REFRACTIVE INDEX LIQUID CALIBRATION USING CALIBRATED REFRACTIVE INDEX SOLIDS

A STANDARD OPERATION PROCEDURE FOR BULK ASBESTOS ANALYSIS BY POLARIZED LIGHT MICROSCOPY

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The electronic formats (MS WORD or Acrobat PDF) of this paper and the *Create_RI_Liquid_Calibration_Convertion_Tables.xls* mentioned on p.2 for generating conversion tables for the liquid-glass combinations other than those in Tables 3 to 6 of this paper are available upon request.

If you have any questions or suggestions concerning this procedure, I can be reached at 302-294-0117 or shuchunsu@gmail.com.

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NIST Handbook 150-3: NVLAP Bulk Asbestos Analysis
Specific Operations Checklist
(Steel et al., 1994)

5.6 The laboratory has calibrated refractive index solids or refractometer (or access to) for calibrating refractive index liquids.

5.7 The laboratory shall have written procedures for calibrating refractive index liquids:
- frequently enough to ensure reliable calibration;
- within an accuracy of ±0.004;
- including room temperature measurement of ±2°C.

Objective

Determine whether the ***actual*** $n_D^{25°C}$, the refractive index (RI) at wavelength 589.3 nm and temperature 25 °C, of an RI liquid used in asbestos identification is within ±0.004 of its ***nominal*** $n_D^{25°C}$, which is printed on the label of the liquid bottle.

Material and equipment

1. Calibrated refractive index solids, e.g., Cargille calibrated refractive index glasses.
2. Polarized light microscope with dispersion staining objective.
3. Thermometer with at least 1 °C division.

Methodology

There are four steps in performing this calibration using calibrated refractive index solids and dispersion staining method (Su, 1992).

1. Determine the liquid temperature, t

Find out what is the temperature, t, of the RI liquid to be calibrated. Generally, it is assumed that the temperature of the liquid is in equilibrium with the room temperature. If that is the case, the room temperature can be measured to represent the liquid temperature.

2. Determine the matching wavelength, λ_0

Compare the liquid's RI with that of a calibrated refractive index solid with accurately and precisely known RI's at various wavelengths to determine at which wavelength their RI values are equal. This wavelength is called the ***matching wavelength***, λ_0, which can be derived from the dispersion staining color exhibited by glass particles (see Table 1).

3. Determine n_D^L

Calculate n_D^L, the RI value of the liquid at the wavelength of Fraunhöfer spectral D line or 589.3 nm and temperature t by using the following equation (Su, 1998):

$$n_D^L = n_D^S - (\Delta^L - \Delta^S) \bullet k_D \,, \qquad (1)$$

where n_D^L – the refractive index of the liquid at 589 nm and t °C;
n_D^S – the RI value of the calibrated refractive index solid at 589 nm, which is listed in the optical constant table supplied with every set of glasses;
Δ^L – the dispersion coefficient, $(n_F - n_C)$, of the liquid, which is printed on the bottle label;
Δ^S – the dispersion coefficient, $(n_F - n_C)$, of the calibrated refractive index solid, which is listed in the optical constant table supplied with every set of glasses;
k_D – a coefficient determined by the matching wavelength λ_0 as listed in Table 2 (Su, 1998).

4. Determine the ***actual*** or ***calibrated*** $n_D^{25°C}$

If the room temperature is not 25 °C, apply temperature correction to n_D^L to find out $n_D^{25°C}$, the RI value of the liquid at 25°C and 589 nm. The equation used for temperature correction is

$$n_D^{25°C} = n_D^L + (25 - t) \bullet dn/dt \,, \qquad (2)$$

where $n_D^{25°C}$ – the refractive index of the liquid at 589.3 nm and 25 °C;
n_D^L – the R.I. value of the liquid at 589.3 nm and t °C;
t – the temperature in centigrade at which the calibration is performed; and
dn/dt – the temperature coefficient of the liquid, which is printed on the bottle label and is always a negative value for R.I. liquids.

No temperature correction is necessary for the glass if t is within the range of 10 °C to 40 °C, because the temperature coefficients of Cargille glasses are so small that the corresponding temperature correction will ***not exceed ±0.0001***.

For RI liquids commonly used in routine asbestos identification, i.e., 1.550, 1.605, 1.680, and 1.700, the calculations in Steps 3 and 4 can be eliminated by using conversion tables as listed in Tables 3 to 9. For the liquid-glass combinations other than those listed in Tables 3 to 9, an Excel workbook *Create_RI_Liquid_Calibration_Convertion_Tables.xls* can be used to generate corresponding conversion tables. Another alternative is to calculate the ***actual*** or ***calibrated*** $n_D^{25°C}$ using Equations (1) and (2) by the aid of the worksheet on p.4.

Procedure

1. Measure and record the room temperature, t, with an accuracy of ±2 °C.

2. Select a calibrated refractive index solid whose RI is closest to that of the liquid to be calibrated. For example, use 1.55 glass for 1.550 liquid, 1.60 glass for 1.605 liquid, and 1.68 glass for 1.680 liquid.

3. Mount the glass in the liquid and observe the glass particles' ***predominant*** dispersion staining (DS) color on a polarized light microscope. Central stop (CS) mode is preferred. *Caution: Sometimes glass fragments with flat edges that are vertical to or at very high angle with the glass slide surface may display false CSDS color at these edges, e.g., orange CSDS color for 1.55 glass fragments in 1.550 liquid. If such false CSDS color appears, observation should be focused on the interior of glass fragments or fragments without those types of steep flat edges.*

4. Convert the observed CSDS color into the corresponding matching wavelength, λ_0, by referring to Table 1 or a DS color chart, which can be found in Reference 4.

5. Convert λ_0 and t into the corresponding $n_D^{25°C}$, i.e., the calibrated $n_D^{25°C}$ or $(n_D^{25°C})_{clb}$, by referring to an appropriate conversion table:

Cargille RI liquid		Cargille Calibrated Refractive Index Solid	Conversion Table No.
Nominal or Labeled $n_D^{25°C}$	Series	Nominal or Labeled RI*	
1.550	E	1.55	3
1.605	E	1.60	4
1.680	B	1.68	5
1.700	B	1.70	6

* The precise and accurate refractive indices at different wavelengths are listed in the accompanying optical constant table.

6. Compare the calibrated $n_D^{25°C}$ or $(n_D^{25°C})_{clb}$ with the nominal $n_D^{25°C}$ or $(n_D^{25°C})_{lbl}$ on the bottle of the RI liquid . If the *absolute difference* between these two values is less or equal than 0.004, this liquid can be used for bulk sample analysis. If the *absolute difference* is greater than 0.004, it should no longer be used.

7. Record the calibration result. Table 9 is a suggested format for recording the RI liquid calibration results.

Example

When the Cargille 1.55 glass of Lot C is used to calibrate a Cargille 1.550 (Series E) RI liquid at the room temperature of 21 °C, the predominant CSDS color observed is bluish purple, whose corresponding λ_0 is found to be 580 nm according to Table 1. By referring to Table 4 (1.550 liquid with 1.55 glass of Lot C), λ_0 = 580 and t = 21 °C yield 1.549, which is the interpolation between 1.548 (20 °C) and 1.549 (22 °C).

The calibration result shows that the RI of this bottle of 1.550 liquid at 589 nm and 25 °C, $(n_D^{25°C})_{clb}$, is ***actually*** 1.549. Because the difference is 0.001, using the ±0.004 criterion, this RI liquid is considered to be acceptable for being used in bulk sample analysis.

Procedure for liquid-glass combinations not listed in Tables 3 to 9

In these cases, Equations 1 and 2 can be used to obtain the calibrated $n_D^{25°C}$, i.e., the RI of the liquid at 589 nm and 25 °C. It is best to illustrate the procedure by using a worksheet as follows.

Data/Parameter		How to obtain it	Symbol Value
Cargille liquid to be calibrated	The dispersion coefficient	Read from the label of the liquid bottle	Δ^L
	The temperature coefficient	Read from the label of the liquid bottle (*negative value*)	dn/dt
	The liquid or room temperature	Read from thermometer	t
Cargille glass used in calibration	The labeled $n_D^{25°C}$	Read from the Column for 5893Å of the optical data sheet	n_D^S
	The dispersion coefficient	Subtract Column 6563 Å from Column 4861 Å of the optical data sheet	Δ^S
	Central dispersion staining color	Observed by using central stop dispersion staining mode	CSDS color
	Corresponding matching wavelength	Read from Table 1 based on the observed CSDS color	λ_0
	Corresponding conversion factor	Read from Table 2 based on the λ_0	k_D
RI of the liquid at 589.3 nm and t °C		$n_D^L = n_D^S - (\Delta^L - \Delta^S) \bullet k_D$	n_D^L
RI of the liquid at 589.3 nm and 25 °C		$n_D^{25°C} = n_D^L + (25 - t) \bullet dn/dt$	$(n_D^{25°C})_{clb}$

Compare the calibrated value $(n_D^{25°C})_{clb}$ with the $n_D^{25°C}$ on the label of the liquid bottle. If the *absolute value* of the difference is >0.004, the liquid should not be used.

Another option is to use *Create_RI_Liquid_Calibration_Convertion_Tables.xls*, which is an Excel program and available upon request, to generate conversion tables for any liquid-glass combination.

References

McCrone, W.C. (1967): "Asbestos Identification", McCrone Research Institute, Chicago

Steel, E.B., Verkouteren, J, and Alderman, D.F. (1994): "Bulk Asbestos Analysis", NIST Handbook 150-3, National Institute of Standards and Technology, U.S. Department of Commerce, Washington.

Su, S.C. (1992): "Calibration of Refractive Index Liquids By Using Optical Glass Standards With Dispersion Staining". Microscope, v.40, pp.95-108.

Su, S.C. (1998) Dispersion Staining: Principles, Analytical Relationships and Practical Applications to the Determination of Refractive Index. Microscope, v.46, pp.123-146.

Table 1. Converting dispersion staining color to corresponding λ_0 (McCrone, 1987)

Matching Wavelength λ_0, nm	Particle Edge Colors[1]		Becke Line Colors[2]	
	Annular Stop[3]	Central Stop[4]	Particle	Liquid
<340	Black violet	white	white	X
<400	dark violet	pale yellow	pale yellow	X
430	violet	yellow	pale yellow	X
455	blue	golden yellow	yellow	violet
485	blue-green	orange	orange	violet
520	green	red purple	orange-red	violet-blue
560	yellow-green	purple	red-orange	blue-violet
595	yellow	deep blue	red	blue
625	orange	blue-green	faint red	blue
660	red-brown	light blue-green	X	blue-green
700	dark red-brown	pale blue-green	X	pale blue-green
1500	black-brown	very pale blue-green	X	very pale blue-green

1. In focus
2. On focusing up
3. Observed on a brightfield
4. Observed on a darkfield

Table 2. Conversion from λ_0 to k_D (Su, 1998)

λ_0 (nm)	k_D
500	0.59
510	0.50
520	0.43
530	0.35
540	0.29
550	0.22
560	0.16
570	0.10
580	0.05
590	–0.00
600	–0.05
610	–0.10
620	–0.14
630	–0.19
640	–0.23
650	–0.27
660	–0.30
670	–0.34
680	–0.37
690	–0.40
700	–0.44
710	–0.47
720	–0.50
730	–0.53
740	–0.55
750	–0.67
800	–0.69

Table 3. Conversion Table for Calibrating RI Liquid **1.550** (Series **E**) Using Cargille Calibrated Refractive Index Solid **1.55**

λ_0 (nm)	20°C	22°C	24°C	26°C	28°C	30°C
520	1.542	1.543	1.544	1.545	1.546	1.547
540	1.544	1.545	1.546	1.547	1.548	1.549
560	1.546	1.547	1.548	1.549	1.550	1.551
580	1.548	1.549	1.550	1.551	1.551	1.552
600	1.549	1.550	1.551	1.552	1.553	1.554
620	1.551	1.552	1.553	1.554	1.554	1.555
640	1.552	1.553	1.554	1.555	1.556	1.557
660	1.553	1.554	1.555	1.556	1.557	1.558
680	1.554	1.555	1.556	1.557	1.558	1.559
700	1.555	1.556	1.557	1.558	1.559	1.560

Solid: $n_F = 1.55862$; $n_D = 1.55077$; $n_C = 1.54753$; $\Delta^S = (n_F - n_C) = 0.01109$
Liquid: $\Delta^L = (n_F - n_C) = 0.0267$; $dn/dt = -0.00049$

Table 4. Conversion Table for Calibrating RI Liquid **1.605** (Series **E**) Using Cargille Calibrated Refractive Index Solid **1.60**

λ_0 (nm)	20°C	22°C	24°C	26°C	28°C	30°C
560	1.597	1.598	1.599	1.600	1.601	1.602
580	1.598	1.599	1.600	1.601	1.602	1.603
600	1.599	1.600	1.601	1.602	1.603	1.604
620	1.600	1.601	1.602	1.603	1.604	1.604
640	1.601	1.602	1.603	1.603	1.604	1.605
660	1.601	1.602	1.603	1.604	1.605	1.606
680	1.602	1.603	1.604	1.605	1.606	1.606
700	1.603	1.603	1.604	1.605	1.606	1.607
720	1.603	1.604	1.605	1.606	1.607	1.608
740	1.604	1.604	1.605	1.606	1.607	1.608
760	1.604	1.605	1.606	1.607	1.608	1.608
780	1.604	1.605	1.606	1.607	1.608	1.609
800	1.605	1.606	1.607	1.607	1.608	1.609

Solid: $n_F = 1.61227$; $n_D = 1.60106$; $n_C = 1.59657$; $\Delta^S = (n_F - n_C) = 0.01570$
Liquid: $\Delta^L = (n_F - n_C) = 0.0243$ $dn/dt = -0.00044$

Table 5. Conversion Table for Calibrating RI Liquid **1.680** (Series **B**) Using Cargille Calibrated Refractive Index Solid **1.68**

λ_0 (nm)	20°C	22°C	24°C	26°C	28°C	30°C
560	1.672	1.673	1.674	1.675	1.676	1.677
580	1.674	1.675	1.676	1.677	1.678	1.679
600	1.677	1.678	1.679	1.680	1.680	1.681
620	1.679	1.680	1.681	1.682	1.683	1.683
640	1.681	1.682	1.682	1.683	1.684	1.685
660	1.682	1.683	1.684	1.685	1.686	1.687
680	1.684	1.685	1.686	1.687	1.688	1.689

Solid: $n_F = 1.68654$; $n_D = 1.67785$; $n_C = 1.67424$; $\Delta^S = (n_F - n_C) = 0.01230$
Liquid: $\Delta^L = (n_F - n_C) = 0.0348$; $dn/dt = -0.00048$

Table 6. Conversion Table for Calibrating RI Liquid **1.700** (Series **B**) Using Cargille Calibrated Refractive Index Solid **1.70**

λ_0 (nm)	20°C	22°C	24°C	26°C	28°C	30°C
520	1.691	1.692	1.693	1.694	1.695	1.696
540	1.694	1.695	1.696	1.697	1.698	1.699
560	1.696	1.697	1.698	1.699	1.700	1.701
580	1.699	1.699	1.700	1.701	1.702	1.703
600	1.701	1.701	1.702	1.703	1.704	1.705
620	1.702	1.703	1.704	1.705	1.706	1.707
640	1.704	1.705	1.706	1.707	1.708	1.709

Solid: $n_F = 1.71406$; $n_D = 1.70189$; $n_C = 1.69697$; $\Delta^S = (n_F - n_C) = 0.01709$
Liquid: $\Delta^L = (n_F - n_C) = 0.0370$; $dn/dt = -0.00048$

Table 7. Results of RI Liquids Calibration

$(n_D^{25°C})_{clb} = n_D^t + (25 - t)\ dn/dt$ (The temperature correction is built in the conversion tables.)

Date	RI Liquid	Cargille Glass		CSDS Color of Glass		Liquid or Room Temperature (°C)	Calibrated RI of Liquid $(n_D^{25°C})_{clb}$	Absolute Difference Between Calibrated and labeled RI $(n_D^{25°C})_{clb} - (n_D^{25°C})_{lbl}$	**A**ccept or **R**eject	Initials of Analyst
	Labeled RI $(n_D^{25°C})_{lbl}$	Labeled RI	Lot No.	Predominant CSDS Color	Corresponding λ_0 (nm)					
1	2	3	4	5	6	7	8	9	10	11
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	

1. Date: 2. The $n_D^{25°C}$ on the label of RI liquid bottle or $(n_D^{25°C})_{lbl}$; 3. The RI value on the label of Cargille calibrated glass vial; 4. The Lot No. on the label of Cargille calibrated glass vial; 5. The predominant central stop dispersion color displayed by glass fragments (do not be confused by the false CSDS color due to edge effect (see p.3). 6. The matching wavelength, λ_0, corresponding to the CSDS color in Column 5; 7. The temperature of the RI liquid or the room if the liquid's temperature can be considered to be in equilibrium with the room atmosphere; 8. The reading based on the values in Columns 6 and 7 from the conversion table for the liquid-glass combination. This value is the actual or calibrated RI of the liquid at 589 nm and 25 °C or $(n_D^{25°C})_{clb}$; 9. Column 8 minus Column 2; 10. If the *absolute* value of Column 9 is less or equal to 0.004, circle A for *acceptable*. Otherwise, circle R for *rejected*. 11. Analyst's initials.