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1984/28. Asbestos recognition and determination of asbestos varieties by optical means

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Abstract

Concern about the possible health hazards of asbestos has resulted in legislation limiting the permitted concentration of fibres in the air in the work place and the home. Because the Threshold Limiting Value (TLV) varies from 0.1 fibre/ml of air for crocidolite (blue asbestos) (and amosite from 1984) to one fibre/ml of air for chrysolite, it is important to identify the type of fibres involved as well as their concentration. A description is given of the optical techniques adopted by the Tasmania Department of Mines. These techniques include refractive index determination, dispersion staining, birefringence and pleochroism.

INTRODUCTION

Asbestos has a number of useful properties which give it a wide variety of applications. It is fibrous with a high tensile strength and low weight to volume ratio. It has a high resistance to heat, electricity, abrasion and attack by chemicals. It also adheres well to cement and is generally inexpensive compared to alternatives. The main uses of asbestos, at present, are in the manufacture of asbestos-cement sheets and moulded goods (used, particularly, in construction), asbestos-cement pipes, friction materials such as brake and clutch linings which can contain up to 90% asbestos, vinyl-asbestos floor materials, textiles, filters used in the production of beverages such as wine, rubber, plastics, paints, adhesives etc., felts, gaskets and insulating materials.

The term 'asbestos' is applied to the fibrous forms of several hydrated silicate minerals belonging to the serpentine and amphibole groups. Only two types of asbestos are used commercially in Australia at present; chrysotile (approximately 85%) and amosite (15%). Crocidolite (blue asbestos) has been used in the past and may be found in some products and buildings.

Exposure to asbestos has been linked to a number of diseases. Asbestosis is a scarring of lung tissue by asbestos fibres in the alveoli and bronchioles, which may cause breathlessness, incapacity or death, depending on the extent of exposure. A study of 17,800 men exposed to asbestos found that asbestosis killed 0.1-0.91% per year (Selikoff, 1982).

The incidence of lung cancer in smokers increases linearly with increased doses of asbestos dust. Asbestos exposure appears to have little effect on susceptibility to lung cancer in non-smokers. In a study of 38,904 asbestos trade workers the death rate due to lung cancer was 18.3% compared to ~6% of males in the general population (Ross, 1981). Mesothelioma, cancer of the lining of the lung or gut, appears to follow relatively low exposure to asbestos, although the incidence increases with exposure and is higher after exposure to crocidolite than chrysotile. Six per cent of the 38,904 asbestos trade workers died of mesothelioma compared to ~0.03% of the general public (Ross, 1981). Asbestos inhalation is also associated with cancer of the larynx, bowel and peritoneal cancer. Generally these diseases do not appear until many years after first exposure to

asbestos. People most at risk are asbestos miners, workers in industries which use asbestos, people in towns near asbestos mines, and the families of asbestos workers. There may be a threshold value below which asbestos has no carcinogenic effect but there is conflicting evidence concerning this and it has even been suggested that the small amount of asbestos fibre released by worn brake linings (<8 fibres/litre of city air) is responsible for the increased incidence of lung cancer in urban populations (Lynch, 1968).

The asbestos problem is difficult to resolve because of uncertainty about safe levels and degree of risk, conflict between the usefulness of asbestos, commercial interests and its risk to health, and the difficulty in finding satisfactory safe substitutes; for example, it is widely used in cheap, efficient construction materials for housing in the Third World and in safety products such as firemen's suits.

Although it has been suspected since the early years of this century that asbestos dust may be a health hazard, it was not until the 1940s and 1950s that there was documented evidence of the link between asbestos and various sorts of cancer. In 1965 a committee was established in Britain to examine the possible hazards of asbestos and it proposed safety standards of two fibres/ml of air for chrysotile. Four years later, in 1969, regulations in the United Kingdom set the acceptable level of crocidolite at 0.2 fibres/ml. The European Commission set the maximum concentration allowable for crocidolite at 0.2 fibres/ml and one fibre/ml for other types of asbestos. In 1978 the National Institute for Health in the U.S.A. estimated that 13-18% of the total cancer mortality over the next thirty years would be due to occupational exposure to asbestos. Although this figure was criticised (most sources estimate that asbestos accounts for only 1-3% of U.S. cancer deaths) it stimulated concern. The importation, mining and use of crocidolite are prohibited in Australia (National Health and Medical Research Council, 1982) and at present the TLV (Threshold Limit Value) for crocidolite is 0.1 fibre/ml of air and for amosite and chrysotile 1.0 fibre/ml. As some authorities consider that the danger from amosite is greater than that of chrysotile, the TLV for amosite will be changed to 0.1 fibres/ml of air from 30 June 1984 (pers. comm., Department of Labour and Industry). Therefore it is important not only to be able to tell if a fibre sample contains asbestos but also to distinguish between the different types.

Levels of airborne asbestos dust are estimated using the membrane filter method. The sample is collected by drawing a measured amount of air through a membrane filter. Once the sample has been collected the filter is made transparent by the use of chemical reagents such as acetone and glycerol triacetate. Fibres are examined under a phase contrast microscope and particles longer than 5 μm and not wider than 3 μm are counted. The sampling technique and recommended counting procedures are specified in the National Health and Medical Research Council Report of October 1976.

ASBESTOS SOURCES

The U.S.S.R. and U.S.A. produce most of the world's chrysotile (see Table 1). South Africa is the main source of chrysotile and amosite.

The only Australian producer is the Baryulgil mine in N.S.W., which produces chrysotile. This mine is currently the subject of an enquiry by a House of Representatives Standing Committee on Aboriginal Affairs which is investigating the effects of asbestos mining on the largely aboriginal community of Baryulgil.

Table 1. WORLD PRODUCTION OF ASBESTOS (tonnes) (after CLARKE, 1982).

Country	1975	1976	1977	1978	1979	1980
Italy	146,984	164,788	149,327	135,401	143,931	157,794
Bulgaria	-	300	7,500	9,600	8,800	na
Czechoslovakia	-	-	-	575	564	617
Soviet Union	1,895,000	1,850,000	1,900,000	1,945,000	2,020,000	2,150,000
Turkey	15,496	9,941	3,975	13,380	38,967	8,724
Yugoslavia	12,336	12,830	9,036	10,304	9,959	12,106
Egypt	479	1,018	478	349	350	na
South Africa						
Amosite	88,411	78,893	66,983	40,526	39,058	57,646
Anthophyllite	1,912	1,506	550	-	-	-
Crocidolite	164,727	178,411	200,966	137,288	118,301	118,148
Chrysotile	99,660	111,025	111,575	79,511	91,828	106,940
Swaziland	37,610	39,327	38,046	36,951	34,294	32,833
Zimbabwe	251,542	281,455	273,194	248,861	2,589,891	250,949
Canada	1,055,667	1,536,091	1,517,360	1,421,808	1,430,614	1,202,511
USA (a)	89,497	104,873	92,256	93,097	93,354	80,079
Argentina	1,130	889	686	697	700	-
Brazil	73,978	92,703	92,773	122,815	138,457	170,000
Afghanistan	-	13,260	13,000	13,000	4,000	na
China	150,000	175,000	200,000	230,000	250,000	250,000
Cyprus	31,602	34,518	36,684	34,359	35,472	35,535
India	20,312	24,119	22,177	24,623	32,094	31,253
Japan	4,612	7,703	6,307	5,746	3,362	na
Republic of Korea	4,345	4,762	6,180	13,616	14,804	9,854
Taiwan	1,737	853	673	2,031	2,957	683
Australia	47,922	60,642	50,601	62,744	79,721	83,466

(a) Sold or used by producers

The main user of asbestos in Tasmania is the Goliath Portland Cement Co. Ltd. Imported chrysolite is used to produce asbestos cement sheets, marketed under the name "Flexiboard", and cement mouldings.

Asbestos is now banned from new public building projects in Tasmania and steps have been taken to seal or remove asbestos in exposed situations.

IDENTIFICATION OF ASBESTOS FIBRES

A preliminary optical test for fibre type is made with a hand lens or binocular microscope. The reaction of fibres to heat (*i.e.* burning match) and the presence of split ends are useful preliminary tests. Vegetable, animal and most synthetic fibres will char with heat, thin fibres of fibreglass will melt but asbestos tends to retain its shape, glows red with heat and only melts at the tips of very fine fibres, if at all. Asbestos fibres have characteristic split ends.

If more detailed identification of fibres are required, the identification chart in Figure 1 may be used and some useful optical properties are listed in Tables 2 and 3.

The methods available for asbestos characterisation are listed below:

(a) Dispersion staining

In the dispersion staining technique a substance is identified by obtaining characteristic colours from dispersion of white light under an optical microscope when the fibre is immersed in a liquid of high dispersion (for explanation, see *Dispersion* section).

(b) X-ray diffraction

X-ray diffraction has the advantage that it can identify samples discoloured by heat more readily than dispersion staining and can be automated. However XRD is more expensive and less easily adapted to very small samples (Dunn and Stewart, 1982).

(c) Scanning electron microscope or electron microprobe

This procedure is useful for samples which contain a high proportion of particles of <1 μm diameter or when accurate size distributions are required. It has the disadvantage of being relatively expensive.

(d) Infra-red spectra

This method is not as efficient as dispersion staining and also has difficulty with heat-affected samples.

It was decided to use optical techniques such as relief and dispersion staining with XRD and SEM as a back-up on the grounds of the lower unit cost of this method. The optical techniques necessary are described below:

Relief

A colourless object is only visible because light rays are refracted and reflected at the interface between it and its surrounding medium. If a transparent substance is immersed in a medium with exactly the same refractive index (RI), then it will be invisible. If the refractive indices of a particle and the medium it is immersed in are closely matched, then the edges of the particle will be very faintly outlined and it is said to have low relief. If there is a considerable difference between the

Table 2. OPTICAL PROPERTIES OF MAIN NON-ASBESTOS FIBRE TYPES (AFTER VAUGHAN, ROOKER AND LEGUEN, 1981).

Fibre	Bulk RI range*	Morphology	Extinction	Birefringence	Pleochroism	Colours with 1st order red plate	Length: fast or slow
Natural vegetable organic fibre	1.50-1.60	long, usually thick, variable diameter	undulose or parallel	strongly birefringent	none	dependent on thickness commonly strong blue/yellow	slow
Natural animal organic fibre	1.54-1.56	long, thick, scaly, coloured lumen	incomplete parallel	birefringent	none	strong blue/yellow	slow
Man made fibre-nylon	1.52-1.58	thick, long, surface pigment	parallel	strongly birefringent	none	two or three orders per fibre	slow
Man made fibre-acrylic	1.52	thick, long smooth	parallel	strongly birefringent	none	blue-green/yellow	fast
Man made fibre-cellulose acetate	1.475	long, smooth lobed cross section	parallel	birefringent	none	blue/yellow	slow
Man made fibre-cellulose triacetate	1.47	long, smooth, lobed cross section	parallel	very weakly birefringent or isotropic	none	very weak blue/yellow	variable fast/slow
Man made fibre-glass wool	1.51	smooth, straight or curved, variable diameter		isotropic	none		
Man made fibre-rock wool	1.50-1.65	smooth, straight or curved, variable diameter		isotropic	none		
Man made fibre-microquartz	1.42-1.46	smooth, straight or curved, variable diameter		isotropic	none		

* Bulk refractive index range parallel to length.

Table 3. OPTICAL CHARACTERISTICS USED IN IDENTIFICATION OF ASBESTOS MINERALS (after MONKMAN, 1979).

Asbestos minerals	Refractive indices		Interference $n_{\gamma} - n_{\alpha}$	Interference colours*	Optical orientation	Optical extinction	Colour and pleochroism
	n_{α} perpendicular to fibre axis	n_{γ} parallel to fibre axis					
Chrysotile	$n_{\alpha} = 1.537-1.549^{\dagger}$	$n_{\gamma} = 1.545-1.556^{\dagger}$	Weak 0.006-0.003	1st Order Yellow	Length slow	Parallel	Colourless to pale yellow-green
Actinolite	$n_{\alpha} = 1.617-1.658$	$n_{\gamma} = 1.641-1.677$	Moderate: strong 0.019-0.024	2nd Order: orange-red	Length slow	Inclined 0-15°	Colourless pale green. Faint pleochroism in green varieties
Amosite	$n_{\alpha} = 1.664-1.686$	$n_{\gamma} = 1.680-1.698$	Moderate 0.014-0.022	2nd Order: yellow	Length slow	Parallel	Colourless to pale green-yellow with slight pleochroism in coloured varieties.
Anthophyllite	$n_{\alpha} = 1.596-1.654$	$n_{\gamma} = 1.625-1.667$	Moderate/ strong 0.016-0.024	2nd Order:	Length slow	Parallel	Colourless to pale coloured with slight pleochroism in coloured varieties
Crocidolite	$n_{\gamma} = 1.681-1.698$	$n_{\alpha} = 1.678-1.694$	Weak 0.004-0.008	1st Order: greyish white masked by body colour	Length fast	Parallel	Pleochroic α = mid to <u>deep blue</u> γ = colourless to light or <u>grey blue</u>
Tremolite	$n_{\alpha} = 1.599-1.620$	$n_{\gamma} = 1.622-1.641$	Moderate/ strong 0.019-0.024	2nd Order: orange-red	Length slow	Inclined 0-20°	Colourless.

*Maximum interference colours shown by bundles up to 40 μm in diameter.

†Range for natural chrysotile: prolonged immersion in cold 3 M HCl will significantly lower the refractive index.

GUIDE TO THE IDENTIFICATION OF FIBRES

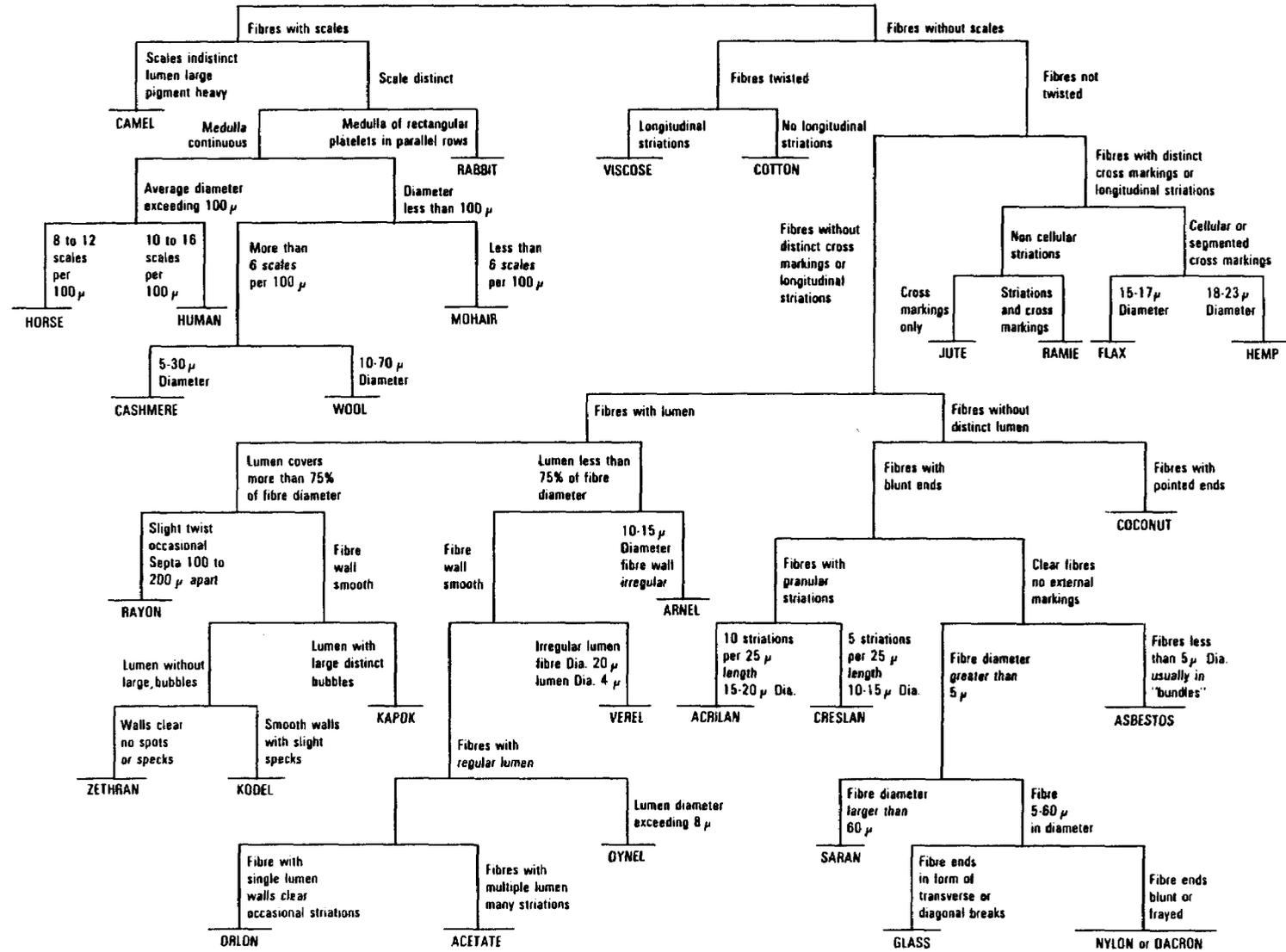


Figure 1.

refractive indices then the edges of the object will be very black and it is said to have high relief. Therefore amosite and crocidolite fibres would be indistinct in a medium with an RI of 1.68 and could be distinguished from other types of asbestos which would have medium to high relief in such a medium. Refractive indices for the various asbestos types are listed in Table 3.

The Becke Line method is used to compare the refractive indices of a particle and an immersion liquid. In this method, a high power objective and convergent light are required, i.e. condenser in and iris diaphragm almost closed.

Initially the boundary of a fibre is brought into sharp focus. Then the microscope objective is raised slightly (or the stage is lowered). If the refractive indices of the fibre and the immersion oil are different, a bright line (Becke Line) will appear near the interface between the fibre and the oil. As the objective is raised further the Becke Line will move towards the medium of higher refractive index. This effect is easiest to observe when relief is low. If the fibre and the immersion liquid have identical refractive indices then the Becke Line will disappear. By using calibrated immersion liquids it is possible to determine the RI of a particle to an accuracy of ± 0.002 .

Dispersion

The refractive index of a particle is not only dependent on the material but also on the ambient temperature and the wave-length of light passing through it. Dispersion is a measure of the influence of the wave-length of light on the RI and is commonly calculated by measuring the refractive indices of the substance using monochromatic light of varying wave-lengths within the visible spectrum.

If a mineral is placed in an immersion oil of high dispersion then dispersion colours which are characteristic of the mineral are produced. For example, if the RI of a mineral such as chrysotile is plotted against λ (i.e. the dispersion curve is plotted on semi-log paper - Hartmann plot) on the same graph as the dispersion curve of an immersion oil of $n_D = 1.580$ ($n_D = \text{RI}$ in yellow light) then the two dispersion curves will intersect in the infra-red region of the spectrum (see fig. 2). Chrysotile, when mounted in oil of $n_D = 1.580$, will appear blue.

The dispersion curve for tremolite would intersect the curve for the immersion oil ($n_D = 1.580$) in the ultra-violet region (fig. 3) and therefore appears yellow-orange.

If the dispersion curve of the mineral overlaps with the dispersion curve of the oil, coloured Becke Lines are produced; e.g. when the mineral and immersion oil in Figure 4 are observed under white light, there will be a blue Becke Line which moves into the oil and a complementary red Becke Line which moves into the mineral.

The colours observed in dispersion staining are generally pale and difficult to see in fibrous minerals and the technique requires converging light and high magnification. Sensitivity is increased by using dark-field and phase contrast microscopy. If a phase contrast microscope is not available, it is easier to use a number of different characteristics such as birefringence, optical orientation, estimation of the RI etc. in asbestos identification. These additional optical parameters are described below after Figure 4.

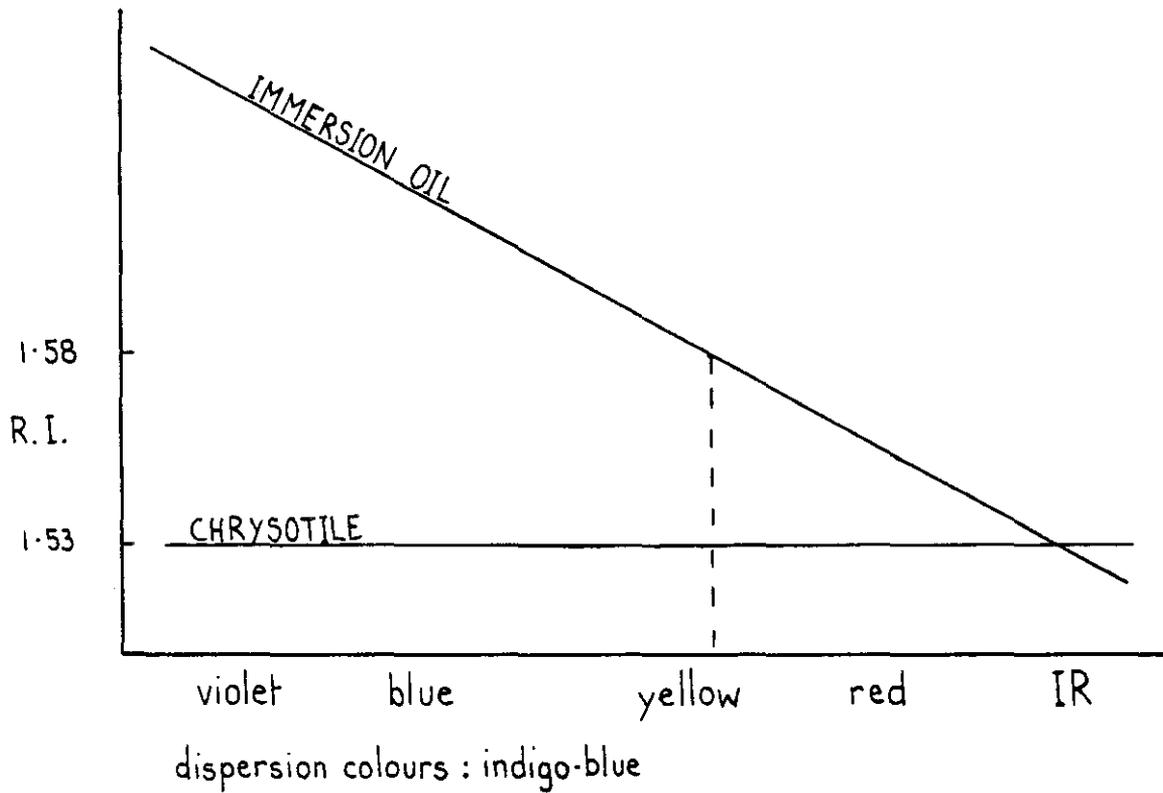


Figure 2. Dispersion staining colours of chrysotile.

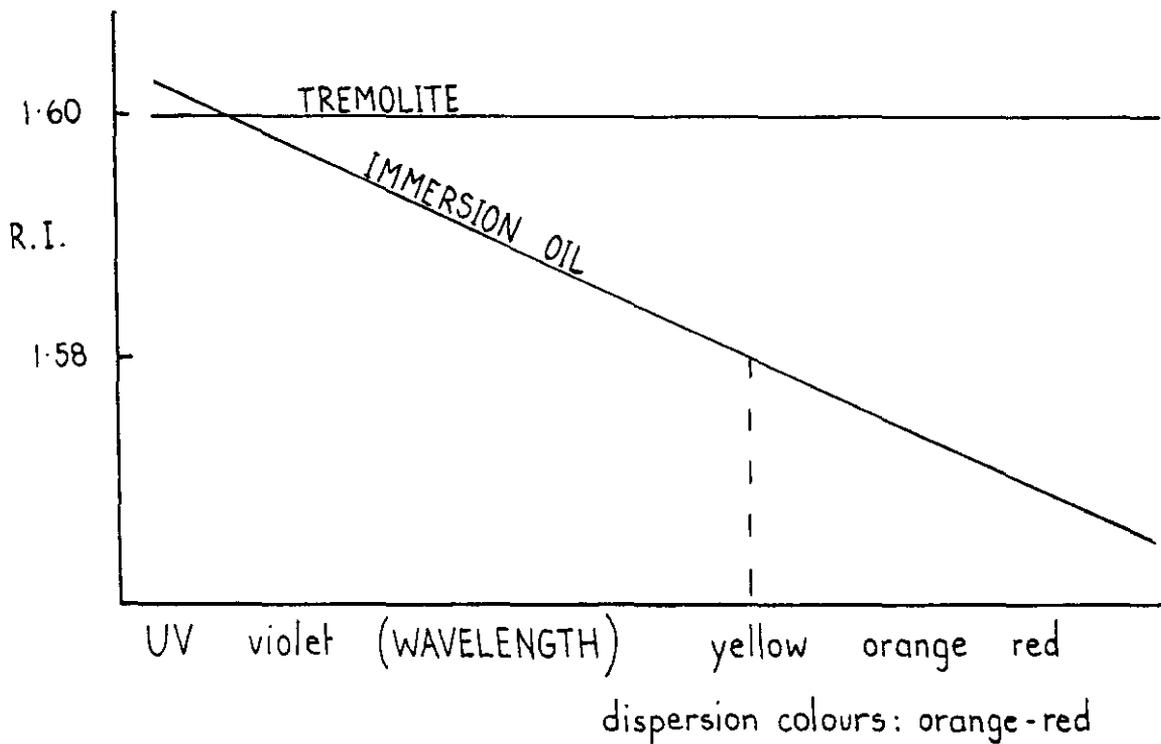


Figure 3. Dispersion staining colours of tremolite.

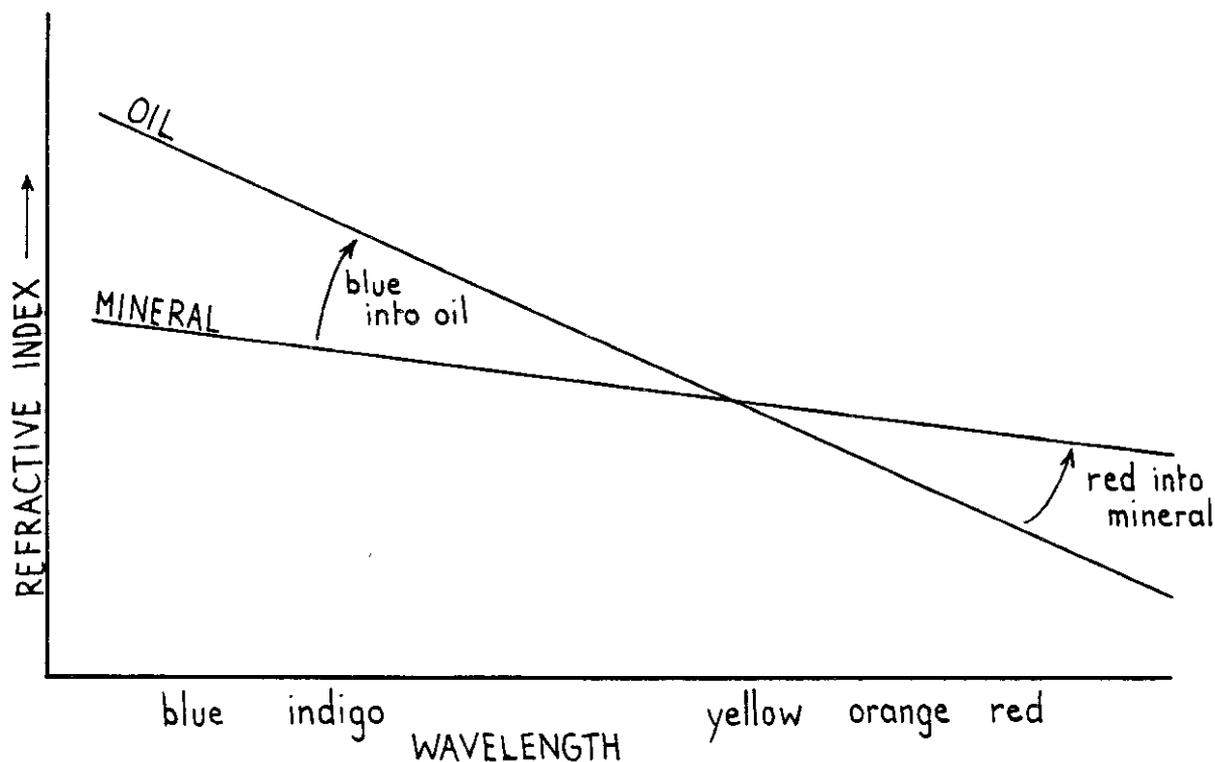


Figure 4.

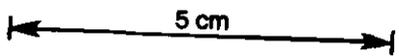
←————— 5 cm —————→

Birefringence

When light passes through a uniaxial anisotropic substance, it is split into two part-rays. One ray passes through the object in a straight line and the other undergoes parallel displacement. With biaxial crystals (which includes asbestos) both rays are displaced. The two part-rays have different velocities associated with different crystallographic directions and hence different refractive indices (n_α , n_β , n_γ). The refractive index of the slowest ray (i.e. the higher refractive index) is n_γ and of the fast ray (lowest RI) is n_α . The difference $n_\gamma - n_\alpha$ is referred to as the birefringence of a substance and is one factor responsible for the birefringence (interference) colours that are produced between crossed polars. The interference colour produced also depends on the thickness of the crystal.

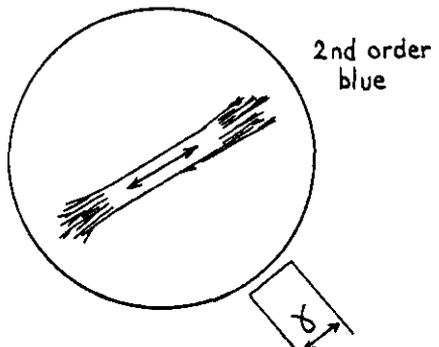
The colours produced by a mineral with low birefringence are referred to as 1st order colours. Minerals with higher birefringence values produce 2nd and 3rd order colours. Thinner crystals produce lower-order colours than thick crystals. Michel-Levy colour charts which relate crystal thickness and birefringence to interference colours are available (e.g. in Phillips, 1971).

Crocidolite is only weakly birefringent and produces low 1st order, grey-white interference colours which are masked by the blue body colour of the mineral. Chrysotile also has a low ($n_\gamma - n_\alpha$) value and produces 1st order grey to yellow. Amosite has moderate birefringence and appears bright green to bright yellow (2nd order colours) and other varieties of asbestos produce 2nd order orange-red interference colours (refer to Table 3).



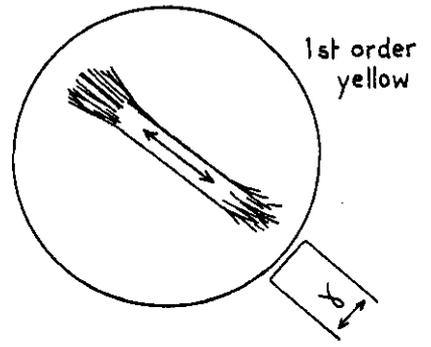
- 1. Chrysotile - length slow, interference colours - 1st order grey
retardation $\sim 150 \mu\text{m}$
retardation of gypsum plate $550 \mu\text{m}$

i. Fibre parallel to γ wave of gypsum plate



retardation = $150 + 550$
= $700 \mu\text{m}$

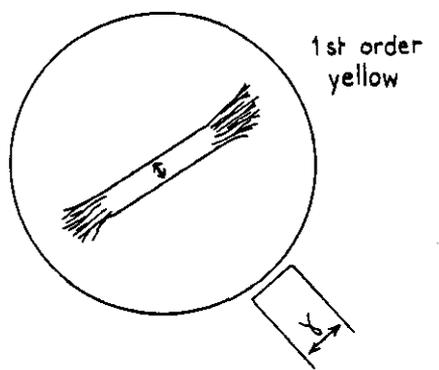
ii. Fibre perpendicular to γ wave of gypsum plate



retardation = $150 - 550$
= $- 400 \mu\text{m}$

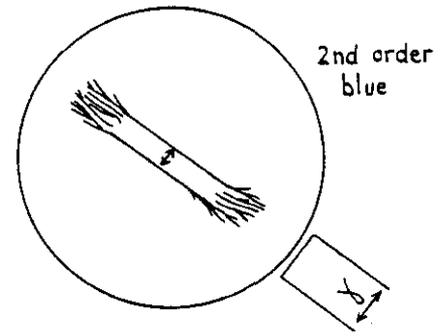
- 2. Crocidolite - length fast, interference colours - 1st order grey
retardation $150 \mu\text{m}$

i. Fibre parallel to γ wave of gypsum plate



retardation = $150 - 550$
= $- 400 \mu\text{m}$

ii. Fibre perpendicular to γ wave of gypsum plate



retardation = $150 + 550$
= $700 \mu\text{m}$

Figure 5. The effect of the gypsum plate on birefringence colours.

It is possible to distinguish crocidolite from other types of asbestos by determining the optical orientation of the mineral. This is done by inserting the gypsum (1st order red) plate into the accessory slot of the microscope (still with crossed polars). This imposes an additional retardation of 550 μm on the polarized light paths. The asbestos fibre is rotated until it is parallel to the polarisation direction of the slow wave of the gypsum plate (this is marked on the plate with an arrow). If the vibration direction of the slow wave in the fibre is parallel to the direction of the slow wave in the gypsum plate then the interference colours of the fibre will advance. Interference colours of higher order will be observed as a result of increased retardation of the light path imposed by the gypsum plate e.g. chrysotile fibres, of average diameter, produce 1st order yellow under crossed polars alone and 2nd order yellow under the gypsum plate when the fibre is parallel to the slow-wave direction of the plate (see fig. 5).

If the slow vibration direction of the fibre is perpendicular to the slow γ -wave of the gypsum plate, then retardation will decrease 550 μm and interference colours of a lower order are produced, e.g. amosite (2nd order bright yellow under crossed polars) would appear a pale 1st order orange. In most types of asbestos the slow ray is parallel to the length of the fibre, so that there is an increase in the retardation value when the fibre is parallel to the γ direction of the gypsum plate and a decrease when the fibre is perpendicular (i.e. the fibre is length slow). Crocidolite is length fast (i.e. the γ vibration direction is perpendicular to the length of the fibre) so when the fibre is parallel to the γ direction of the gypsum plate, a decrease in retardation is observed (fig. 5).

Angles of Extinction

If a mineral is rotated under crossed polars then it is noticed that there are 4 positions where the fibre becomes dark or even invisible (i.e. at extinction). There are four angles of extinction with 90° between them. There are also four positions of maximum brightness which are 45° from a position of extinction. For most types of asbestos the fibre is at extinction when it is parallel to the vibration direction of the polariser or analyser. However actinolite and tremolite are at extinction when the fibre is at an oblique angle to the N-S or E-W planes ($0-15^\circ$ for actinolite and $0-20^\circ$ for tremolite).

Pleochroism

Pleochroism is observed with plane polarised light (i.e. uncrossed nicols). With most types of asbestos, pleochroism is essentially absent and if the stage is rotated there is almost no change in fibre colour. Crocidolite appears mid-deep blue in certain positions and colourless to light blue if it is rotated 90° . These colours are related to vibration direction (i.e. α = mid to deep blue and γ = colourless to light blue).

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