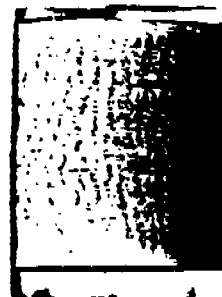


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Identification of Asbestos Fibers by Microscopical Dispersion Staining*

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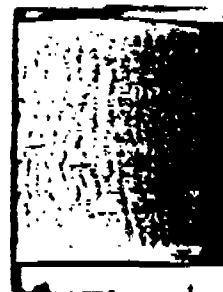
McCrone Associates, Inc., Chicago, Illinois, U.S.A.

Abstract

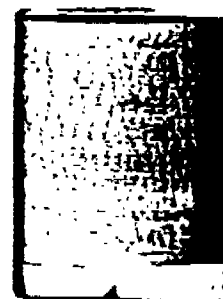
The Cherkasov focal screening dispersion staining procedure has been successfully applied to the identification of asbestos. The various types of asbestos can be differentiated by noting the refractive index of the Cargille liquid giving matching wavelengths in the region near 550 nm. It is not necessary to use polarized light although the much more definitive data obtained with polarized light may eventually permit identification of the mine from which each asbestos came.

Although too few samples of amosite and crocidolite have been studied, it appears that these two types can be differentiated by dispersion staining. In Cargille liquid $n_D^{25} 1.680$ the central stop without polars will show colors in the blue magenta region ($\lambda_0 = 550-650$ nm) for amosite and in the golden yellow region ($\lambda_0 = 400-500$ nm) for crocidolite. Furthermore, with polarized light crocidolite will show lower birefringence than amosite; in terms of λ_0 difference in a given liquid amosite will show 160-190 nm and crocidolite 120-140 nm. Finally, the higher value of λ_0 is observed for the vibration direction parallel to the length for crocidolite (three samples) but perpendicular to the length for amosite (two samples).

* Presented at INTER/MICRO-69, London, England.



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Through publications¹⁻³ and current research, the pneumoconiotic (lung hardening) and frequently cancer-producing hazards of asbestos are finally fully acknowledged. Hence, there is need of a method of determining qualitatively and quantitatively environmental pollution by respirable asbestos dust (1-7 μ m).

Asbestos is a fibrous form of silicate rock. There are six types: chrysotile, amosite, crocidolite, actinolite, tremolite and anthophyllite; only the first three are of commercial importance. All are amphiboles (minerals with chains of silica tetrahedra as their basic structure) except chrysotile which is a serpentine (mineral made up of layers of silica tetrahedra).

The property of asbestos that best lends itself to identification is refractive index. The fibers are so fine that electron microscopy would be necessary if morphology were to be used. There are no dependable differences in absorption color (except possibly for crocidolite) nor specific gravity (other than chrysotile) (Table I). If we wish to use chemical composition for identification of single asbestos fibers, we require highly sophisticated instruments and techniques—the electron microscope or an electron (or ion) microprobe. X-ray diffraction requires considerable sample and is not sensitive to small percentages in any sample (*i.e.*, < 5-10%). Very small asbestos fibers can, however, be identified simply and quickly using dispersion staining.

Experimental

The McCrone dispersion staining objective, based on the focal screening method of Cherkasov⁴, was used in this study of asbestos (Figure 2).

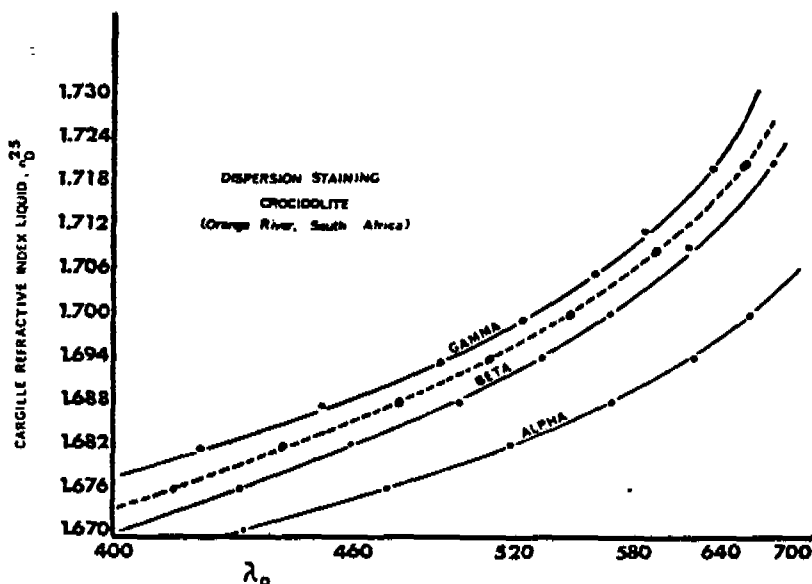


Figure 1. Dispersion staining data for a typical sample of crocidolite.

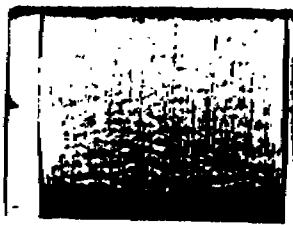
TABLE 1
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SERPENTINES		AMPHIBOLES				
	Chrysotile	Actinolite	Tremolite	Anthophyllite	Amosite	Crocidolite
Composition	$3\text{Mg}0.2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$	$2\text{Ca}0.4\text{Mg}0. \text{Fe}0.8\text{SiO}_2 \cdot \text{H}_2\text{O}$	$2\text{Ca}0.5\text{Mg}0. 8\text{SiO}_2 \cdot \text{H}_2\text{O}$	$7\text{Mg}0.8\text{SiO}_2 \cdot \text{H}_2\text{O}$	$5.5\text{Fe}0. 1.5\text{Mg}0. 8\text{SiO}_2 \cdot 11\text{H}_2\text{O}$	$\text{Na}_2\text{O} \cdot \text{Fe}_2\text{O}_3 \cdot 3\text{Fe}0.8\text{SiO}_2 \cdot 11\text{H}_2\text{O}$
Spec. Grav.	2.36-2.5	3.03-3.5	2.9-3.2	2.85-3.4+	2.6-3.0	3.0-3.45
Cryst. Syst.	Monoclinic	Monoclinic	Monoclinic	Orthorhombic	Monoclinic	Monoclinic
Extinction	$\gamma \wedge L^* = 0^\circ$	$\gamma \wedge L = 10-15^\circ$	$\gamma \wedge L = 10-21^\circ$	$\gamma \wedge L = 0^\circ$	$\gamma \wedge L = 14-21^\circ$	$\alpha \wedge L = 3-15^\circ$
Sign (elong.)	+	+	+	+	+	—

*L = long direction of fibers

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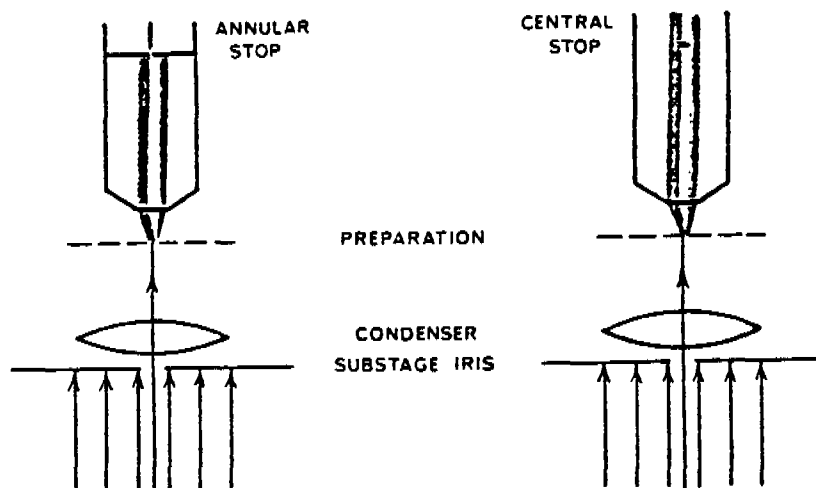
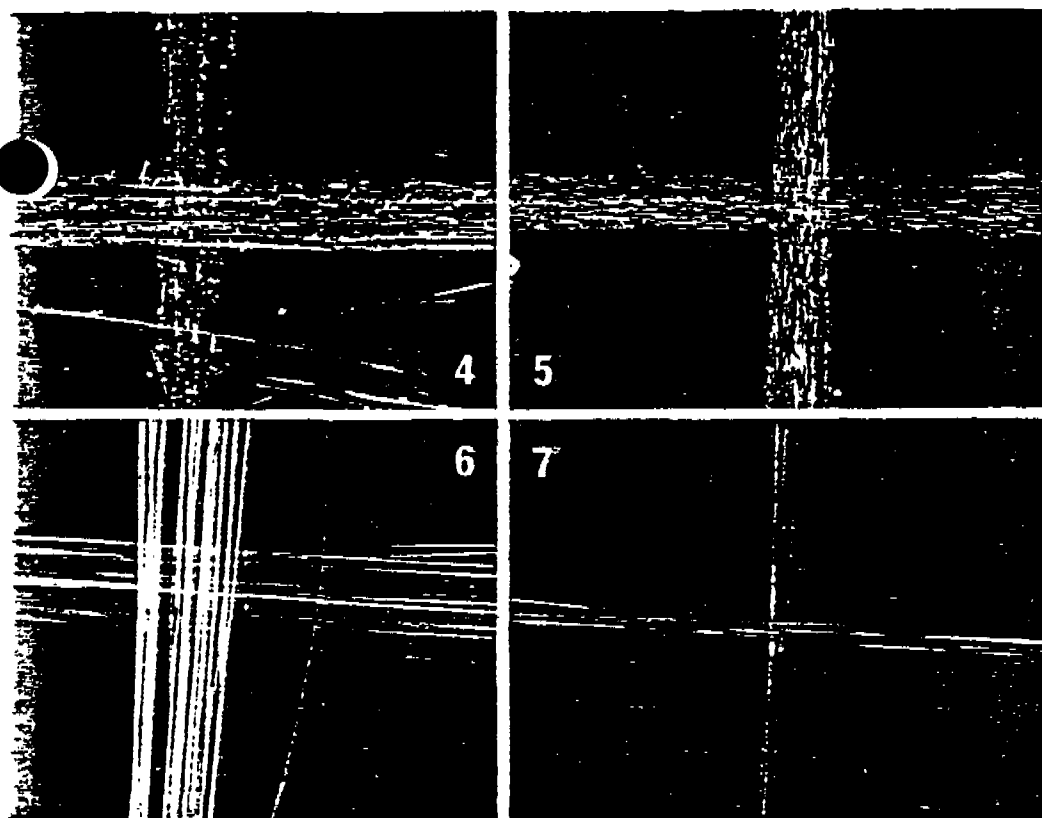


Figure 2. The arrangement for focal screening: the annular stop permits passage of the matching wavelength and the central stop white light minus the matching wavelength.



Figures 4-7. Central stop colors for chrysotile (Figure 4), anthophyllite (5), amosite (6) and crocidolite (7) each with the appropriate Cargille refractive index liquid, 1.560 (Figure 4), 1.610 (Figure 5), 1.670 (Figure 6), 1.700 (Figure 7). The vibration direction of the polar is east-west in each figure.

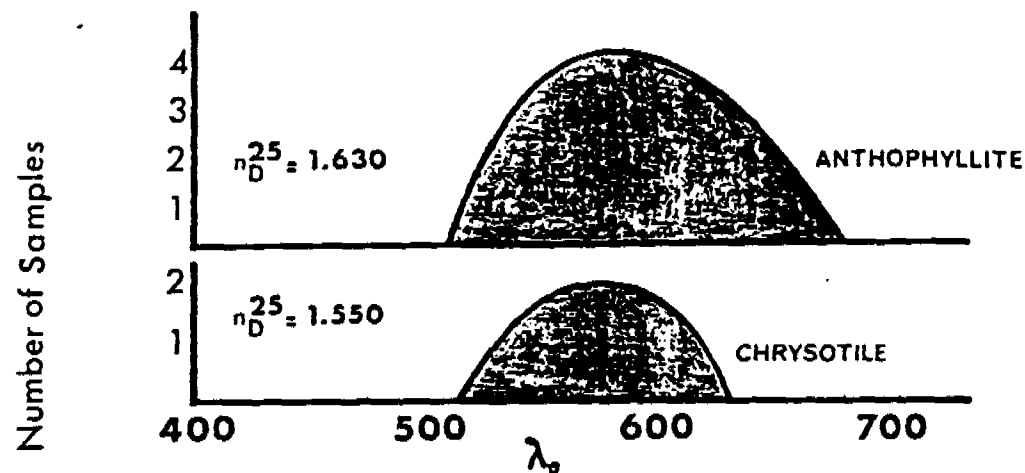
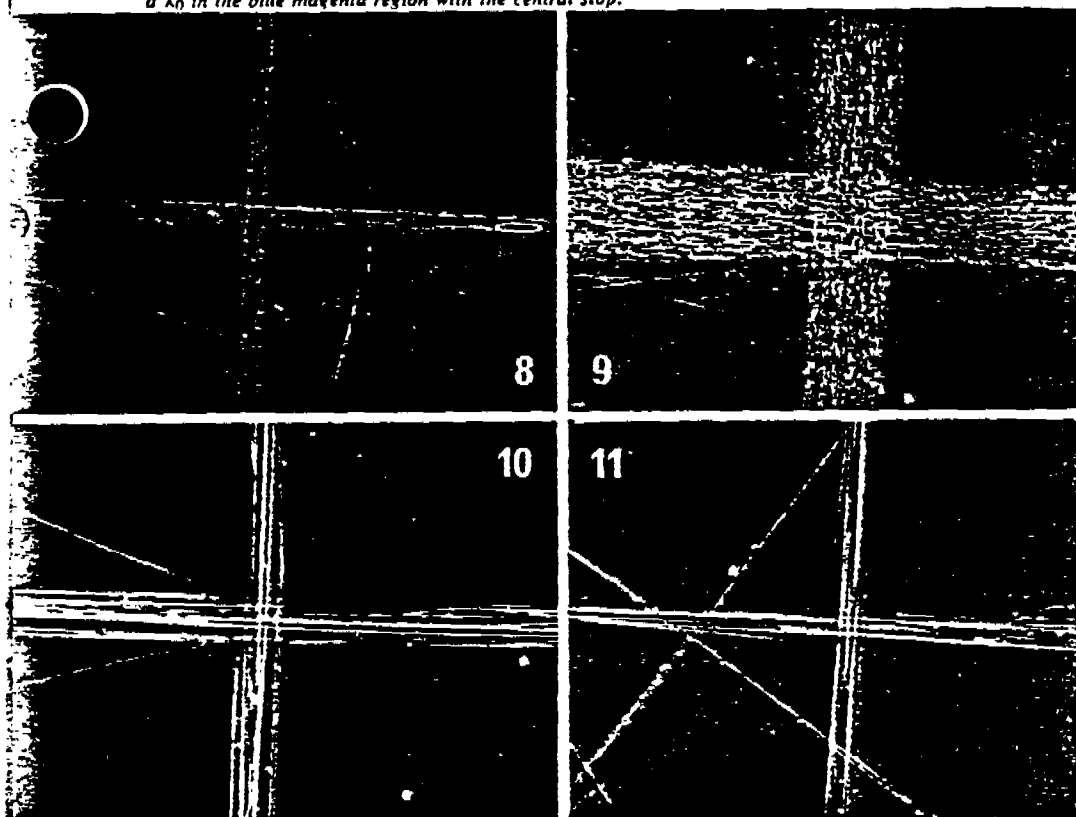


Figure 3. The range of matching wavelengths shown by anthophyllite and chrysotile with unpolarized light. The Cargille liquid was chosen so that most samples of each asbestos show a λ_0 in the blue magenta region with the central stop.



Figures 8-11. Central stop colors for chrysotile (Figure 8), anthophyllite (9), amosite (10) and crocidolite (11) in the same Cargille liquids as Figures 4-7 respectively. Unpolarized light was used for this series.

TABLE II
Wavelengths Corresponding to Observed Dispersion Staining Colors

Matching Wavelength λ_0 , nm	Colors observed	
	Central stop	Annular stop
<420	light yellow	dark-blue
420-440	yellow	blue-violet
440-470	golden yellow	blue
470-500	golden magenta	blue-green
500-540	reddish-magenta	green
540-580	magenta	yellow-green
580-610	blue-magenta	yellow
610-640	blue	orange
640-680	blue-green	orange-red
>680	blue-white	brownish-red

Though phase contrast and dark-ground illumination are often used for dispersion staining, the data obtained by these methods are empirical. Pure colors are not obtained and the mixture of colors observed is dependent upon the particular optical system used. With the Cherkasov procedures, axial illumination is utilized and hence essentially pure reproducible colors are obtained.

A graph of matching wavelength, λ_0 , was determined as a function of immersion liquid refractive index for each of 26 asbestos samples. The central stop in conjunction with Table II was found to be most useful in estimating λ_0 because it provides better resolution and contrast; hence higher sensitivity for small single fibers. The procedure involved:

1. mounting each asbestos successively in those Cargille refractive index liquids imparting dispersion colors to it.
2. noting λ_0 for the sample using both polarized and unpolarized light.
 - (a) fibers oriented parallel to the vibration direction of the polar show α (crocidolite) or γ (all other asbestos fibers).
 - (b) fibers oriented crosswise show β and γ (crocidolite) or α and β (all other asbestos fibers). Random orientations under these conditions show crocidolite in $\beta \leq n \leq \gamma$ positions and all other asbestos fibers in $\beta \geq n \geq \alpha$ positions. The two extreme colors noted for crosswise vibrations are designated α (highest λ_0) and β (lowest λ_0) for all asbestos fibers other than crocidolite or β (highest λ_0) and α (lowest λ_0) for crocidolite.

The small oblique extinction angle often observed with asbestos causes no significant variation in λ_0 . Figure 1 shows the data for one sample of crocidolite determined in this way.

When the polarizer is removed, asbestos fibers show nominally a single color that is a mixture of all the wavelengths observed during rotation of the polarizer. In practice, due to polarization by reflection

from the microscope mirror, there is a slight change in color when the stage is rotated. All our data were taken with the fibers normal to the mirror reflection vibration direction.

Figures 4-7 show the dispersion staining colors of representative samples of chrysotile, anthophyllite, amosite and crocidolite respectively. Each is shown in the various orientations (relative to the polarizer vibration direction) necessary for determination of α , β and γ . Careful study of these figures may indicate the simplified procedure used to orient the fibers at precise 90 degree angles to each other.

To determine if the simpler application of dispersion staining without a polarizer is adequate for identifying asbestos, the fields of view of Figures 4-7 were also taken with no polar (Figures 8-11). The λ_0 curves obtained without polar were plotted in a single graph (Figure 12). The bunching of the curves of each different type of asbestos indicates that for identification purposes a liquid can be chosen that will impart a characteristic color to one species of asbestos only. A polarizing microscope is not, therefore, required. However, we can anticipate that with additional samples of crocidolite and amosite there might be some overlap. For this reason, the distinction between the signs of elongation (Figures 6 and 7) and the crocidolite blue absorption color are mentioned as special aids for differentiation. In this case a polarizer is useful.

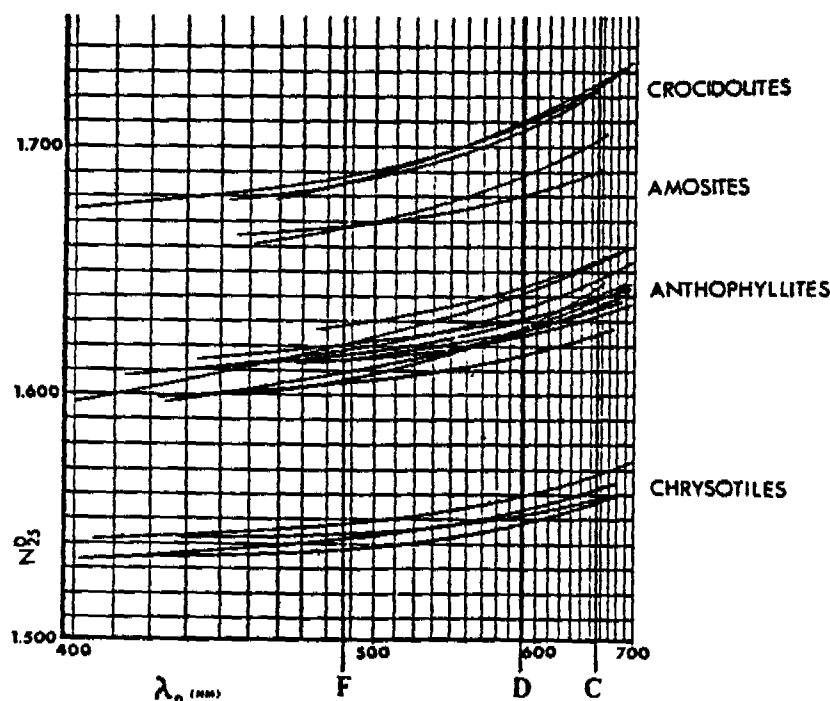


Figure 12. Matching wavelengths shown by different samples of four types of asbestos when mounted in the Cargille refractive index liquids shown and observed with unpolarized light.

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The characteristic color will vary somewhat for different samples of the same type of asbestos. So, the problem arose of determining the ranges of colors for each asbestos in the chosen liquid (the liquid in which most samples of any asbestos type give a color of wavelength 580-620 nanometers). To determine this range of colors for any type of asbestos, the matching wavelengths observed in the chosen liquid are plotted against the number of different samples showing that λ_0 . The area under the resulting curve (colored in Figure 3 as per Table II) shows all the colors shown by our samples of anthophyllite and chrysotile. Too few samples of amosite or crocidolite have been studied to justify their inclusion in Figure 3.

Conclusion

The limited results obtained indicate that asbestos can be identified by dispersion staining. More work on many more samples will establish the full range of λ_0 values for each asbestos. Relating λ_0 data to asbestos source may then also pinpoint the mine source of unknown asbestos samples.

Acknowledgement

The authors are grateful to Dr. Bertram G. Woodland, Curator of Igneous and Metamorphic Petrology of the Field Museum of Natural History in Chicago for the samples of asbestos from various sources.

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APPENDIX

Dispersion Staining Data, $n_D^{25^\circ\text{C}}$ of Cargille refractive index liquid

Chrysotiles

Sample	Orientation	Matching wavelength, λ_D , nm				
		450	486(F)	520	590(D)	656(C)
Thetford Mines, Quebec	γ	1.539	1.542	1.546	1.553	1.559
	δ	1.535	1.539	1.543	1.550	1.556
	β	1.533	1.539	1.543	1.547	1.556
	α	1.528	1.531	1.534	1.541	1.546
Eden, Vermont E19048**	γ	1.549	1.553	1.558	1.566	1.574
	δ	1.543	1.547	1.551	1.559	1.566
	β	1.543	1.547	1.551	1.559	1.566
	α	1.534	1.539	1.543	1.550	1.557
Thetford Mines, Quebec E20436	γ	1.545	1.549	1.552	1.559	1.565
	δ	1.540	1.546	1.547	1.553	1.558
	β	1.538	1.541	1.544	1.550	1.554
	α	1.536	1.539	1.542	1.548	1.552
Thetford Mines, Quebec E12185	γ	1.543	1.547	1.550	1.556	1.563
	δ	1.540	1.543	1.546	1.552	1.558
	β	1.536	1.540	1.543	1.549	1.555
	α	1.531	1.534	1.537	1.543	1.548
King's Mine, Quebec	γ	1.543	1.547	1.551	1.559	1.566
	δ	1.539	1.543	1.547	1.554	1.561
	β	1.535	1.539	1.543	1.550	1.557
	α	1.529	1.532	1.535	1.542	1.547
Barquisimento, Venezuela	γ	1.536	1.541	1.546	1.556	1.566
	δ	1.530	1.537	1.541	1.550	1.559
	β	1.530	1.534	1.539	1.547	1.555
	α	1.524	1.528	1.532	1.540	1.548

* no polars were used.

** sample identification number of the Field Museum of Natural History, Chicago, Illinois, U.S.A.

Anthophyllites

Minas Gerais, Brazil E16696	γ	1.604	1.613	1.620	1.633	1.645
	δ	1.602	1.608	1.614	1.628	1.638
	β	1.599	1.606	1.612	1.625	1.636
	α	1.582	1.588	1.594	1.606	1.616
Maryland E3713	γ	1.617	1.623	1.630	1.642	1.658
	δ	1.613	1.618	1.624	1.636	1.649
	β	1.609	1.614	1.619	1.630	1.642
	α	1.599	1.604	1.609	1.620	1.630
New Mexico E3709	γ	1.620	1.625	1.629	1.638	1.648
	δ	1.616	1.621	1.625	1.634	1.642
	β	1.609	1.612	1.616	1.620	1.628
	α	1.601	1.604	1.607	1.613	1.618
Macon Co., N. Carolina E3700	γ	1.614	1.618	1.623	1.632	1.641
	δ	1.610	1.614	1.619	1.627	1.635
	β	1.606	1.610	1.614	1.622	1.629
	α	1.594	1.597	1.604	1.607	1.613

APPENDIX (Continued)

Lincoln Co., Nevada E1101	γ	1.609	1.611	1.616	1.623	1.630
	δ	1.602	1.605	1.609	1.615	1.621
	β	1.596	1.599	1.602	1.609	1.615
	α	1.589	1.592	1.595	1.600	1.606
Rio Grande de Sul, Brazil E14494	γ	1.610	1.615	1.620	1.630	1.641
	δ	1.605	1.610	1.615	1.624	1.635
	β	1.603	1.607	1.611	1.620	1.628
	α	1.596	1.600	1.604	1.611	1.618
California E14464	γ	1.619	1.624	1.630	1.642	1.656
	δ	1.614	1.619	1.624	1.635	1.646
	β	1.612	1.616	1.622	1.632	1.642
	α	1.601	1.605	1.610	1.618	1.627
Pine Mt., Georgia	γ	1.607	1.613	1.619	1.632	1.647
	δ	1.602	1.608	1.614	1.625	1.638
	β	1.597	1.602	1.607	1.617	1.628
	α	1.589	1.593	1.597	1.606	1.614
Encampment, Wyoming	γ	1.619	1.620	1.626	1.630	1.638
	δ	1.613	1.616	1.619	1.625	1.630
	β	1.609	1.612	1.615	1.621	1.626
	α	1.598	1.600	1.603	1.607	1.611
Bedford, Virginia E12790	γ	1.614	1.619	1.622	1.634	1.644
	δ	1.609	1.613	1.618	1.628	1.638
	β	1.606	1.610	1.615	1.624	1.633
	α	1.599	1.603	1.607	1.614	1.622
Crocidolites						
Orange River, South Africa	γ	1.688	1.694	1.700	1.712	1.726
	δ	1.684	1.690	1.696	1.707	1.721
	β	1.680	1.686	1.692	1.702	1.713
	α	1.674	1.678	1.682	1.690	1.698
Westerburg, South Africa	γ	1.686	1.692	1.697	1.708	1.718
	δ	1.682	1.687	1.692	1.701	1.710
	β	1.679	1.683	1.688	1.697	1.706
	α	1.674	1.678	1.683	1.691	1.699
South Africa	γ	1.691	1.696	1.701	1.712	1.723
	δ	1.685	1.690	1.695	1.705	1.714
	β	1.680	1.685	1.690	1.700	1.710
	α	1.674	1.679	1.683	1.692	1.700
Amosites						
Lydenburg Dist., Transvaal	γ	1.676	1.682	1.687	1.699	1.712
	δ	1.665	1.670	1.676	1.687	1.698
	β	1.658	1.663	1.668	1.678	1.689
	α	1.652	1.657	1.662	1.672	1.682
Penge, Transvaal	γ	1.672	1.677	1.682	1.692	1.701
	δ	1.665	1.669	1.674	1.682	1.691
	β	1.660	1.664	1.668	1.676	1.684
	α	1.653	1.657	1.661	1.669	1.676