 mm, this can cause an uncertainty of 0.2 mm.

(b) The alignment laser spot is over 2 mm in diameter in the sample compartment causing another 0.2 mm uncertainty.

(c) The position of the filter in its holder can easily be uncertain by 0.3 mm.

(d) The device for measuring uniformity itself has a position uncertainty of 0.2 mm.

If all the above act in the same direction a conservative 3σ estimate is 0.9 mm, rounded to 1.0 mm. Therefore, non-uniformity was estimated for a 2 mm displacement from the center position at 548.5 nm for the MAP filters and 550 nm for the OMH filters. This is a conservative 3σ estimate that assumes the NIST had a placement error of 1 mm in one direction, and the particular laboratory had a 1 mm placement error in the opposite direction. The 2 mm displacement of the filters was made in four directions – up, down, left and right – with all movement perpendicular to the light beam. The displacement that gave the largest change in transmittance from that in the center position was chosen as the displacement which gave an upper bound for transmittance to the non-uniformity estimate. Separate estimates were made for the 8 mm and 15 mm spot size. The spot sizes of the illuminating light are given in Table 1. The temperature effect for neutral glasses of this type has been measured by other researchers for filters with transmittances of 0.1, 0.2, and 0.3 [8]. According to them, an uncertainty of 0.5 °C in temperature causes an average change of transmittance of approximately 0.0003 at 546.1 nm. Because of the spectral neutrality of these filters, a similar relative uncertainty in transmittance is expected for filters with different transmittances. In any case, for this intercomparison, changes due to temperature variability will not change U_{LAB} or U_{TOTAL} significantly. U_{TOTAL} is the square root of the quad-

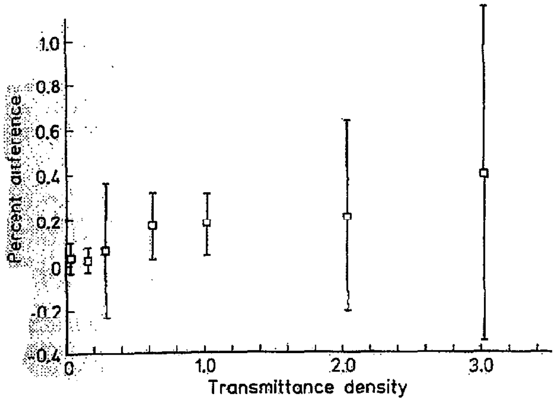

Fig. 1. Percent difference between PTB and NIST versus transmittance (optical) density

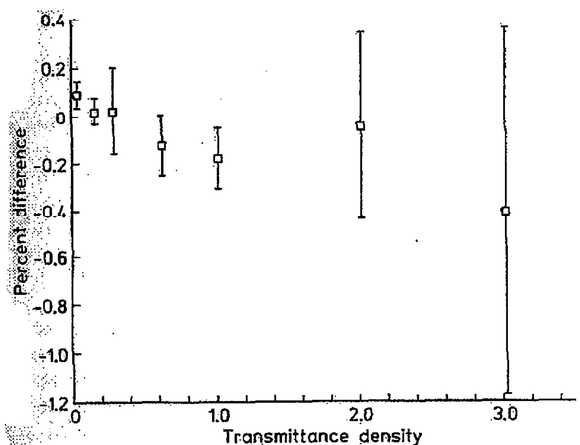

Fig. 2. Percent difference between NPL and NIST versus transmittance (optical) density

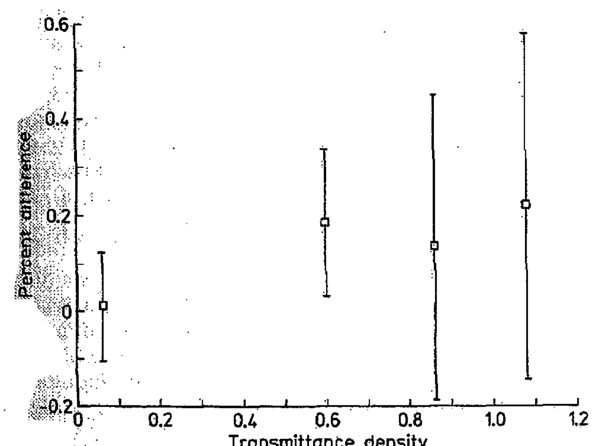

Fig. 3. Percent difference between OMH and NIST versus transmittance (optical) density

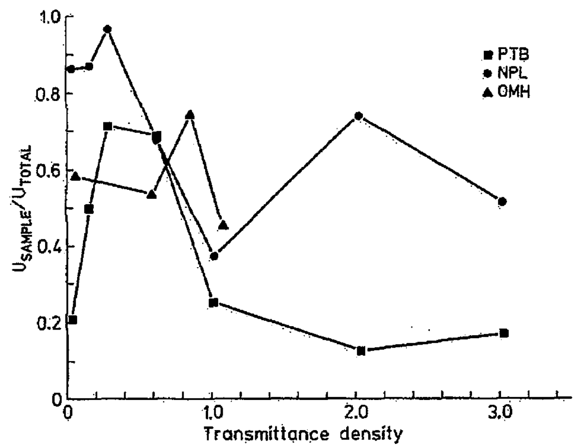

Fig. 4. U_{SAMPLE}/U_{TOTAL} for PTB, NPL, OMH versus transmittance (optical) density

rature sum of U_{NIST}, U_{LAB}, U_{SAMPLE}

$$U_{TOTAL} = [U_{NIST}^2 + U_{LAB}^2 + U_{SAMPLE}^2]^{1/2} \quad (2)$$

The difference, $\Delta\tau$, is the difference of transmittance measurements between the particular laboratory and the NIST, $\Delta\tau = \tau_{LAB} - \tau_{NIST}$ where τ_{NIST} is the average of the NIST measurements before and after τ_{LAB}. An analysis of variance was made for each set of differences according to wavelength and filter. These analyses showed a dependence of the transmittance difference on the magnitude of the measurement, but not on wavelength. The averages over all wavelengths of the percent difference plotted versus transmittance are shown for the three laboratories in Figs. 1–3. It may also be seen that in almost all cases this difference is smaller than the total uncertainty, U_{TOTAL}. To illustrate this, U_{TOTAL} as a percentage of transmittance is shown by the error bars in Figs. 1–3. The transmittance differences between laboratories are larger than expected for filters 2-4, 2-5, 3-1, 3-5 and 2. Filter 3-5 had acquired a small smudge by the end of the intercomparison. The differences for the remaining filters remain unexplained. Thus, the differences between the laboratories, although real, are so small as to be of no practical significance. The major role of U_{SAMPLE} is seen in Fig. 4 where the average over wavelength of U_{SAMPLE}/U_{TOTAL} is plotted versus transmittance for the PTB, NPL, and OMH.

5. Conclusions

This intercomparison shows the state-of-the-art for transmittance measurements since four reference instruments built and maintained by national standards laboratories were used. Since the disagreement between the four laboratories is small, the results of the intercomparison must be considered quite satisfactory. Our analysis of the intercomparison leads to the following conclusions:

(a) The measured transmittance differences between the three laboratories and NIST, although real, generally were less than the uncertainty of the intercomparison,

U_{TOTAL}. The PTB always measures τ larger than the NIST while the NPL measures τ larger than the NIST for the three highest transmittances, and measures τ smaller than the NIST for the four smallest transmittances.

(b) The relative sample uncertainty U_{SAMPLE} is a major contributing factor to the total relative uncertainty, U_{TOTAL}, for most of the filters studied (see Fig. 4). U_{SAMPLE} is 40% or more of U_{TOTAL} except in a few cases.

(c) The results of this intercomparison indicate that sample-selection, sample-preparation and/or sample-handling techniques probably need further investigation.

Acknowledgements. Kenneth L. Eckerle of NIST wishes to thank Klaus D. Mielenz and Jack J. Hsia for their support. He would also like to acknowledge Chris E. Kuyatt for his critical review of this paper, and John Mandel for his analysis of variance and suggestions for Figs. 1–3.

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