

A Bilateral Intercomparison of Disseminated Transmittance Standards

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Abstract

Spectrophotometry is a widely used technique for both qualitative and quantitative analysis in the pharmaceutical and chemical industries as well as in medical laboratories. The National Physical Laboratory (NPL) in the UK, and the National Institute of Standards and Technology (NIST) in the USA, both issue transfer standards in the form of solid filters that provide traceability for transmittance measurements to users in industry. The two laboratories have held an intercomparison to see how well these disseminated scales agree. The standards included both glass and metal-on-silica filters, with transmittances ranging from 90% to 1%, calibrated at selected wavelengths between 250 nm and 635 nm. The results show an acceptable level of agreement between the two laboratories for transmittances between 90% and 10%. Further work will be required to establish the reasons for the poorer agreement for transmittances below 10%.

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1 INTRODUCTION

Spectrophotometry is a widely used technique for both qualitative and quantitative analysis in the pharmaceutical and chemical industries as well as in medical laboratories. The National Physical Laboratory (NPL) in the UK, and the National Institute of Standards and Technology (NIST) in the USA, both issue transfer standards in the form of solid filters that provide traceability for transmittance measurements to users in industry. The purpose of this intercomparison was to investigate how well these disseminated scales agree.

2 MATERIALS

NPL supplied two sets of its neutral density filters for the intercomparison [1]. One set contained seven filters, with nominal transmittances of 90%, 73%, 60%, 30%, 10%, 3% and 1%, made from Schott NG series glass. The other set contained six filters, with nominal transmittances of 90%, 60%, 30%, 10%, 3% and 1%, made from silica substrates coated on one major face with a nickel chromium alloy (except for the 90% filter which is uncoated).

NIST supplied three of its Standard Reference Materials (SRMs) for the intercomparison. SRMs 930e and 1930 contain a total of six filters, with nominal transmittances of 50%, 30%, 20%, 10%, 3% and 1%, made from Schott NG series glass [2]. SRM 2031a contains three filters with nominal transmittance of 90%, 30% and 10% [3]. The silica base plates of the 10% and 30% filters carry different thicknesses of semi-transparent chromium metal that are optically contacted to silica cover plates. The nominal 90% filter is a single clear silica plate.

All the filters were supplied in metal frames that fit into standard holders for 10 mm path length cuvettes.

3 MEASUREMENTS

The measurement wavelengths selected were 440, 465, 546.1, 590 and 635 nm for the glass filters, and 250, 280, 340, 360, 400, 465, 500, 546.1, 590 and 635 nm for the metal-on-silica filters. The ambient temperature selected was 21 ± 1 °C. These conditions are used by NIST to certify its SRMs (NPL does not have standard conditions for its transfer standard calibrations).

The filters were cleaned before the intercomparison began. Subsequently, they were only blown with filtered, compressed air to remove dust particles.

The NPL measurements were made using a Varian Cary 5E spectrophotometer, a commercial double beam instrument, with a double monochromator and a photomultiplier tube detector. The accuracy of this instrument has been determined and is periodically checked using transfer standards calibrated on the NPL Reference Spectrophotometer. The spectral bandwidth was 1.0 nm, and the measured area was a

patch approximately 13 mm x mm, centred horizontally and with its centre 15 mm above the base of the frame.

The NIST measurements were made using the high-accuracy spectrophotometer designed and built in the NIST Analytical Chemistry Division, a single beam reference instrument with an integrating sphere detection system [4]. The spectral bandwidth was either 0.8 nm or 1.6 nm, depending on the filter and wavelength, and the measured area was a patch approximately 8 mm x 1 mm, centred horizontally and with its centre 16 mm above the base of the frame.

The measurement sequence was NPL-NIST-NPL for the NPL filters, and NIST-NPL-NIST for the NIST filters. The intercomparison began in November 1998 and was completed in June 1999.

4 RESULTS

The results of the intercomparison are given in Tables 1-4. The transmittance values obtained by either NPL or NIST at the beginning and end of the exchange are given in the 'before' and 'after' columns, with the mean given in the 'average' column, and the corresponding values obtained by the other laboratory in the next column.

The expanded uncertainties U_{NPL} and U_{NIST} are stated for a coverage factor $k = 2$, providing a level of confidence of approximately 95% [5]. Three factors contribute to the NIST uncertainties being higher than the NPL ones:

- NIST includes a larger uncertainty for temperature (± 2 °C compared to ± 1 °C);
- 2. NIST includes an uncertainty for non-uniformity, NPL does not;
- 3. NIST includes an uncertainty for drift (over a two year period), NPL does not

For the purposes of this intercomparison, the allowances made by NIST for temperature and drift are generous, since both laboratories made measurements at 21 ± 1 °C, and the intercomparison was completed within seven months.

The final two columns give the combined uncertainties U_{TOTAL} (calculated as $[U_{\text{NPL}}^2 + U_{\text{NIST}}^2]^{1/2}$) and the NIST-NPL differences. All uncertainties and differences are expressed in absolute terms.

The two laboratories can be said to agree if the NIST-NPL differences are less than or equal to the combined uncertainties U_{TOTAL} .

5 DISCUSSION AND CONCLUSIONS

The following comments can be made:

1. The agreement is good for higher transmittances, getting progressively worse as transmittance decreases (see Figures 1-4). The two laboratories agree within the combined uncertainty U_{TOTAL} for all the filters except G1.0, G2.0, M1.5, M2.0 and 1-170. The measurements on filters M1.5 and M2.0 were made outside the normal operating range of the NIST instrument, which uses a linearity correction determined for transmittances from 100% to 6.125%.
2. NIST consistently reports higher transmittance values than NPL (with one exception, see below). A subsequent check by NIST has revealed a small drift in their linearity correction during the last two years (+0.01% at 10% and 0.0015% at 1%). Correcting for this drift would move the NIST values closer to NPL's, but not enough to explain the observed differences.
3. The instrument used by NPL has been designed to minimise errors due to possible inter-reflections between the sample and optics. In contrast, the NIST instrument uses lenses which could produce such effects. A comparison of the differences obtained in the visible region for metal-on-silica and glass filters of the same nominal transmittance does not provide conclusive evidence for the existence of inter-reflection effects.
4. The agreement is generally better with the NIST rather than the NPL filters (see Figures 5 and 6). NIST tests every filter for non-uniformity and rejects those outside a given tolerance, which NPL does not. In addition, the NPL filters cannot easily be removed from their frames, unlike the NIST ones, which restricts the techniques that can be used for cleaning. These factors may have resulted in larger non-uniformities for the NPL filters.
5. The differences for the metal-on-silica filters appear to show some dependence on wavelength, with a peak around 360 nm (see Figures 2-4). For the NPL filters, the larger differences coincide with a steep slope in the spectral profile of the filters, suggesting wavelength or bandwidth errors. Alternatively, the differences may be due to the change of source lamp between the UV and visible regions.
6. Glass filter G0.05, which drifted significantly during the intercomparison, is the only filter for which NIST reported significantly lower transmittance values than NPL (see Figure 7).

The main conclusions are

1. The agreement between NPL and NIST is acceptable for transmittances between 100% and 10%, although an offset exists between the two scales.
2. Further work will be required to establish the reasons for the poorer agreement for transmittances below 10%.

3. Careful selection and preparation of the filters appears to be important in producing high quality transfer standards.

6 ACKNOWLEDGEMENTS

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Table 1
Results for NPL glass filters

Filter	Wl (nm)	NPL before	Spectral Transmittance (T%)			U _{NPL}	U _{NIST}	U _{TOTAL}	NIST- NPL
			NPL after	NPL average	NIST				
G0.05	440.0	91.12	91.36	91.24	91.07	0.30	0.38	0.48	-0.17
	465.0	91.25	91.47	91.36	91.21	0.30	0.38	0.48	-0.15
	546.1	91.49	91.65	91.57	91.42	0.30	0.38	0.48	-0.15
	590.0	91.55	91.70	91.62	91.47	0.30	0.38	0.48	-0.15
	635.0	91.59	91.73	91.66	91.51	0.30	0.38	0.48	-0.15
G0.15	440.0	70.99	71.00	71.00	71.06	0.21	0.33	0.39	0.06
	465.0	73.05	73.06	73.05	73.13	0.22	0.33	0.39	0.08
	546.1	72.94	72.95	72.95	73.02	0.21	0.33	0.39	0.07
	590.0	71.33	71.35	71.34	71.42	0.21	0.33	0.39	0.08
	635.0	70.63	70.64	70.64	70.70	0.20	0.33	0.39	0.07
G0.25	440.0	56.87	56.85	56.86	56.94	0.14	0.28	0.32	0.08
	465.0	60.36	60.34	60.35	60.41	0.15	0.28	0.32	0.06
	546.1	60.14	60.12	60.13	60.21	0.15	0.28	0.32	0.08
	590.0	58.48	58.46	58.47	58.57	0.15	0.28	0.32	0.10
	635.0	58.31	58.30	58.30	58.41	0.15	0.28	0.32	0.11
G0.5	440.0	29.87	29.89	29.88	29.93	0.10	0.16	0.19	0.05
	465.0	32.30	32.32	32.31	32.37	0.10	0.16	0.19	0.06
	546.1	31.18	31.21	31.19	31.28	0.10	0.16	0.19	0.09
	590.0	29.85	29.87	29.86	29.97	0.10	0.16	0.19	0.11
	635.0	31.52	31.55	31.54	31.65	0.10	0.16	0.19	0.11
	440.0	10.41	10.40	10.40	10.47	0.05	0.06	0.08	0.07
	465.0	12.11	12.10	12.10	12.16	0.05	0.06	0.08	0.06
	546.1	11.29	11.28	11.28	11.36	0.05	0.06	0.08	0.08
	590.0	10.35	10.34	10.35	10.45	0.05	0.06	0.08	0.10
	635.0	11.50	11.49	11.50	11.59	0.05	0.06	0.08	0.09
	440.0	2.857	2.853	2.855	2.872	0.017	0.040	0.043	0.017
	465.0	3.520	3.515	3.517	3.538	0.020	0.040	0.045	0.021
	546.1	3.278	3.274	3.276	3.305	0.019	0.040	0.044	0.029
	590.0	3.047	3.042	3.044	3.078	0.018	0.040	0.044	0.034
	635.0	3.708	3.704	3.706	3.746	0.021	0.040	0.045	0.041
	440.0	0.909	0.909	0.909	0.919	0.006	0.010	0.012	0.010
	465.0	1.231	1.230	1.231	1.242	0.008	0.010	0.013	0.011
	546.1	1.147	1.146	1.147	1.162	0.008	0.010	0.013	0.015
	590.0	1.079	1.077	1.078	1.096	0.007	0.010	0.012	0.018
	635.0	1.434	1.433	1.433	1.454	0.009	0.010	0.014	0.021

Table 2
Results for NPL metal-on-silica filters

Filter	Wl (nm)	NPL before	Spectral Transmittance (T%)			U _{NPL}	U _{NIST}	U _{TOTAL}	NIST- NPL
			NPL after	NPL average	NIST				
M0.05	250.0	91.00	91.00	91.00	91.02	0.30	0.38	0.48	0.02
	280.0	92.16	92.16	92.16	92.13	0.30	0.38	0.48	-0.03
	340.0	92.66	92.68	92.67	92.73	0.30	0.38	0.48	0.06
	360.0	92.79	92.79	92.79	92.83	0.30	0.38	0.48	0.04
	400.0	92.93	92.94	92.94	92.95	0.30	0.38	0.48	0.01
	465.0	93.09	93.09	93.09	93.08	0.30	0.38	0.48	-0.01
	500.0	93.16	93.16	93.16	93.17	0.30	0.38	0.48	0.01
	546.1	93.22	93.22	93.22	93.21	0.30	0.38	0.48	-0.01
	590.0	93.25	93.26	93.26	93.27	0.30	0.38	0.48	0.01
	635.0	93.29	93.31	93.30	93.30	0.30	0.38	0.48	0.00
M0.25	250.0	51.81	51.80	51.80	51.89	0.14	0.33	0.36	0.09
	280.0	52.48	52.48	52.48	52.58	0.14	0.33	0.36	0.10
	340.0	56.30	56.32	56.31	56.42	0.14	0.33	0.36	0.11
	360.0	57.03	57.04	57.03	57.18	0.15	0.33	0.36	0.15
	400.0	57.64	57.66	57.65	57.73	0.15	0.33	0.36	0.08
	465.0	57.76	57.80	57.78	57.88	0.15	0.33	0.36	0.10
	500.0	57.75	57.78	57.76	57.85	0.15	0.33	0.36	0.09
	546.1	57.72	57.75	57.74	57.85	0.15	0.33	0.36	0.11
	590.0	57.69	57.72	57.71	57.81	0.15	0.33	0.36	0.10
	635.0	57.65	57.69	57.67	57.76	0.15	0.33	0.36	0.09
M0.5	250.0	26.90	26.89	26.90	27.03	0.10	0.26	0.28	0.14
	280.0	27.41	27.41	27.41	27.51	0.10	0.26	0.28	0.10
	340.0	30.54	30.54	30.54	30.67	0.10	0.26	0.28	0.13
	360.0	30.95	30.95	30.95	31.10	0.10	0.26	0.28	0.15
	400.0	31.06	31.06	31.06	31.13	0.10	0.26	0.28	0.07
	465.0	30.76	30.76	30.76	30.84	0.10	0.26	0.28	0.08
	500.0	30.73	30.73	30.73	30.83	0.10	0.26	0.28	0.10
	546.1	30.84	30.84	30.84	30.93	0.10	0.26	0.28	0.09
	590.0	30.92	30.95	30.94	31.05	0.10	0.26	0.28	0.11
	635.0	30.99	31.02	31.00	31.12	0.10	0.26	0.28	0.12
M1.0	250.0	5.94	5.94	5.94	6.03	0.03	0.09	0.10	0.09
	280.0	7.12	7.12	7.12	7.21	0.04	0.09	0.10	0.09
	340.0	9.75	9.76	9.76	9.85	0.05	0.09	0.10	0.09
	360.0	10.05	10.06	10.06	10.16	0.05	0.09	0.10	0.10
	400.0	10.20	10.20	10.20	10.27	0.05	0.09	0.10	0.07
	465.0	10.19	10.19	10.19	10.26	0.05	0.09	0.10	0.07
	500.0	10.29	10.29	10.29	10.36	0.05	0.09	0.10	0.07
	546.1	10.48	10.48	10.48	10.54	0.05	0.09	0.10	0.06
	590.0	10.61	10.61	10.61	10.70	0.05	0.09	0.10	0.09
	635.0	10.67	10.68	10.67	10.76	0.05	0.09	0.10	0.09

Table 2
Results for NPL metal-on-silica filters

Filter	Wl (nm)	NPL before	Spectral Transmittance (T%)			U _{NPL}	U _{NIST}	U _{TOTAL}	NIST- NPL
			NPL after	NPL average	NIST				
	250.0	1.063	1.062	1.063	1.100	0.008	0.030	0.031	0.037
	280.0	1.650	1.648	1.649	1.692	0.011	0.030	0.032	0.043
	340.0	3.066	3.067	3.066	3.125	0.017	0.030	0.035	0.058
	360.0	3.207	3.206	3.207	3.267	0.019	0.030	0.036	0.060
	400.0	3.213	3.209	3.211	3.255	0.019	0.030	0.036	0.044
	465.0	3.077	3.073	3.075	3.121	0.018	0.030	0.035	0.046
	500.0	3.063	3.059	3.061	3.107	0.018	0.030	0.035	0.046
	546.1	3.083	3.080	3.082	3.124	0.018	0.030	0.035	0.042
	590.0	3.090	3.087	3.089	3.141	0.018	0.030	0.035	0.053
	635.0	3.085	3.083	3.084	3.134	0.018	0.030	0.035	0.050
	250.0	0.290	0.290	0.290	0.316	0.002	0.010	0.010	0.025
	280.0	0.533	0.532	0.532	0.563	0.004	0.010	0.011	0.031
	340.0	1.149	1.148	1.149	1.191	0.007	0.010	0.012	0.043
	360.0	1.191	1.190	1.190	1.235	0.008	0.010	0.013	0.044
	400.0	1.144	1.142	1.143	1.174	0.008	0.010	0.013	0.031
	465.0	1.009	1.008	1.009	1.040	0.007	0.010	0.012	0.031
	500.0	0.986	0.985	0.985	1.018	0.007	0.010	0.012	0.033
	546.1	0.995	0.994	0.994	1.028	0.007	0.010	0.012	0.034
	590.0	1.011	1.010	1.010	1.045	0.007	0.010	0.012	0.035
	635.0	1.026	1.025	1.026	1.060	0.007	0.010	0.012	0.034

Table 3
Results for NIST glass filters

Filter	Wl (nm)	NIST before	Spectral Transmittance (T%)			U _{NPL}	U _{NIST}	U _{TOTAL}	NIST- NPL
			NIST after	NIST average	NPL				
50-170	440.0	50.32	50.29	50.31	50.26	0.13	0.24	0.28	0.04
	465.0	53.73	53.70	53.72	53.67	0.14	0.26	0.29	0.04
	546.1	53.14	53.12	53.13	53.05	0.14	0.26	0.29	0.08
	590.0	50.33	50.29	50.31	50.24	0.13	0.24	0.28	0.07
	635.0	49.37	49.33	49.35	49.29	0.13	0.24	0.27	0.06
30-2273	440.0	28.58	28.57	28.58	28.56	0.10	0.15	0.18	0.01
	465.0	31.88	31.88	31.88	31.86	0.10	0.17	0.20	0.02
	546.1	31.01	31.01	31.01	30.98	0.10	0.17	0.20	0.03
	590.0	28.93	28.92	28.92	28.88	0.10	0.16	0.19	0.05
	635.0	29.88	29.85	29.87	29.82	0.10	0.16	0.19	0.05
20-2273	440.0	18.40	18.40	18.40	18.36	0.07	0.10	0.12	0.04
	465.0	20.68	20.68	20.68	20.64	0.07	0.11	0.13	0.04
	546.1	19.61	19.61	19.61	19.56	0.07	0.10	0.12	0.05
	590.0	18.26	18.26	18.26	18.20	0.07	0.10	0.12	0.06
	635.0	19.67	19.66	19.67	19.60	0.07	0.10	0.12	0.07
10-2273	440.0	6.35	6.35	6.35	6.31	0.03	0.04	0.05	0.03
	465.0	7.72	7.71	7.72	7.68	0.04	0.04	0.05	0.03
	546.1	7.19	7.17	7.18	7.13	0.03	0.04	0.05	0.05
	590.0	6.45	6.44	6.44	6.39	0.03	0.04	0.05	0.05
	635.0	7.40	7.38	7.39	7.34	0.03	0.04	0.05	0.05
3-170	440.0	2.393	2.392	2.393	2.381	0.013	0.027	0.030	0.012
	465.0	3.140	3.148	3.144	3.127	0.017	0.036	0.039	0.017
	546.1	2.959	2.967	2.963	2.939	0.016	0.033	0.037	0.024
	590.0	2.753	2.763	2.758	2.731	0.015	0.031	0.035	0.027
	635.0	3.427	3.436	3.432	3.402	0.018	0.039	0.043	0.030
	440.0	0.689	0.688	0.689	0.680	0.005	0.009	0.010	0.009
	465.0	0.951	0.953	0.952	0.942	0.006	0.012	0.013	0.010
	546.1	0.865	0.870	0.868	0.854	0.006	0.011	0.012	0.014
	590.0	0.804	0.806	0.805	0.792	0.005	0.010	0.011	0.013
	635.0	1.062	1.065	1.064	1.049	0.006	0.013	0.015	0.015

Table 4
Results for NIST metal-on-silica filters

Filter	Wl (nm)	NIST before	Spectral Transmittance (T%)			U _{NPL}	U _{NIST}	U _{TOTAL}	NIST- NPL
			NIST after	NIST average	NPL				
416-90	250.0	91.47	91.53	91.50	91.54	0.30	0.38	0.48	-0.04
	280.0	92.05	92.04	92.05	92.11	0.30	0.38	0.48	-0.06
	340.0	92.65	92.66	92.66	92.63	0.30	0.38	0.48	0.03
	360.0	92.74	92.74	92.74	92.72	0.30	0.38	0.48	0.02
	400.0	92.88	92.85	92.87	92.90	0.30	0.38	0.48	-0.03
	465.0	92.99	93.02	93.01	93.03	0.30	0.38	0.48	-0.02
	500.0	93.07	93.10	93.08	93.09	0.30	0.38	0.48	-0.01
	546.1	93.13	93.14	93.14	93.15	0.30	0.38	0.48	-0.01
	590.0	93.15	93.17	93.16	93.19	0.30	0.38	0.48	-0.03
	635.0	93.19	93.16	93.17	93.23	0.30	0.38	0.48	-0.05
416-30	250.0	36.20	36.20	36.20	36.14	0.11	0.32	0.34	0.06
	280.0	36.22	36.23	36.23	36.17	0.11	0.32	0.34	0.05
	340.0	34.89	34.86	34.88	34.79	0.11	0.31	0.33	0.09
	360.0	34.51	34.50	34.51	34.43	0.11	0.30	0.32	0.07
	400.0	33.88	33.88	33.88	33.84	0.11	0.30	0.32	0.03
	465.0	33.81	33.81	33.81	33.75	0.11	0.30	0.32	0.06
	500.0	34.32	34.34	34.33	34.27	0.11	0.30	0.32	0.06
	546.1	35.42	35.44	35.43	35.37	0.11	0.31	0.33	0.06
	590.0	36.64	36.68	36.66	36.61	0.11	0.32	0.34	0.05
	635.0	37.94	37.95	37.95	37.86	0.11	0.33	0.35	0.09
416-10	250.0	6.72	6.71	6.72	6.63	0.04	0.07	0.08	0.08
	280.0	7.14	7.13	7.13	7.06	0.04	0.07	0.08	0.07
	340.0	7.65	7.64	7.64	7.57	0.04	0.07	0.08	0.08
	360.0	7.90	7.91	7.90	7.82	0.04	0.08	0.09	0.08
	400.0	8.36	8.38	8.37	8.30	0.04	0.08	0.09	0.07
	465.0	9.42	9.43	9.43	9.36	0.04	0.09	0.10	0.07
	500.0	10.20	10.21	10.20	10.14	0.05	0.10	0.11	0.06
	546.1	11.34	11.37	11.35	11.29	0.05	0.11	0.12	0.06
	590.0	12.41	12.44	12.42	12.36	0.05	0.12	0.13	0.06
	635.0	13.40	13.40	13.40	13.33	0.06	0.13	0.14	0.07

NIST-NPL differences for filter M0.05 (NPL 90% silica)

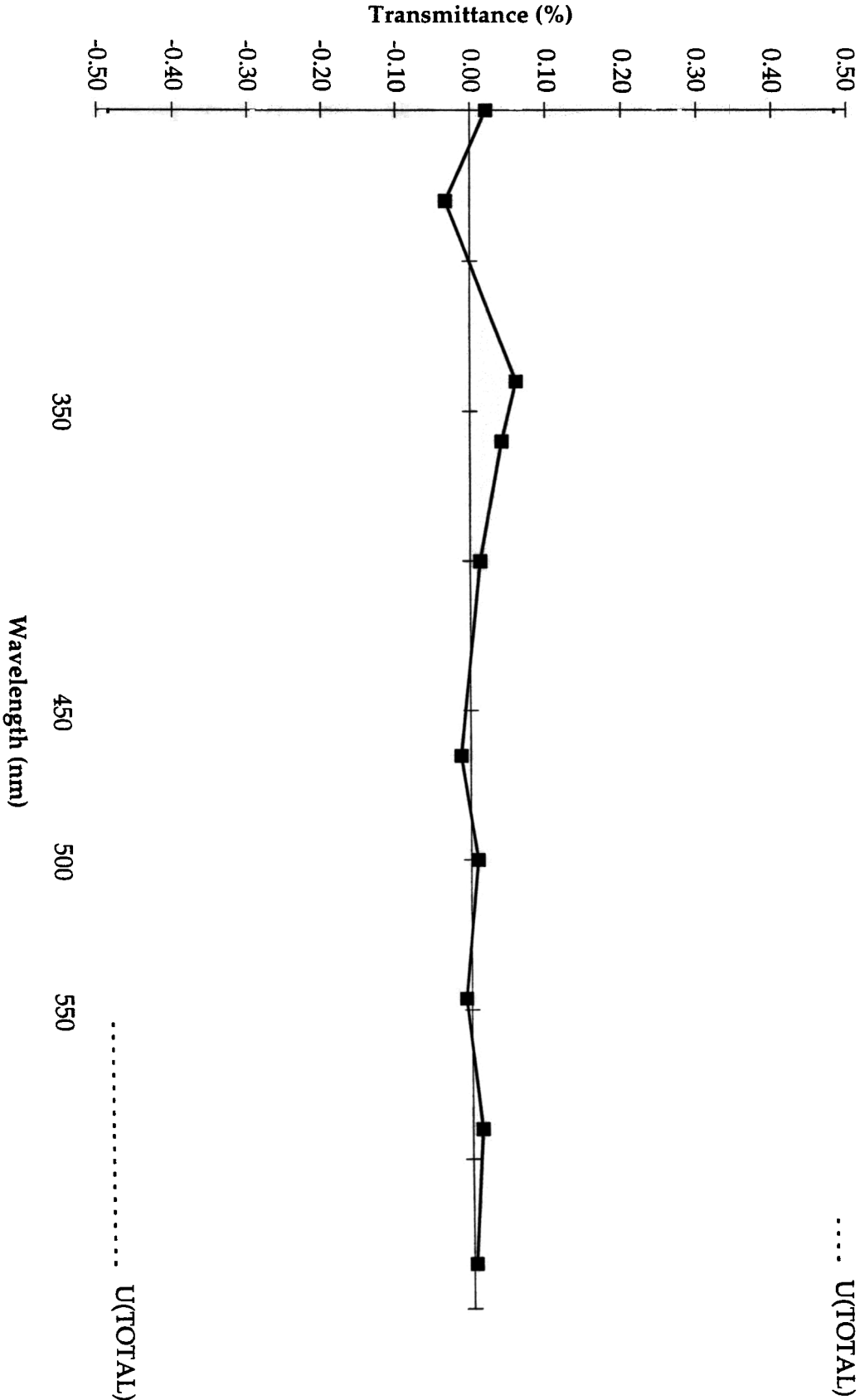


Figure 1

NIST-NPL differences

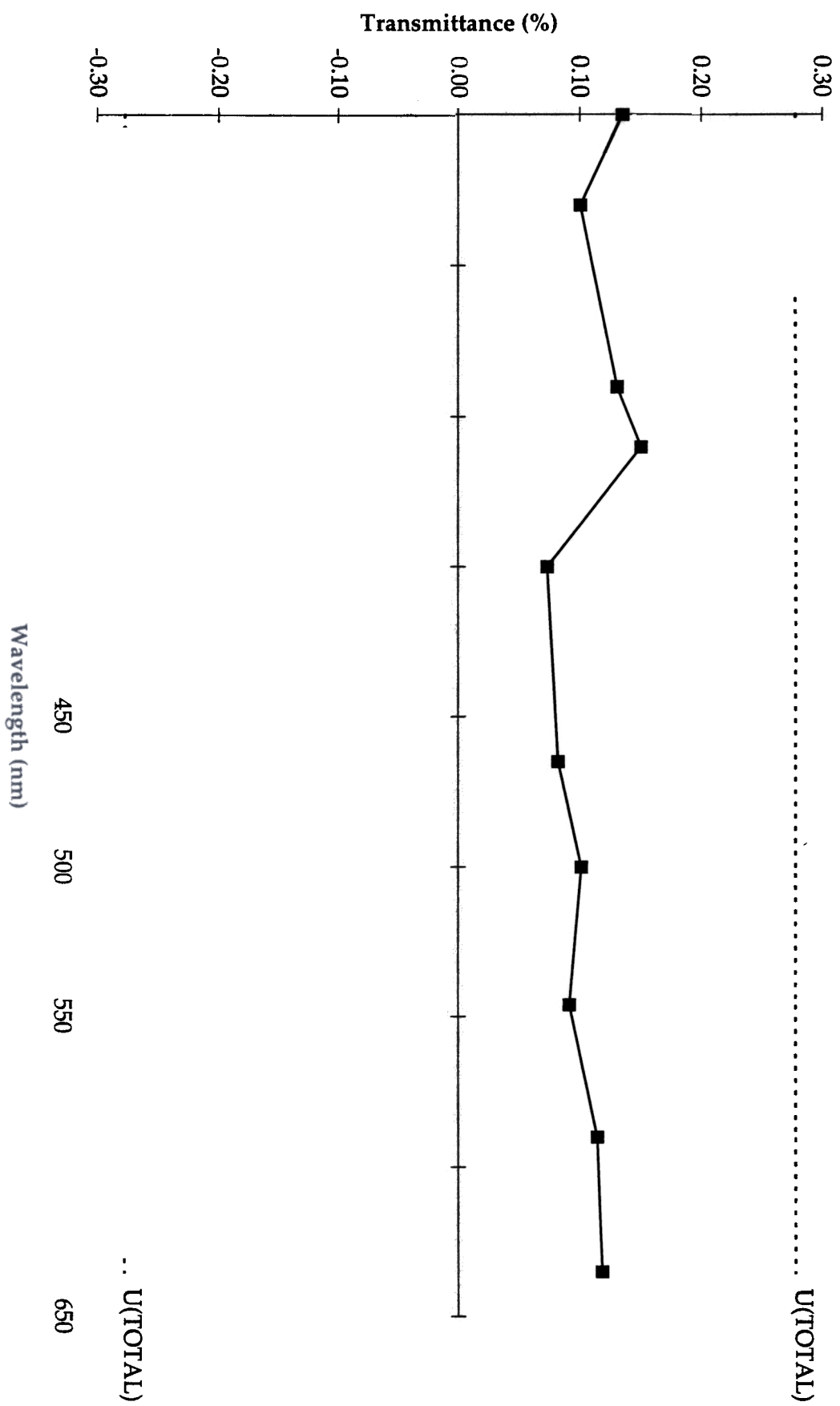


Figure 2

NIST-NPL differences for filter M1.0 (NPL 10% metal-on-silica)

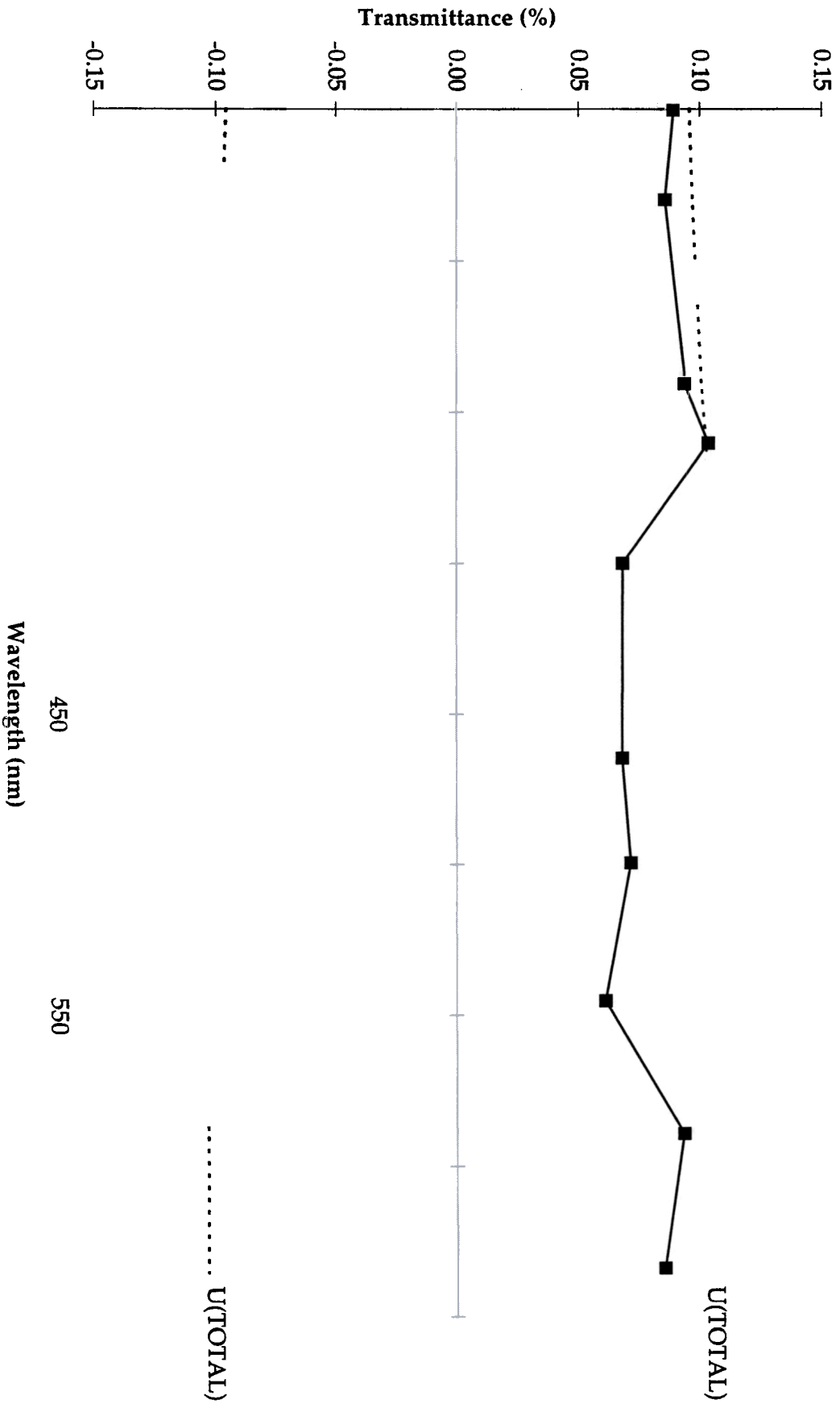


Figure 3

NIST-NPL differences for filter M2.0 (NPL 1% metal-on-silica)

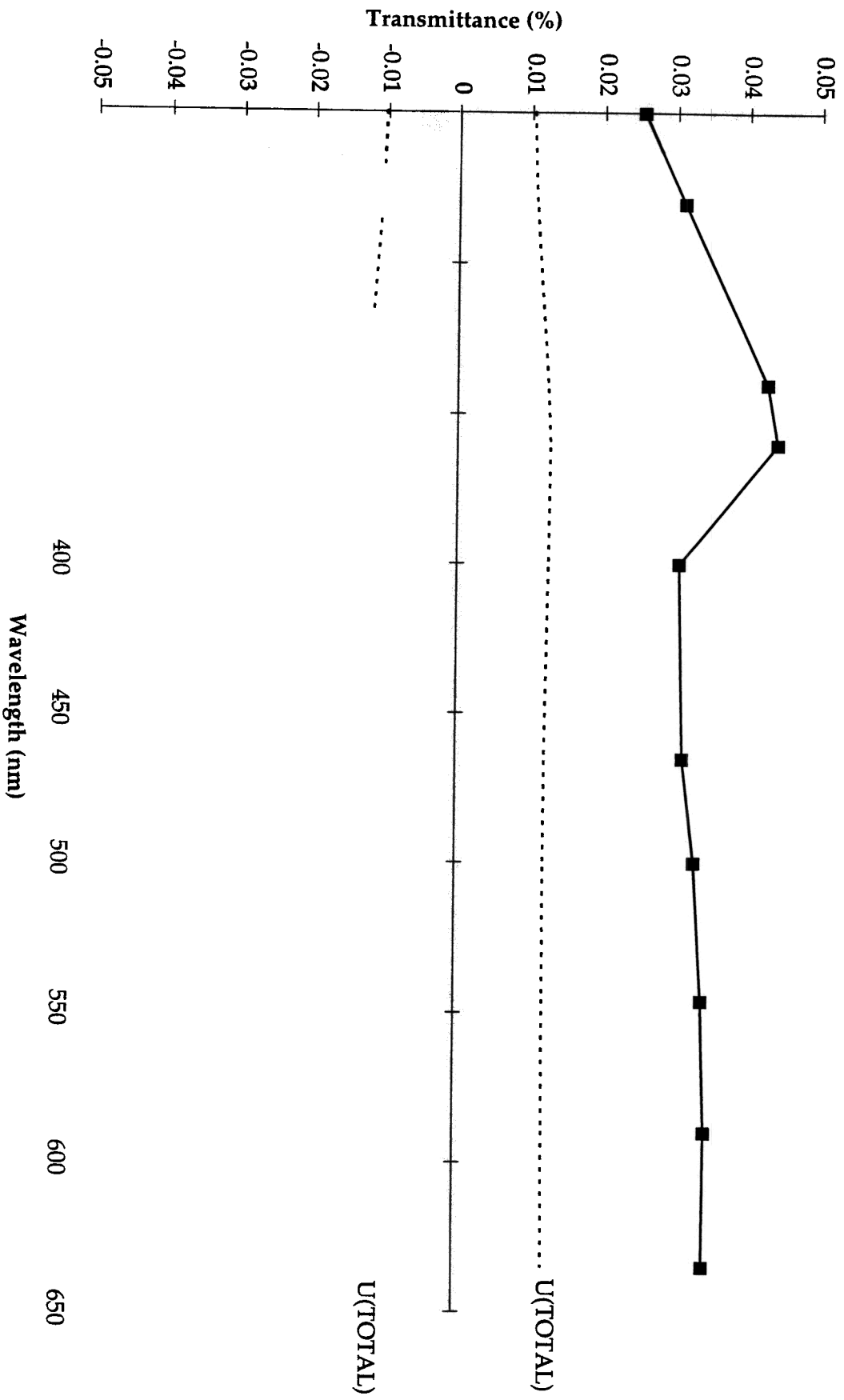


Figure 4

NIST-NPL differences for filter G0.5 (NPL 30% glass)

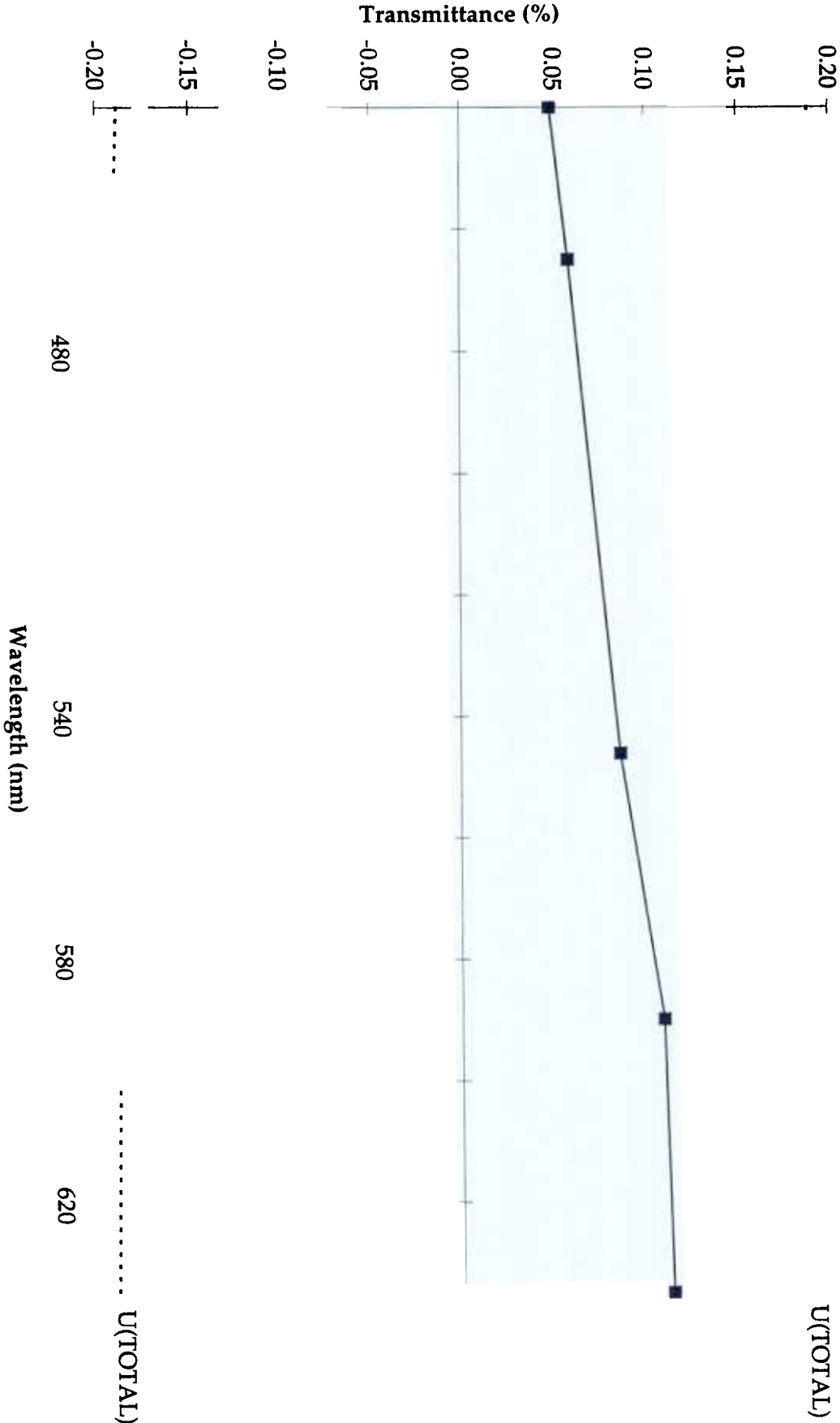


Figure 5

NIST-NPL differences for filter 30-2273 (NIST 30% glass)

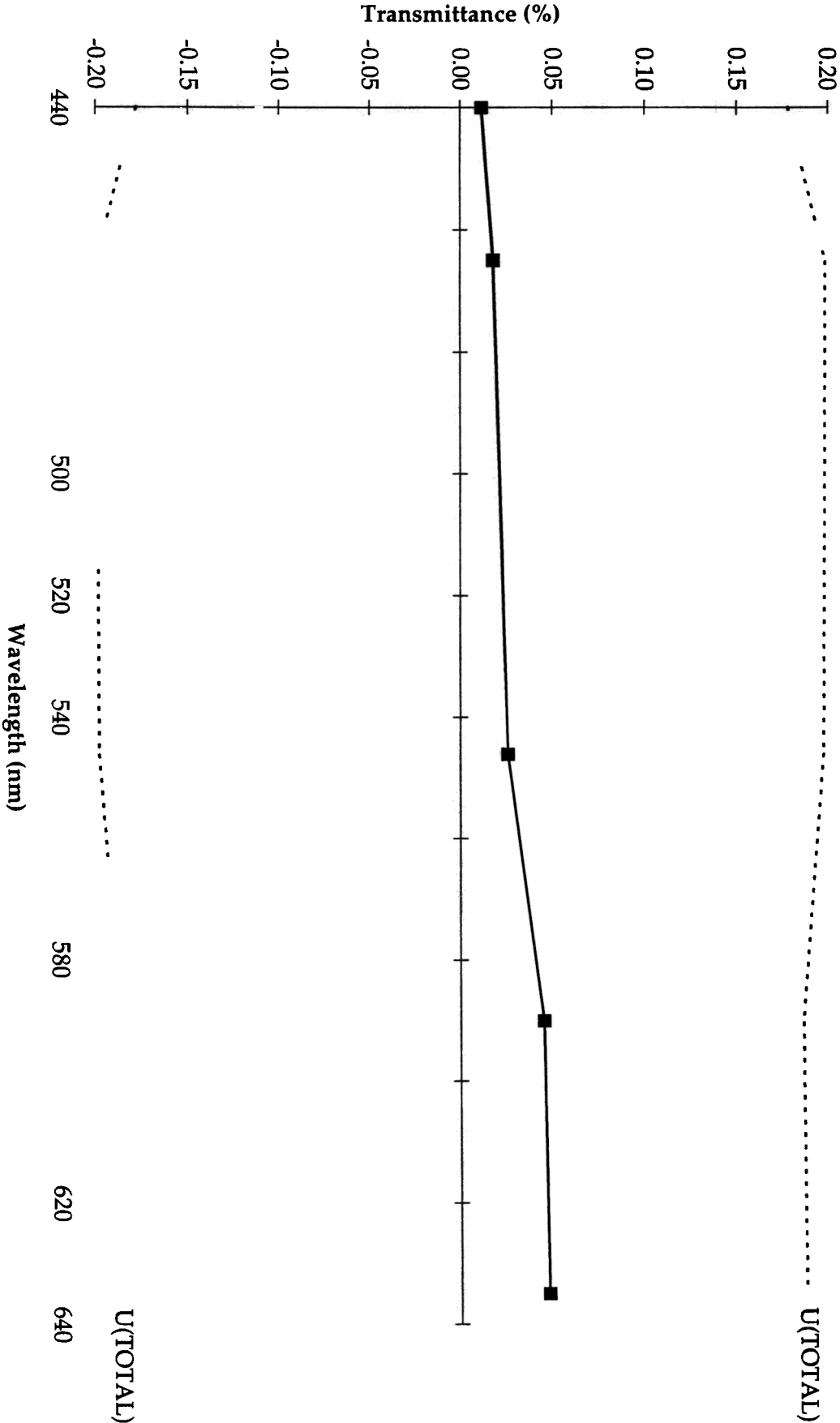


Figure 6

NIST-NPL differences for filter G0.05 (NPL 90% glass)

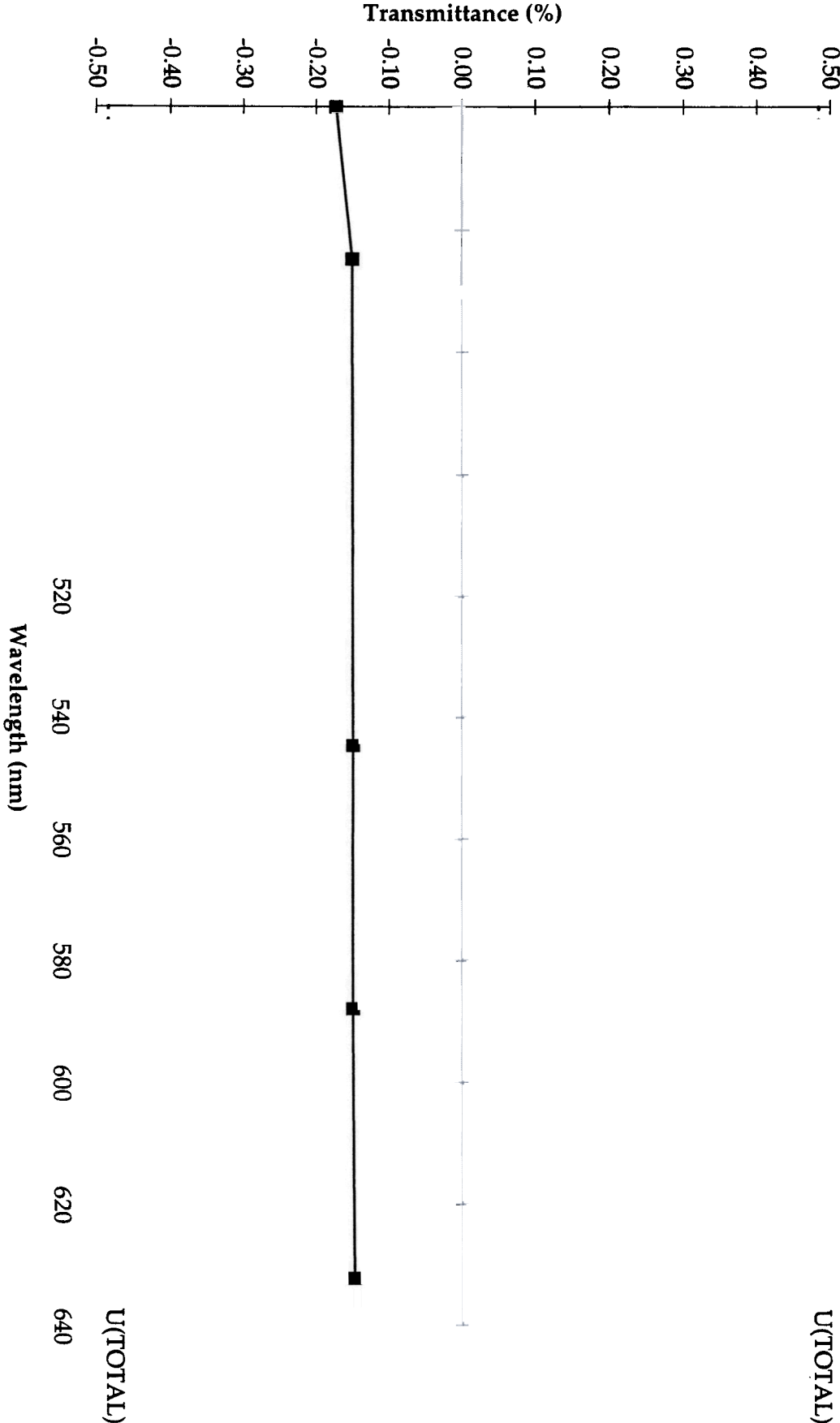


Figure 7