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A rapid and accurate procedure for the determination of refractive indices of regulated asbestos minerals

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ABSTRACT

By using dispersion staining methods and pre-constructed conversion tables, it is possible to quickly and accurately determine two principal refractive indices (RI) of the six regulated asbestos minerals, chrysotile, grunerite (amosite), riebeckite (crocidolite), tremolite, actinolite, and anthophyllite, in a single immersion oil mount. This procedure is especially suitable for commercial environmental laboratories specializing in the analysis of asbestos components in bulk building materials. The effectiveness of this practical procedure has been proven through rigorous testing and extensive usage over the last decade by the majority of environmental laboratories in the U.S. The principle of this procedure is also readily applicable to RI determination in other applications: mineralogy, forensics, pharmaceutical research, particle identification, etc.

INTRODUCTION

Upon the enactment of the Asbestos Hazard Emergency Response Act (AHERA) in 1986, the U.S. Environmental Protection Agency (EPA) authorized the National Institute of Standards and Technology (NIST) to add bulk asbestos and airborne asbestos analyses to the National Voluntary Laboratory Accreditation Program (NVLAP) for the purpose of monitoring the quality performances of hundreds of American environmental laboratories specializing in asbestos analysis. NVLAP accreditation is required for any commercial laboratories engaging in work related to asbestos survey, abatement, and monitoring projects at American public schools. To meet the requirements set forth by EPA (1993) and NIST (1994), analysts must measure and record two principal refractive indices, n_α and n_γ, of every type of asbestos mineral found in bulk samples. Facing a daily workload of 60 or more samples, analysts must be able to quickly determine the RI of suspected asbestos minerals with an accuracy ±0.005 to 0.010 as required by the NVLAP. The Becke line method is too time-consuming because more than one preparation is needed to bracket the RI of every type of asbestos minerals in a sample. Therefore, the dispersion staining method (McCrone 1987; Su 1998) became the method of choice because of its simplicity and effectiveness. Tens of thousands of bulk asbestos samples are being analyzed every day; the significance of any procedure that facilitates this special environmental analysis cannot be overemphasized.

DISPERSION STAINING

The dispersion staining (DS) method uses a central stop (CS) or an annular stop (AS) at the back focal plane of a 10× objective lens to block (CS mode) or single out (AS mode) the wavelength at which the RI of a solid particle equals that of the surrounding immersion liquid (Su 1998). This wavelength is defined as the matching wavelength, λ_0 (McCrone 1987) or λ_m (Bloss 1999), at which the dispersion curve of the solid intersects that of the liquid. The DS color forming at the edge of the solid particle is a function of λ_0 and the dispersion property of both the solid and the liquid. In the CS mode, it consists of a mixture of wavelengths in the visible spectrum with λ_0 removed by the central stop. In the AS mode, it consists of λ_0 with the rest of the visible spectrum removed by the annular stop. It has been proven (Su 1993) that n_D^S, the RI of the solid at λ_D, Fraunhöfer spectral D line (589 nm), is related to λ_0 and the dispersion coefficients of the solid and the liquid by the following equation:

$$n_D^S = n_D{}^L + (\Delta^L - \Delta^S)\, k_D \qquad (1)$$

where n_D^L = the refractive index of the liquid at λ_D and the temperature of the liquid T in °C;
Δ^L = the dispersion coefficient, $(n_F - n_C)$, of the liquid, where n_F is the RI at λ_F, Fraunhöfer spectral F line (456 nm), and n_C is the RI at λ_C, Fraunhöfer spectral C line (656 nm);
Δ^S = the dispersion coefficient, $(n_F - n_C)$, of the solid;
k_D = a coefficient related to λ_0 and Fraunhöfer F, D, and C wavelengths in accordance with Hartmann dispersion relationship (Bloss 1981), which is equal to

$$[(\lambda_0 - 200)^{-1} - (\lambda_D - 200)^{-1}]/(\lambda_F - 200)^{-1} - (\lambda_C - 200)^{-1}] \text{ or}$$
$$[(\lambda_0 - 200)^{-1} - 0.002571]/0.001304.$$

It should be emphasized that Equation 1 is based on the dispersion coefficients of both the immersion oils and asbestos minerals. Almost every environmental laboratory in the U.S. uses immersion oils manufactured by Cargille Laboratories. Because the manufacturer has adopted fixed formulations for its products, the dispersion coefficients for the oils used in asbestos identification (Series E for 1.500 to 1.640 and Series B for 1.642 to 1.700) remain unchanged over the years.

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Table 1 lists typical RI values of the six regulated asbestos minerals. Although the absolute RI values of the same type of asbestos minerals from different localities and environments may vary in a significant range, their dispersion coefficients ($n_F - n_C$) generally remain constant within experimental errors. This is because any factor that increases (or decreases) n_D will also increase (or decrease) n_F and n_C at the same time, resulting in an unchanged ($n_F - n_C$) or an ($n_F - n_C$) with negligible change. For an asbestos mineral immersed in an immersion oil, both n_D^L and Δ^L are known for the oil and Δ^S is known for the asbestos; n_D^S is then directly related to k_D or λ_0.

THE OPERATION PROCEDURE

Preliminary identification of suspected fibrous components by stereo microscopy

Preliminary identification of suspected asbestos components is done by studying the material under stereomicroscope. Grayish to whitish curly fiber bundles are usually chrysotile, which comprises more than 95% of asbestos found in building materials. Straight fibers are usually amphiboles, among which only amosite and crocidolite are commonly present in building materials. Crocidolite has a dark blue color whereas amosite is generally gray to light brown. Tremolite, actinolite, and anthophyllite are rarely seen in building materials. In most cases, experienced analysts can reach a fairly accurate preliminary identification of the fibrous components that are suspected to be asbestos minerals through stereomicroscope examination.

Preparation of immersion oil mounts

The suspected asbestos fiber should be mounted in the oil specified in Table 1. If more than one type of asbestos is found and different oils are needed, prepare separate mounts using the corresponding oils. In the RI range of 1.500 to 1.640, Cargille offers two series of oils: Series A (normal dispersion) and Series E (high dispersion). For dispersion staining applications, Series E is exclusively used in asbestos identification because the greater the ($\Delta^L - \Delta^S$) value, the more distinctive the CSDS color. The procedure described in this paper is based on Series E oil for oils between 1.500 and 1.640. For oils above 1.640, Series B is used because it is the only series manufactured by Cargille Laboratories.

Confirmation of the preliminary identification of asbestos species by polarized light microscopy (PLM)

The six asbestos minerals can be easily identified under a polarized light microscope by examining their color, pleochroism, dispersion staining colors, birefringence, extinction behaviors, and elongation sign (McCrone 1987). The current regulations do not require the differentiation of tremolite from actinolite by PLM. They can be reported to be tremolite, actinolite or tremolite/actinolite.

Observation of the CSDS (central stop dispersion staining) colors of asbestos fibers

The CS mode is preferred in asbestos RI determination because it produces vivid DS colors against a dark background. By aligning a confirmed asbestos fiber's n_α, which is perpendicular to fiber elongation axis for chrysotile and amosite or parallel to fiber elongation axis for crocidolite, or n_γ, which is parallel to fiber elongation axis for chrysotile and amosite or perpendicular to fiber elongation axis for crocidolite, parallel to the vibration direction of polarizer, the respective CSDS colors can be easily observed.

Because it is impossible to obtain interference figures on fine fibrous tremolite, actinolite, and anthophyllite, statistical observation is used to estimate n_α and n_γ. To estimate n_α, analysts randomly select a fiber, rotate it to the extinction position when the fiber elongation axis is nearly parallel (tremolite or actinolite) or exactly parallel (anthophyllite) to the N-S cross hair (assuming the vibration direction of polarizer is E-W) and observe its CSDS color. After 10 to 20 fibers are examined in this way, the fiber with the longest λ_0 is assumed to exhibit the RI value closest to n_α. To estimate n_γ, 10 to 20 fibers are examined at the extinction position when the fiber elongation axis is nearly parallel (tremolite or actinolite) or exactly parallel (anthophyllite) to the E-W cross hair; the fiber with the shortest λ_0 is then assumed to exhibit the RI value closest to n_γ.

Conversion of an observed CSDS color to the corresponding matching wavelength λ_0

By referring to Table 2 (McCrone 1987), the observed CSDS color can be quickly converted into the corresponding λ_0 by direct match or interpolation based on the hue, saturation, and intensity of the observed CSDS color. As in the case of Becke line method, with proper training and sufficient practice, an analyst's ability to estimate λ_0 from CSDS colors can ensure the 0.005 to 0.010 accuracy required by the regulatory agencies. To further improve the accuracy of λ_0 estimation, analysts should calibrate the particular polarized light microscope used in routine analysis by examining standard reference materials whose n_α and n_γ are accurately known.

TABLE 1. Refractive indices and dispersion coefficients of six regulated asbestos minerals

Mineral		n_F	n_D	n_C	$n_F - n_C$	Oil used*	References
chrysotile	n_α	1.5563	1.5490	1.5456	0.0107	1.550 (E)	NIST SRM† 1866
	n_γ	1.5649	1.5560	1.5530	0.0119		
grunerite (amosite)	n_α	1.6931	1.6790	1.6734	0.0197	1.680 (B)	NIST SRM 1866
	n_γ	1.7156	1.7010	1.6951	0.0205		
riebeckite (crocidolite)	n_α	1.7132	1.7015	1.6971	0.0161	1.700 (B)	McCrone (1987)
	n_γ	1.7206	1.7072	1.7032	0.0174		
tremolite	n_α	1.6128	1.6063	1.6036	0.0092	1.620 (E)	NIST SRM 1867
	n_β	1.6299	1.6230	1.6201	0.0098		
	n_γ	1.6423	1.6343	1.6310	0.0113		
actinolite	n_α	1.6201	1.6126	1.6095	0.0106	1.625 (E)	NIST SRM 1867
	n_β	1.6369	1.6288	1.6254	0.0115		
	n_γ	1.6485	1.6393	1.6355	0.0130		
anthophyllite	n_α	1.6227	1.6148	1.6116	0.0111	1.625 (E)	NIST SRM 1867
	n_β	1.6350	1.6273	1.6241	0.0109		
	n_γ	1.6449	1.6362	1.6326	0.0123		

* The letter in the parentheses following the numerical values of immersion oils denotes the Series ID used by the manufacturer Cargille Laboratories Inc. See text for details.

† SRM: standard reference materials issued by NIST, Gaithersburg, Maryland.

TABLE 2. Conversion of dispersion staining colors to the corresponding λ_0 (McCrone 1987)

Matching Wavelength (nm)	Particle edge colors*		Becke line colors†	
	Annular stop‡	Central stop§	Particle	Liquid
<340	black violet	white	white	–
400	dark violet	pale yellow	pale yellow	–
430	violet	yellow	pale yellow	–
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	–	blue-green
700	dark red-brown	pale blue-green	–	pale blue-green
1500	black-brown	very pale blue-green	–	very pale blue-green

* In focus.
† On focusing up.
‡ Observed on a brightfield.
§ Observed on a darkfield.

TABLE 3. Conversion of the matching wavelength λ_0 to the corresponding RI values

Mineral	Chrysotile	Amosite	Crocidolite	Tremolite			Actinolite or Anthophyllite		
Oil $n_D^{25°C}$	1.550	1.680	1.700	1.620	1.610	1.635	1.625	1.610	1.635
Oil Series	E	B	B	E	E	E	E	E	E
RI	n_α or n_γ	n_α or n_γ	n_α or n_γ	n_α or n_γ	n_α	n_γ	n_α or n_γ	n_α	n_γ
λ_0 (nm)									
400	1.579	1.707	1.738	1.651	1.640	1.668	1.654	1.636	1.666
420	1.573	1.702	1.731	1.645	1.634	1.662	1.649	1.632	1.660
440	1.569	1.698	1.725	1.640	1.629	1.657	1.644	1.627	1.655
460	1.565	1.694	1.720	1.636	1.626	1.652	1.640	1.624	1.651
480	1.562	1.691	1.716	1.633	1.622	1.649	1.637	1.621	1.648
500	1.559	1.689	1.712	1.630	1.619	1.645	1.634	1.618	1.645
520	1.557	1.686	1.709	1.627	1.617	1.643	1.632	1.616	1.642
540	1.554	1.684	1.706	1.625	1.615	1.640	1.629	1.614	1.640
560	1.552	1.682	1.703	1.623	1.613	1.638	1.627	1.612	1.638
580	1.551	1.681	1.701	1.621	1.611	1.636	1.626	1.611	1.636
589	1.550	1.680	1.700	1.620	1.610	1.635	1.625	1.610	1.635
600	1.549	1.679	1.699	1.619	1.609	1.634	1.624	1.609	1.634
620	1.548	1.678	1.697	1.618	1.608	1.632	1.623	1.608	1.633
640	1.546	1.677	1.695	1.616	1.606	1.631	1.621	1.607	1.631
660	1.545	1.676	1.694	1.615	1.605	1.630	1.620	1.606	1.630
680	1.544	1.675	1.692	1.614	1.604	1.628	1.619	1.605	1.629
700	1.543	1.674	1.691	1.613	1.603	1.627	1.618	1.604	1.628
720	1.542	1.673	1.690	1.612	1.602	1.626	1.617	1.603	1.627
740	1.542	1.672	1.689	1.611	1.601	1.625	1.616	1.602	1.626
760	1.541	1.671	1.688	1.610	1.600	1.624	1.616	1.601	1.625
780	1.540	1.670	1.687	1.609	1.600	1.623	1.615	1.601	1.624
800	1.539	1.670	1.686	1.609	1.599	1.623	1.614	1.600	1.624
850	1.538	1.668	1.684	1.607	1.597	1.621	1.613	1.599	1.622
900	1.537	1.667	1.682	1.606	1.596	1.619	1.611	1.598	1.621
1000	1.534	1.665	1.679	1.603	1.594	1.617	1.609	1.596	1.618
Δ^L	0.0267	0.0348	0.0370	0.0267	0.0251	0.0291	0.0275	0.0251	0.0291
Δ^S	0.0113	0.0201	0.0168	0.0103	0.0092	0.0113	0.0118	0.0109	0.0127
$\Delta^L - \Delta^S$	0.0154	0.0147	0.0203	0.0164	0.0159	0.0178	0.0157	0.0142	0.0164

Note: Temperature Correction: If oil temperature is not 25 °C, for every 2 °C decrease (increase) in temperature, add (subtract) 0.001 to (from) the listed values.

Conversion of λ_0 to the corresponding RI value

By referring to Table 3, which was calculated from Equation 1, the λ_0 derived from the observed CSDS color can be quickly converted into the corresponding numerical RI value, n_D^S.

The RI values in Table 3 are calculated assuming the liquid or ambient temperature, T, at the time of measurement is 25 °C. If T is not 25 °C, temperature correction should be applied to n_D^L to obtain $n_D^{25°C}$ by using the following equation

$$n_D^{25°C} = n_D^L + (T - 25)\,\mathrm{d}n/\mathrm{d}t \qquad (2)$$

where dn/dt is the immersion oil's temperature coefficient, a negative value. For practical purposes, the quickest way to apply the temperature correction is to add (or subtract) 0.001 from the Table 3 reading for every 2 °C decrease (or increase) from 25 °C.

Because the Δ^S of actinolite is very close to that of anthophyllite, the two minerals are grouped together. For each asbestos mineral, both n_α and n_γ can be quickly determined with a reasonable accuracy (±0.005 to 0.010) in a single oil mount during routine analysis. However, when higher accuracy is desirable, e.g., when analyzing the Proficiency Testing samples administered by NVLAP, n_α and n_γ can be separately determined using two different oils as suggested in Table 3.

APPLICATIONS TO OTHER RI DETERMINATION TASKS

Although this procedure was designed primarily for the operations of commercial environmental laboratories, its principle can be readily applied to other tasks requiring the determination or screening of the refractive indices of solid particles to improve the efficiency and accuracy of the measurement, especially in the case of completely unknown materials. For details, please see Su (1998). It can also be applied to the determination of the composition of olivine, orthopyroxene, augite, and plagioclase (Su 1994).

APPLICATION OF THE METHOD TO UNKNOWN MATERIALS

The following is an example of measuring the RI of a pure unknown chemical by an operator with minimum experience of dispersion staining technique.

To test how quickly an operator with minimum experience could determine the refractive index of an unknown isotropic material, I gave some powders of NH_4Cl to a technician with one-hour training in comparing refractive index of solid particles against the surrounding immersion oil using both the Becke line and the central stop dispersion staining methods. I told him to start with any oil within the range available in our laboratory, which is 1.400 to 1.750. He chose 1.550 (Series E) because it happened to be on the counter. The colorless powder of NH_4Cl showed high relief and a bright white Becke line that moved into the grain when the distance between the slide and the 10× objective was increased. By checking Table 2, he decided that the matching wavelength λ_0 was approximately 300 nm. Then I told him to use Equation 1 to calculate the RI of this material based on the observed 300 nm matching wavelength, $n_D^L = 1.550$, $\Delta^L = 0.0267$, $k_D = 5.7$, which was calculated from $\lambda_0 = 300$, and $\Delta^S = 0.0084$, which was calculated from the following empirical equation:

$$\log(n_F - n_C) = 5.9 \log n_D - 3.2. \tag{3}$$

Equation 3 was derived by Stoiber and Morse (1994) based on rock-forming mineral data in Tröger (1979). Since the calculated n_D^S was 1.654, the operator chose 1.650 to prepare the second mount of the unknown powder, which showed pale blue-green CSDS color or $\lambda_0 = 700$ nm. Based on the results of the 1.650 preparation, n_D^S was calculated to be 1.542. The operator chose 1.540 oil for the third preparation and the unknown particles showed a deep blue CSDS color, indicating an exact match between the solid and the liquid at 589 nm, resulting in $n_D^S = 1.540$. Since the temperature of oils were in equilibrium with room temperature 21 °C and the temperature coefficient of 1.550 (E) oil is $-0.00042/°C$, n_D^S was then calculated to be 1.642. According to Winchell and Winchell (1964), NH_4Cl (salammoniac) has $n_D = 1.6426$.

It should be emphasized that the analyst had only very minimal training and only three oils were used to determine the RI of the unknown isotropic material to an accuracy of ±0.001. My experience is that it usually takes no more than three oils to determine the RI of an unknown material to ±0.003 to 0.005.

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