Working Report No. 24 2000
Arbejdsrapport fra Miljøstyrelsen

A Random sampling examination of Crayons in Denmark

An examination of crayon fillers for a possible content of asbestos

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Miljøstyrelsen vil, når lejligheden gives, offentliggøre rapporter og indlæg vedrørende forsknings- og udviklingsprojekter inden for miljøsektoren, finansieret af Miljøstyrelsens undersøgelsesbevilling.

Det skal bemærkes, at en sådan offentliggørelse ikke nødvendigvis betyder, at det pågældende indlæg giver udtryk for Miljøstyrelsens synspunkter.

Offentliggørelsen betyder imidlertid, at Miljøstyrelsen finder, at indholdet udgør et væsentligt indlæg i debatten omkring den danske miljøpolitik.

Report prepared for
Annette Orloff
Danish Environmental Protection Agency
Copenhagen, Denmark.
Our j. No. 2000 – 54 – 5.2
Case officer: senior advisor Ole Jørgensen.
September 2000.

Report of Analysis

A RANDOM SAMPLING EXAMINATION OF CRAYONS IN DENMARK.

An examination of crayon fillers for a possible content of asbestos.

Summary:
At the request of the Danish Environmental Protection Agency 15 crayon products in Denmark were examined for asbestos by a random sampling. Two products were represented by two and six boxes respectively. The other products were represented by one box each. From each of the boxes three crayons were selected for analysis.

After destruction of the wax by heating, the inorganic filler was analysed by X-Ray Diffraction (XRD), Polarised Light Microscopy (PLM), Phase Contrast Microscopy (PCM) and Scanning Electron Microscopy (SEM) plus Energy Dispersive Spectrometry (EDS).

The result of the examination shows that six of 63 fillers contained chrysotile, tremolite or anthophyllite asbestos. No other types of asbestos were found. Some samples showed no asbestos, but cleavage fragments of tremolite and fibres of sepiolite, talc ("transitional fibres") or nemalite. Based on the number of observed asbestos fibres the concentrations of asbestos in the fillers were estimated to be equal to or less than 0.1% w/w.

The observed mineral fibres are a natural pollutant of the talc and dolomite, which is used as fillers.

Though no asbestos was found in a sample containing talc or dolomite, there is 95% chance that the asbestos concentration is less or equal to the concentration, which would correspond to an observation of four fibres in the sample. This applies particularly to samples that contain cleavage fragments of tremolite, anthophyllite or another amphibole mineral. That means, if a potential risk exists, that a material may contain asbestos, no observed fibres do not mean that the material is asbestos-free.

The present examination shows that asbestos generally do not occurs in crayons, but that a potential risk exists, that asbestos may occurs if the crayons contain rock materials like talc or dolomite.

Resumé

Efter anmodning fra Miljøstyrelsen er der foretaget en stikprøveundersøgelse af voksfarvestifter for et muligt indhold af asbest. Der er undersøgt 15 forskellige produkter som sælges i Danmark. To produkter er repræsenteret ved henholdsvis to og seks æsker stifter. De øvrige produkter er repræsenteret ved en enkelt æske. Fra hver æske blev der udtrukket tre farvestifter til analyse.

Efter at voksen var destrueret ved opvarmning, blev det uorganisk fyldstof analyseret ved hjælp af røntgen pulver diffraktometri, optisk polarisations mikroskopi og scanning elektron mikroskopi kombineret med energi dispersiv grundstof spektrometri.

Undersøgelsen viste, at blandt 63 prøver af fyldstoffer var der *seks* prøver, som enten indeholdt chrysotil, tremolit eller anthophyllit asbest. Der blev ikke fundet andre typer asbest. Nogle prøver indeholdt derimod spaltestykker af tremolit eller fibre af talk (”transitional fibres”), sepiolit eller nemalit. Ud fra antallet af observerede fibre, er koncentrationen af asbest skønnet til at være mindre end eller lig med 0.1 vægt-% af fyldstoffets masse.

De observerede mineralfibre er en naturlig forekommende forurening af de mineralske fyldstoffer, der anvendes af industrien.

Hvis der ikke er observeret asbestfibre i en prøve, som indeholder talk, dolomit eller spaltestykker af tremolit, er der 95% chance for, at asbestkoncentrationen er mindre eller lig den koncentration, som svarer til, at der blev observeret fire asbestfibre i prøven. Dette gælder specielt de prøver, som indeholder tremolit eller et af de amfibolmineraler som kan udvikles som asbest. Dette skal fortolkes på den måde, at hvis der er en potentiel risiko for, at materialet kan indeholde asbest, er ingen observerede fibre *ikke* ensbetydende med, at materialet er asbestfrit.

Konklusionerne af denne undersøgelse er, at asbest generelt ikke forekommer i de undersøgte farvestifter, men at der eksisterer en potentiel risiko for, at asbest kan forekomme, hvis farvestifterne indeholder et bjergarts materiale som talkum eller dolomit.

Introduction

In USA and Canada some types of crayons (colour sticks) used by children has been suspected to contain asbestos. However, an investigation carried out by RJ Lee Group, Inc, Pennsylvania [1] could not certify this statement. In the light of this, the Danish Environmental Protection Agency decided to initiate a Danish random sample examination of colour sticks that occur on the Danish marked.

Colour sticks are a mixture of wax, inorganic filler and colour pigments. The filler may be talc, dolomite, kaolinite etc. Such fillers used by the industry contains small amounts of foreign minerals and some times asbestos. The type and amount of foreign minerals/asbestos vary from one quarry to another, but within the same quarry variations may also occur. Consequently, crayons produced at different times may contain different amounts of foreign minerals. This is important to remember when crayons are checked for asbestos. In order to make a reliable check, one most checks more than one crayon.

The present investigation covers all types of crayons that occur on the Danish market (Table 1). The products are divided into two groups:

- The first group includes crayons that were screened for the first time. These products are represented by one box each and three colours stick were examined form each of the boxes.

- The second group including Crayola products. An earlier examination of three Crayola crayons from one box (Table 1, sample No. CYA) showed that two crayons contained anthophyllite asbestos while no asbestos was observed in the third one [13]. The occurrence of asbestos may be an incident. In order to check those observations, 15 crayons from five new boxes of Crayola crayons were analysed at the present investigation.

EU method

The EU method [2] describes an analytical procedure by which asbestos in bulk materials can be identified and quantified using Polarising Light Microscopy (PLM) and Phase Contrast Microscopy (PCM). The method can be used to assess mass concentration of asbestos in bulk materials above 0.01%. From Fig. 1A and 1B it can be seen that the quantification follows a stepwise approach, beginning with a simple screening of the bulk material by stereo microscope for large fibres and identification by PLM. The mass percentage of chrysotile and amphibole asbestos is quantified by PLM/PCM. If this does not suffice to reach a conclusion, analysis by Scanning Electron Microscopy (SEM), or even high-resolution Transmission Electron Microscopy (TEM) may be required.
X-Ray Diffraction analysis (XRD) is optional for high asbestos concentration (the detection limit of the method is about 0.5 % w/w of a phase in a mixture), but the method cannot distinguish between the fibrous and non-fibrous form of the minerals (see below).
Pre-concentration can be used to make an analysis easier or to improve the precision of the quantification, if the asbestos concentration of the bulk sample is low.

Present investigation.

The purpose of the present investigation was to check the samples in Table 1 for a possible content of asbestos. If asbestos occurs, the concentration will be determined by a following investigation.

The analytical procedure follows the first steps shown in the flow scheme in Fig. 1A and 1B. In order to remove the wax, a known amount of crayon (without cover paper) was heated to 460°C for 10 hours. After cooling in disiccator, the inorganic filler was weighed and the residue mass fraction was calculated.

The main components of the fillers were identified by XRD. The analysis was carried out on a Philips diffractometer PW 1800 equipped with Cu-tube, graphite monochromator and rotating sample stage. Data of the diffractograms were collected between 5 and 100° 2θ . The phases of the samples were identified automatically by the computer on the instrument, which compared the diffractogram of the sample to 2000 ASTM standard diffractograms of known minerals and substances. However, the result of the XRD analysis gives only the most possible names of phases.

It is therefore necessary to check the result of the XRD analysis by PLM or by SEM/TEM plus energy dispersive element analysis (EDS).

The examination of the fillers was carried out by a Leitz Laborlux 12 Pol microscope at magnification between 40 and 630 times. At the high power magnification the resolution of the microscope is about 0.5 μm. The examination was carried out on 1 to 3 mg filler, which was immersed in a liquid with a refractive index of 1.600 (see below) and 100 fields of view was examined between one and two polarisers respectively. If mineral fibres were observed, the fibres were identified by the PLM procedure described in [12].

Distinguishing features

Crayons that contains talc as filler forms a special problem. Investigations carried out by [3, 4, 5, 6, 7, 8, 9] show that talc may contain fibre-formed talc and intergrowths between talc and anthophyllite/ tremolite fibres. In a report from the RJ Lee Group, Pennsylvania [1] the intergrowth between talc and anthophyllite are mentioned as *"transitional fibres*". In addition to the transitional fibres, fibres of anthophyllite and tremolite have been observed in talc from the New York talc district [5, 7]. Consequently, by screening one must be able to distinguish between transitional fibres and fibres of chrysotile and amphibole asbestos.

Transitional fibres can be distinguished from asbestos fibres by its refractive index. For transitional fibres the highest refractive index (n_λ) is parallel to the length of the fibre and the refractive index vary between 1.590 and 1.600 [1]. For amphibole fibres and chrysotile the λ direction of light vibration is also parallel/sub parallel to the length of the fibres, but $n_\lambda > 1.600$ for the amphibole fibres [10] and for chrysotile the refractive index varies between 1.545 and 1.556[11]. That means, when a transitional fibre is immersed in a liquid with a refractive index of 1.600 and the fibre is oriented parallel to the direction of light vibration of the microscope, the fibre will be nearly invisible, while fibres of amphiboles and chrysotile can be seen. Chrysotile and amphibole asbestos can be distinguished from each other by observing the movement of the Becke line when the distance between the sample and the objective of the microscope is increased.

The International Standards Organisation defines asbestos as follows (ISO 10312:1995):
" Asbestos: a collective term applied to specific serpentine and amphibole minerals which have been crystallised in asbestiform habit, causing them to separate into long, thin, strong fibres when crushed or processed. The most common forms are …….".

While chrysotile always have asbestiform habit, there exist a large number of transitional habits between amphibole crystals and amphibole asbestos and the different habits may occur together in the same sample. Fig. 2, 3 and 4 shows the habit variation of tremolite. Needle shaped cleavage fragments of amphibole crystals and acicular crystals can be distinguished from the asbestoid habit of the mineral by its morphology and the extinction between crossed polarisers. The cleavage fragments and the acicular crystal are "single-crystals" that shows distinct extinction between crossed polarisers. Since the amphiboles are monoclinic crystals, the direction of extinction can either be parallel or oblique relative to the longitudinal axe of the grain - depending on the orientation of the crystals on the specimen stage.

Fibres of asbestos are composed of numerous fibrils. The length of the fibrils is parallel to each other and to the longitudinal axe of the fibre. However, within the single fibre the fibrils are rotated about the longitudinal axe of the fibrils. The result is that an asbestos fibre is not a single-crystal, but an aggregate of fibrils with more or less random orientation. Aggregates have other optical properties than single-crystals. When the asbestos fibres are observed between two polarisers, the fibres shows undulating extinction that are more or less parallel to the longitudinal axe of the fibre.

It was mentioned above that x-ray diffraction could not be used for discrimination between the fibrous and non-fibrous form of the asbestos. The reason is that the two habits of the same mineral are identical with respect to chemical composition and physical properties. Only microscopic methods and the human eye are able to discriminate between the asbestiform and non-asbestiform habit of a mineral.

Rules for data recording

According to the EU method [2] all particles with a length to width ratio (aspect ratio) greater than 3:1 and a length exceeding 5 μm are recorded as fibres. There is no upper limitation of the fibre length.
Asbestos is by definition the six fibrous varieties of the following serpentine and the amphibole minerals:

Chrysotile, CAS No. 12001-29-5.
Amosite (Grunerite asbestos), CAS No. 12172-73-5.
Crosidolite (Riebeckite asbestos), CAS No. 12001-28-4.
Tremolite asbestos, CAS No. 77536-68-6.
Anthophyllite asbestos, CAS No. 77536-67-5.
Actinolite asbestos, CAS No. 77536-66-4.

Observations

The result of the examination is summarised in Table 2A and 2B. From the table it can be seen that the major components of fillers are talc, calcite, dolomite, quartz, tremolite, muscovite and illite and a mixture of two or more of these minerals. Gypsum was only found in Sample no. KS which is chalk for road painting. TiO_2, ZnO and hematite (Fe_2O_3) is white and brown colour pigment respectively that has survived the heating to 450°C. The other colour pigments were organic substances, which were destructed by the heating.
By mounting the filler in a liquid with a refractive index of 1.600 it was possible to discriminate between transitional fibres of talc and other mineral fibres with a refractive index, which is higher or less than 1.600. When fibres were observed, the fibres were identified by its optical properties and chemical composition. The result of the identification is summarised in table 3. By comparing Table 2A,B and Table 3 it can be seen that only *six* out of 63 samples contains asbestos and that two samples contained other types of mineral fibres. The following types of asbestos were found by the examination; chrysotile, tremolite and anthophyllite. Two samples contained fibres of the non-asbestos minerals nemalite and sepiolite. These fibrous minerals are frequently found as a natural pollutant of talc and dolomite.

It was mentioned above that tremolite and the other amphibole minerals can crystallise as solid crystals as well as acicular fibres and asbestos. However, the tremolite containing samples No. 910, 912, 913, 914, 915 and 916 the mineral occurs only as *cleavage fragments*.
In the mentioned samples some fibres are visible when the filler is mounted in a liquid with a refractive index of 1.600 (Fig. 11), but the morphology and the chemical composition (delaminated by EDS on SEM) suggests that the fibres are transitional fibres of talc of which the refractive index is higher than that of the typical transitional fibre. The transitional fibres are an oriented intergrowth between talc and anthophyllite or tremolite [3, 4, 5, 6, 7, 8, 9]. Since the chemical composition of these high index fibres are identical with transitional fibres that are invisible in a liquid with a refractive index of 1.600, we must conclude that the change in refractive index is caused by the internal texture of the fibre i.e. the order / disorder of the intergrowth. The optical properties are extremely sensitive to structural and textural order / disorder and the phenomena is well known form a large number of minerals e.g. the feldspar [15].

Table 3. Mineral fibres identified in crayon fillers. Samples containing asbestos are indicated by *

Sample No.	Mineral fibre	N_{100}	Method of identification	Fig.
904-2	Sepiolite	10	PLM, SEM +EDS	6A, B, C.
909-1	Nemalite	7	PLM, SEM+EDS	7A, B, C.
910-1*	Tremolite	3	PLM, SEM+EDS	8A, B.
910-2*	Tremolite	3	PLM, SEM+EDS	9A, B.
910-3*	Chrysotile	9	PLM+EDS	10.
CYA-1*	Anthophyllite	27	PLM, SEM+EDS	[13]
CYA-2*	Anthophyllite	36	PLM, SEM+EDS	[13]
MO-3*	Tremolite	10	PLM, SEM+EDS	

N_{100}, number of observed fibres per 100 field of view. By PLM the area of one field of view is 0.0079 mm^2

Sepiolite ($Mg_4 (OH)_2 Si_6O_{15}, 6H_2O$) and nemalite (fibre formed brucite $Mg(OH)_2$, Fig. 7A , B) are minerals which can be confused with chrysotile and transitional fibres of talc respectively.

Sepiolite is nearly identical to chrysotile ($Mg_3 Si_2O_5(OH)_4$) both with respect to morphology (Fig. 6A, B) and chemical composition. However, the refractive index of sepiolite ($n_{\gamma'} = 1.529$, $n_{\alpha'} = 1.519$) is lower than that of chrysotile ($n_{\gamma'} = 1.545 – 1.556$, $n_{\alpha'} = 1.532 – 1.549$). Consequently, sepiolite cannot be distinguished from chrysotile by its element spectrum (Fig. 6C), but only by its lower refractive index.

The refractive index of nemalite ($n_{\gamma'}$ =1,580 – 1.600, $n_{\alpha'} = 1.560 – 1.590$) is identical to that of transitional fibres of talc (see p. 4), but in contrast to transitional fibres, n_α is parallel to the length of the nemalite fibre. That means, by inserting a gypsum plate in the tubus of the microscope, it can be seen that nemalite is “length fast” (negative elongation) while transitional fibres is “length slow” (positive elongation) (Fig. 7A). By SEM + EDS the two minerals can easily be distinguished from each other, because transitional fibres is a magnesium silicate while nemalite is magnesium hydroxide (Fig. 7B and C).

Tremolite and anthophyllite was identified by its chemical composition (Fig. 9A, B) and optical properties (Fig. 8). Magnesium-rich anthophyllite may be distinguished from tremolite by its lower refractive index and birefringence compared to that of tremolite (n_γ = 1.615 for anthophyllite, n_γ = 1.622 for tremolite. Birefringence Δn = 0.013 for anthophyllite, Δn = 0.027 for tremolite). By application of SEM + EDS anthophyllite ($(Mg, Fe)_7[Si_8O_{22}](OH)_2$) may be distinguished from tremolite ($Ca_2(Mg, Fe)_5[Si_8O_{22}](OH)_2$) by the lack of calcium in the element spectrum.

Discussion

As mentioned in the introduction mineral fillers used by the industry may contain small amounts of foreign minerals as a natural "pollutant". It is therefore surprising to see that only six of 63 samples contained asbestos and that only two samples contained foreign minerals. That means that 9,5 % of the examined filler contained asbestos. However, the concentration of asbestos must be low. Based on the observed number of asbestos fibres, the concentration of asbestos was estimated to be about or less than 0.1 % w/w of the mass of the filler.

The result of the present investigation is in good agreement with the result obtained by a random sample examination of asbestos in imported products in Denmark [14]. That investigation showed that 12 out of 83 samples contained tremolite asbestos or that asbestos was found in 14.5% of the samples. The concentration of tremolite asbestos ranges from 2×10^{-12} to 4×10^{-2} % w/w.

Though no fibres of asbestos were found in a sample which contained talc, tremolite or dolomite there is a 95% chance that the asbestos concentration is less or equal to the concentration, which would correspond to an observation of four fibres in the sample [16]. That means, if a potential risk exists that a material may contain asbestos, no observed fibre do not mean that the material is asbestos-free.

Conclusion

The present examination shows that asbestos is an infrequent substance in crayons. Out of 63 samples, chrysotile, tremolite or anthophyllite was found only in six samples. Based on the number of observed fibres the concentration was estimated to be equal or less than 0.1% w/w of the mass of the filler. No other types of asbestos were observed, but some of the samples contained fibres of the non-asbestos mineral nemalite and sepiolite. However there exists a potential risk, that asbestos may occurs in crayons, which contain talc, dolomite or tremolite.

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Table 1. Crayons sampled by the Danish Environmental Protection Agency

Sample No.	Name of product	Registered trade mark Origin.	Number of crayons pr. box	Size of crayon in mm.
900	Color Kids.	TOP-TOY, Roskilde. Denmark	6	15 x 110
901	18 Stik-Sammen farver	Top-Color	18	15 x 40
902	6 Starter Crayons.	SENSE. Haning. Sweden.	6	30 x 50
903	12 Farvestifter i flotte farver.	Artkids. Made in Malaysia.	12	8 x 85
904	Noris Club, 8 wax crayons.	Staedtler, Nürenberg. Germany. Made in Indonesia.	8	8 x 90
905	No sample			
906	6 Wax Crayons.	APP Scandinavia A/S, DK. Denmark	6	8 x 90
907	Color & Co, 16 Crayons.	Le France & Bourgeois. Made in China.	16	8 x 90
908	32 Crayoner.	Dan Pen. Made in China.	32	8 x 90
909	10 Super Voks Farver.	Penol A/S, Denmark.	10	12 x 120
910	Jumbo Crayons.	Free & Easy, Holland. Made in China.	6	14 x 100
911	48 Crayons.	Creative Kids, Inc. Made in Malaysia.	48	8 x 90
912	First 24 Crayons	Crayola, Binny & Smith. Made in England.	24	14 x 67
913	-	-	24	14 x 67
914	24 Crayola.	-	24	8 x 100
915	-	-	24	8 x 100
916	-	-	24	8 x 100
917	Oliekridt 103/12	Filia. Made in Denmark.	12	7 x 60
CYA	24 Crayola	Crayola, Binny & Smith. Made in England.	24	8 x 100
CS	64 Crayons	APP Scandinavia A/S, DK. Made in Indonesia.	64	8 x 90
MO	Motive	FDB, Albertslund, DK. Made in Thailand.	24	8 x 90
KS	4 Kids Tykke Gadekridt	VN. Legetøj A/S. DK.	6	25 x 100

Table 2A. Screening of crayons by XRD and PLM.

Sample No.	Colour	Conc. of filler (%w/w)	Major component of filler identified by XRD and PLM	Mineral fibres obs. by PLM ($n_{liq.}$= 1.600)	
				Yes	No
900-1	Red	21.7	Talc,		X
900-2	Yellow	22.2	Talc, calcite, TiO_2 .		X
900-3	Violet	23.4	Talc, dolomite TiO_2 .		X
901-1	Orange	10.2	Talc, TiO_2 .		X
901-2	Blue	12.5	Talc, dolomite, TiO_2 , ZnO.		X
901-3	Green	13.1	Talc, TiO_2 , ZnO		X
902-1	Pink	38.3	Talc, dolomite,TiO_2		X
902-3	Violet	38.4	Calcite, dolomite, Talc, TiO_2 , ZnO		X
902-3	Blue	39.0	Talc, TiO_2 , ZnO		X
903-1	Black	8.2	Talc, calcite, TiO_2.		X
903-2	Brown	7.4	Quartz, hematite, talc.		X
903-3	Green	6.0	Talc, TiO_2.		X
904-1	Green	11.5	Talc, TiO_2 .		X
904-2	Black	13.8	Hematite, talc	X[1)]	
904-3	Brown	12.9	Talc		X
905-1 905-2 905-3	No samples				
906-1	Red	15.9	Calcite.		X
906-2	Yellow	8.7	Calcite.		X
906-3	Blue	7.9	Calcite		X
907-1	Peach	23.1	Calcite, ZnO.		X
907-2	Magenta	21.5	Calcite, $BaSO_4$.		X
907-3	Sky blue	19.6	Calcite, $BaSO_4$.		X
908-1	Amber	21.8	Talc, dolomite, TiO_2.		X
908-2	White	24.4	Talc, dolomite, TiO_2.		X
908-3	Gr. Yellow	25.4	Talc, dolomite, TiO_2		X
909-1	Yellow	14.0	Talc, TiO_2	X [2)]	
909-2	Pink	19.1	Talc, TiO_2		X
909-3	Violet	19.6	Talc, TiO_2		X
910-1	Black	6.1	Quartz, calcite, tremolite.	X [3)]	
910-2	Orange	10.5	Quartz, calcite, tremolite	X [3)]	
910-3	Red	6.9	Quartz, calcite, tremolite	X [4)]	

1) Sepiolite. 2)Nemalite. 3) tremolite. 4) chrysotile.

Table 2B. Screening of crayons by XRD and PLM .

Sample No.	Colour	Conc. of filler (%w/w)	Main component of filler identified by XRD and PLM	Mineral fibres obs. by PLM ($n_{liq.}$= 1.600)	
				Yes	No
911-1	Pine gr.	12.3	Quartz, TiO_2, Talc.		X
911-2	Turquoise	7.5	Quartz, TiO_2, Talc.		X
911-3	Blue	5.0	Talc, dolomite, quartz.		X
912-1	Yellow	31.7	Tremolite.		X
912-2	Light blue	23.5	Tremolite, TiO_2.		X
912-3	Deep gr.	29.8	Tremolite.		X
913-1	Mint	31.7	Tremolite,		X
913-2	Orange y.	23.5	Tremolite, calcite.		X
913-3	Cyclamen	29.8	Tremolite, calcite.		X
914-1	Maize	17.3	Tremolite, hematite		X
914-2	Sky blue	18.1	Tremolite, TiO_2.		X
914-3	Grey	19.1	Tremolite, TiO_2.		X
915-1	Red	14.1	Tremolite.		X
915-2	Orange y.	9.5	Tremolite.		X
915-3	Light green	13.3	Tremolite, talc.		X
916-1	Green	12.4	Tremolite, TiO_2.		X
916-2	Aquamarine	13.0	Tremolite, talc.		X
916-3	Grey	20.0	Tremolite, TiO_2, ZnO.		X
917-1	Black	65.9	Muscovite, illite, quartz.		X
917-2	Violet	64.6	Muscovite, illite		X
917-3	Deep blue	67,1	Muscovite, illite		X
CYA-1	Red	17.6	Tremolite, anthophyllite, quartz.	X [1)]	
CYA-2	Apricot	17.5	Tremolite, quartz	X [1)]	
CYA-3	Violet	20.3	Tremolite.		X
CS-1	Cherry red	5.9	Calcite, quartz.		X
CS-2	Yellow	11.6	Calcite, quartz		X
CS-3	Deep green	7.9	Calcite, quartz		X
MO-1	White	12.6	TiO_2, quartz, dolomite.		X
MO-2	Violet red	20.6	Calcite, dolomite, talc.		X
MO-3	Green blue	16.2	Calcite, talc	X [2)]	
KS-1	Blue	100	Gypsum		X
KS-2	Red	100	Gypsum		X
KS-3	Green	100	Gypsum		X

1) anthophyllite. 2) tremolite

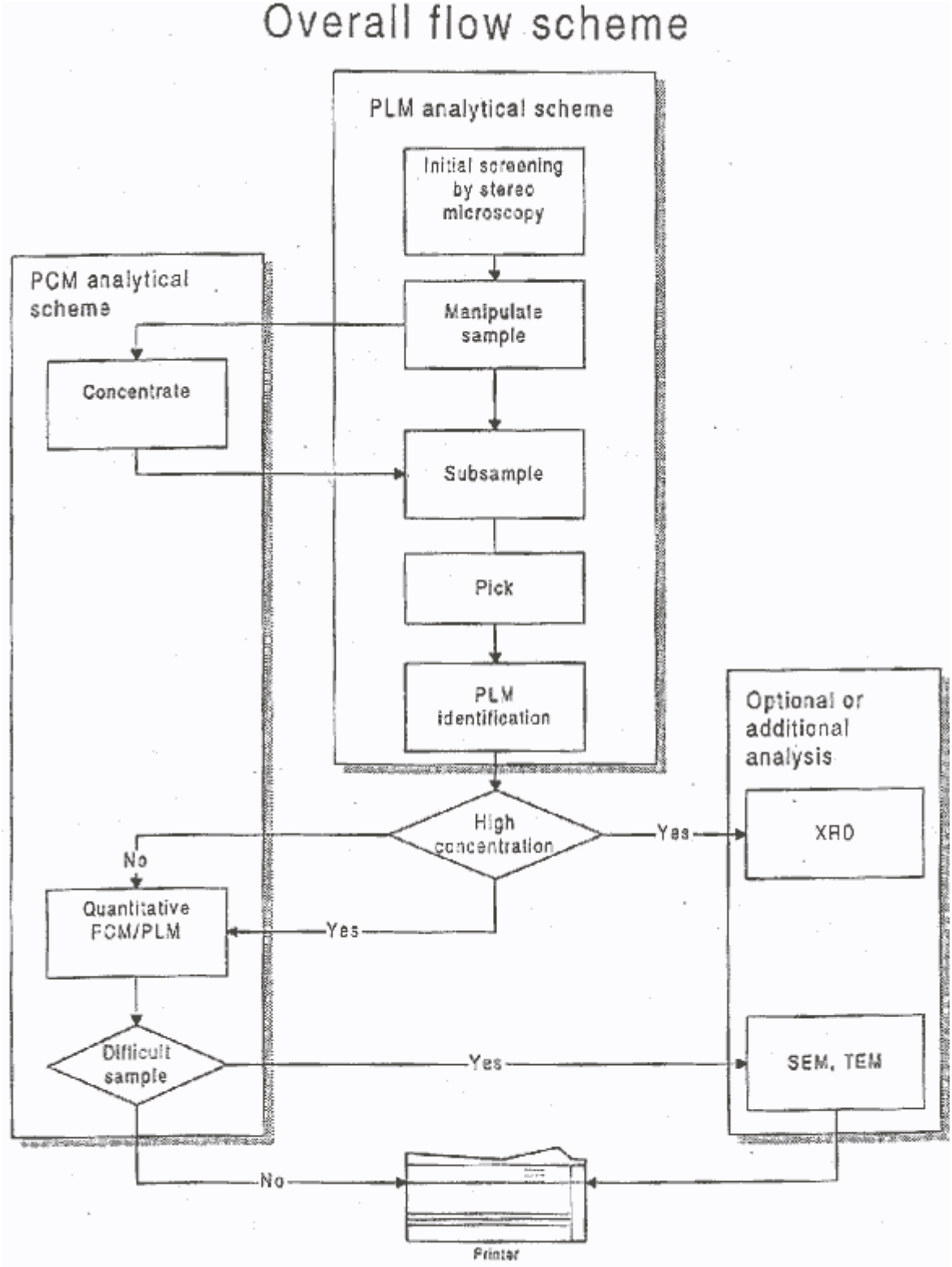
Overall flow scheme
PLM analytical scheme
Initial screening by stereo microscopy
Manipulate sample
Subsample
Pick
PLM identification
PCM analytical scheme
Concentrate
Quantitative PCM/PLM
Difficult sample
Optional or additional analysis
XRD
SEM, TEM
High concentration
Yes
No
Yes
Yes
No
Printer

Fig. 1A

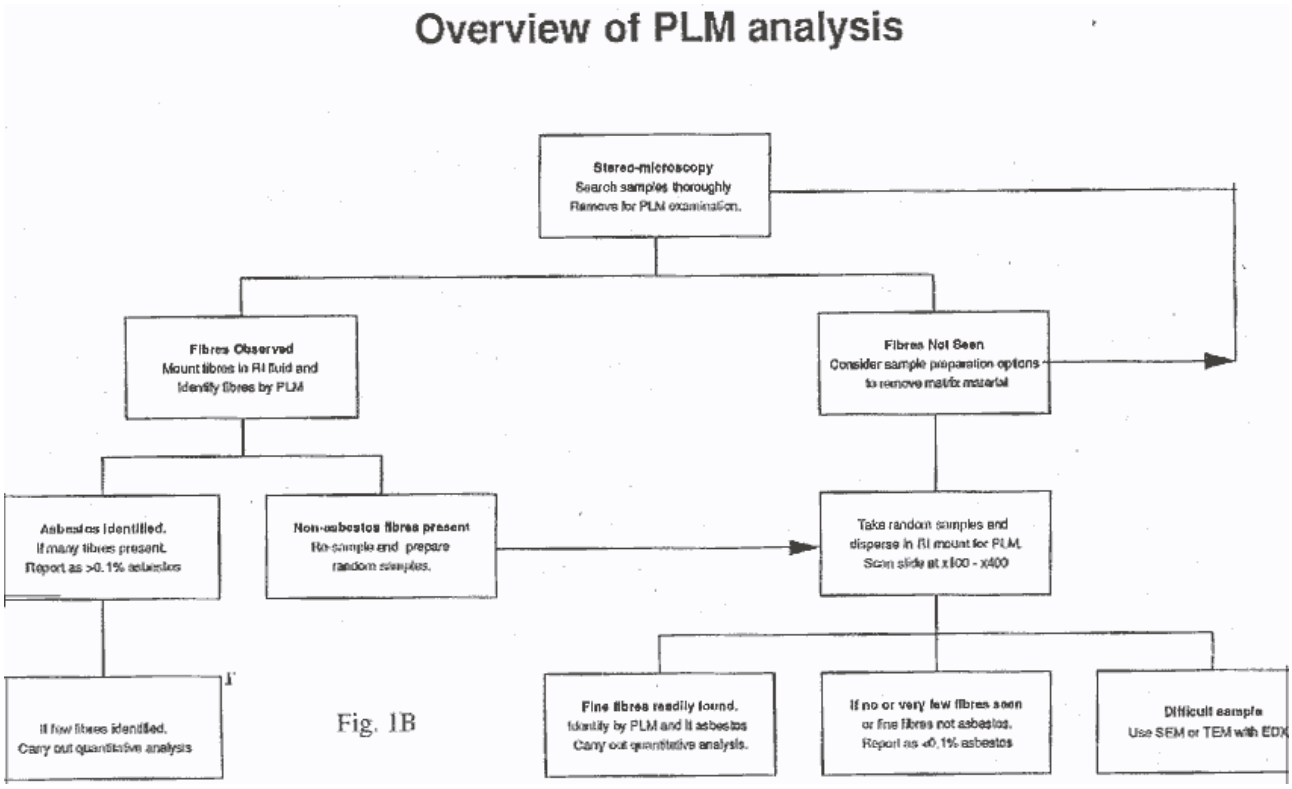
Overview of PLM analysis
Stereo-microscopy
Search samples thoroughly
Remove for PLM examination.
Fibres Observed
Mount fibres in RI fluid and
identify fibres by PLM
Fibres Not Seen
Consider sample preparation options
to remove matrix material
Asbestos identified.
If many fibres present.
Report as >0.1% asbestos
Non-asbestos fibres present
Re-sample and prepare
random samples.
Take random samples and
disperse in RI mount for PLM.
Scan slide at x100 - x400
If few fibres identified.
Carry out quantitative analysis
Fine fibres readily found.
Identify by PLM and if asbestos
Carry out quantitative analysis.
If no or very few fibres seen
or fine fibres not asbestos.
Report as <0.1% asbestos
Difficult sample
Use SEM or TEM with EDX

Fig. 1B

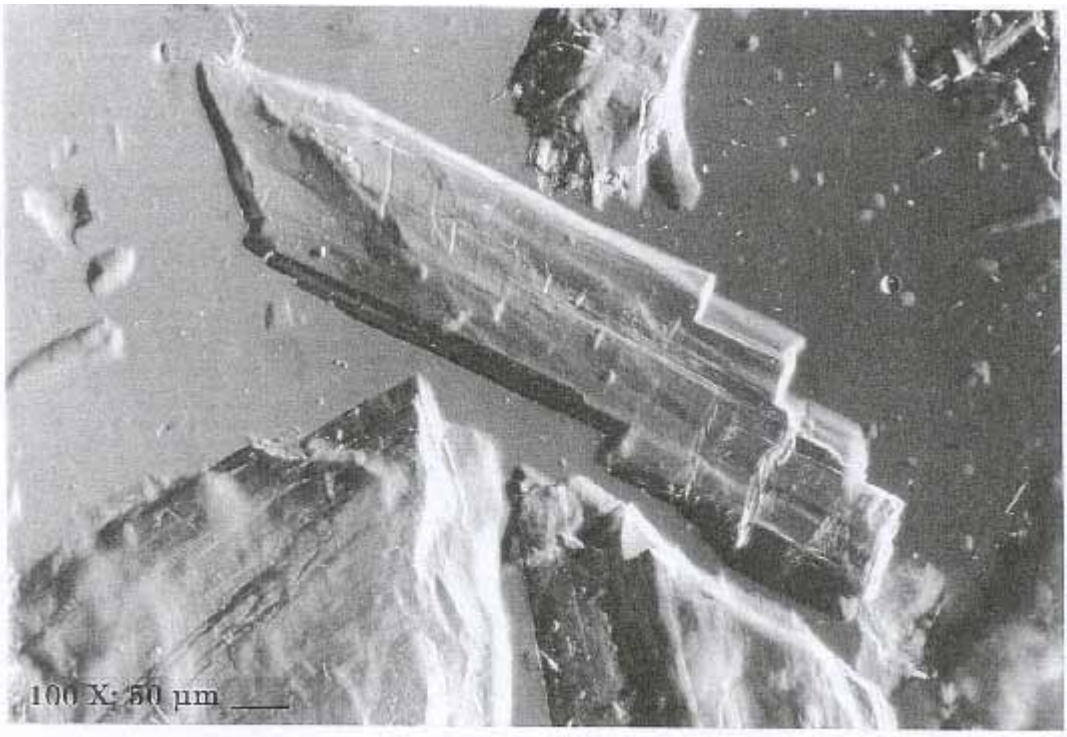

Fig. 2. Coarse grained cleavage fragments of tremolite defined as "amphibolic tremolite". Shininess, Lairg. Optical microscopy, oblique illumination. Photo Ole Jørgensen, AMI.

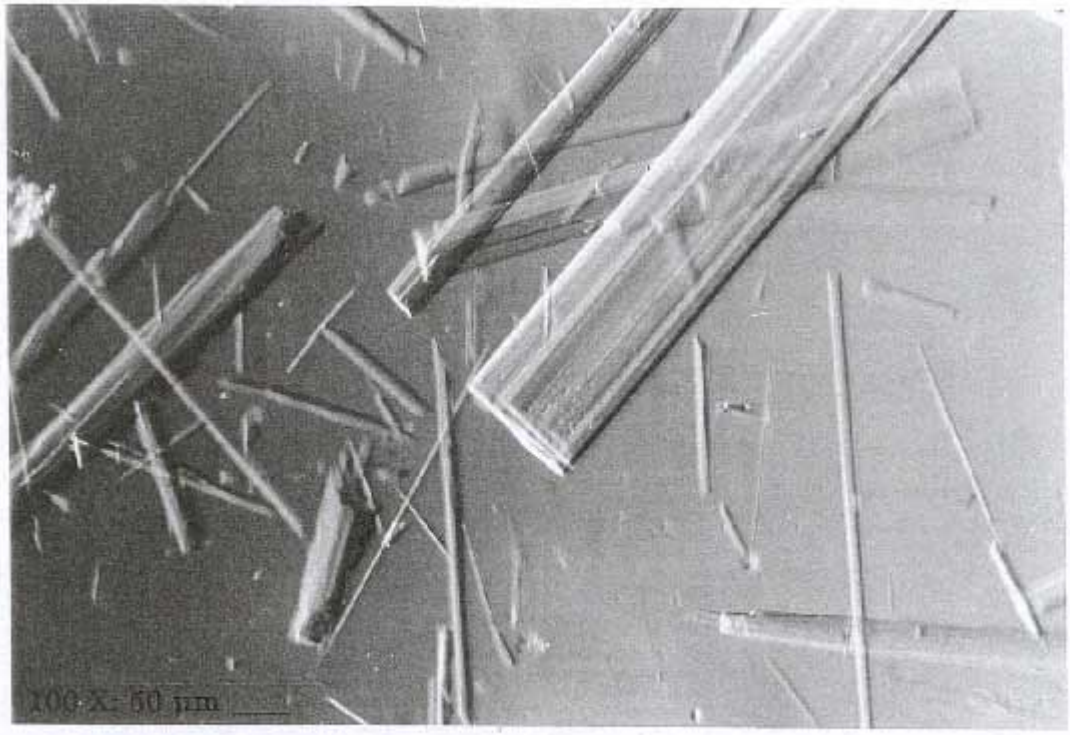

Fig. 3. Lamellas and acicular crystals of tremolite defined as "acicular tremolite". Ala de Stura, Italy. Optical microscopy, oblique illumination. Photo Ole Jørgensen, AMI.

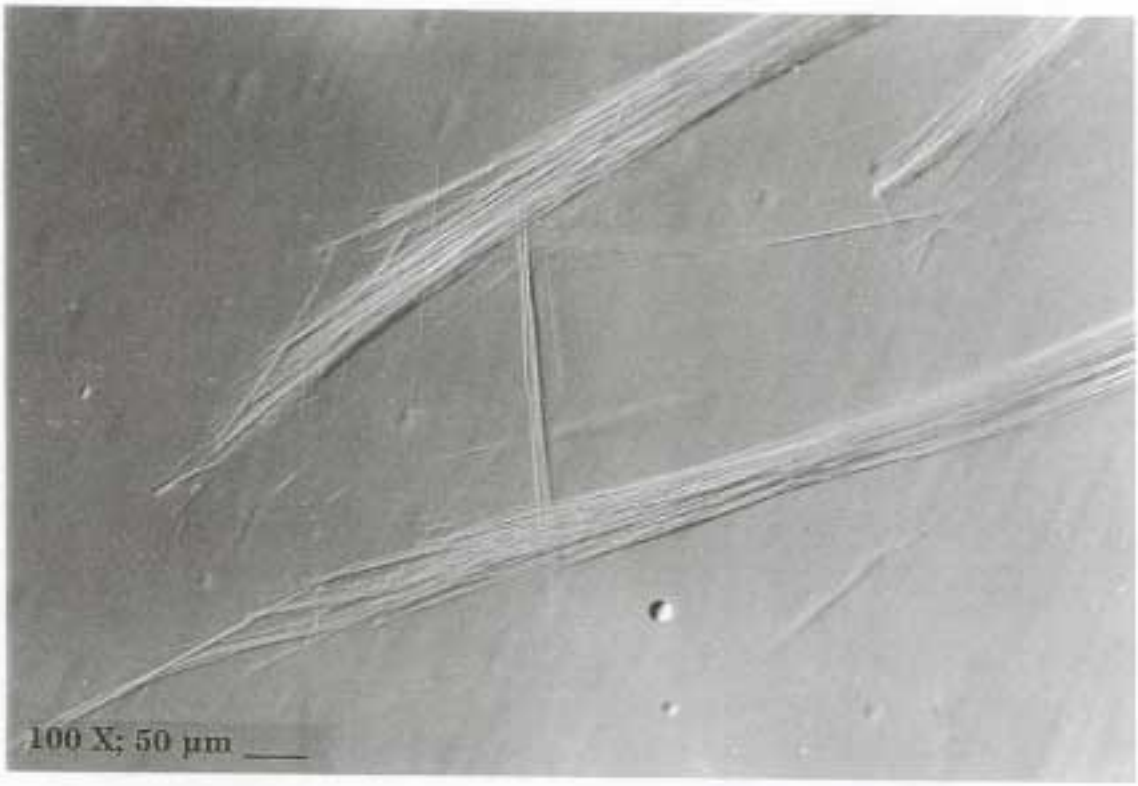

Fig. 4. Single fibres and bundles of fibres of tremolite defined as "asbestoid tremolite". Jamestown, California. Optical microscopy, oblique illumination. Photo Ole Jørgensen, AMI.

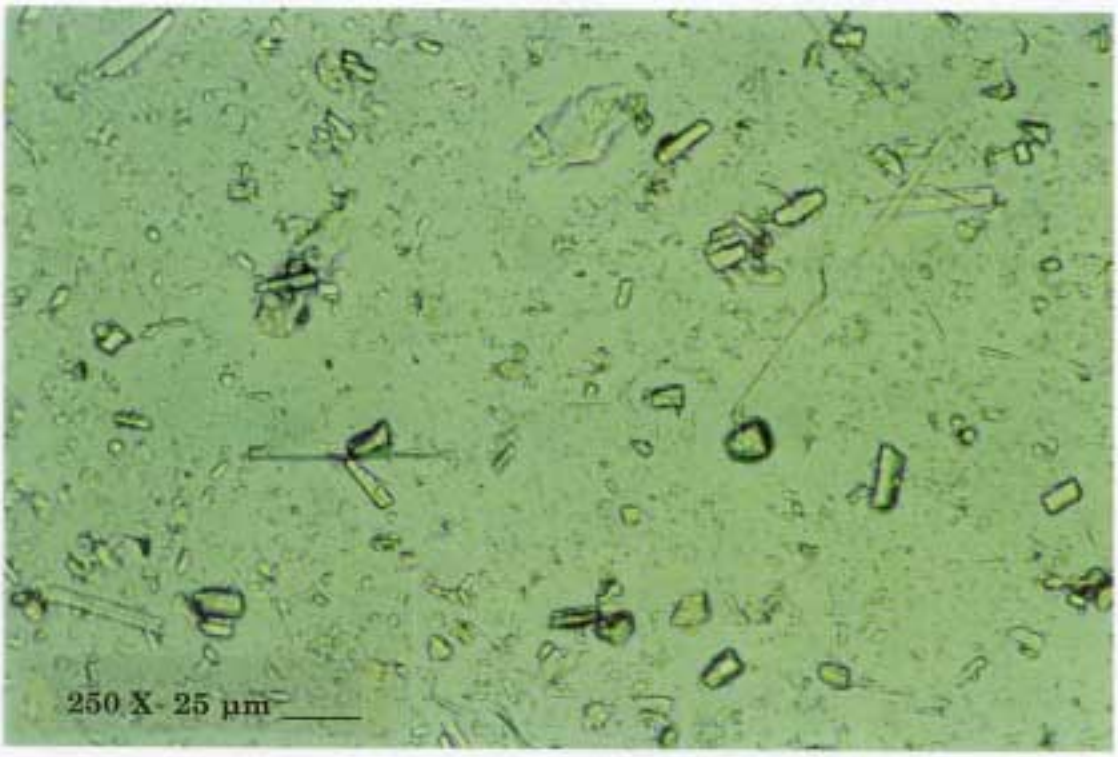

Fig. 5A. Particles of a talc containing filler deposited on the surface of a mixed cellulose ester filter.

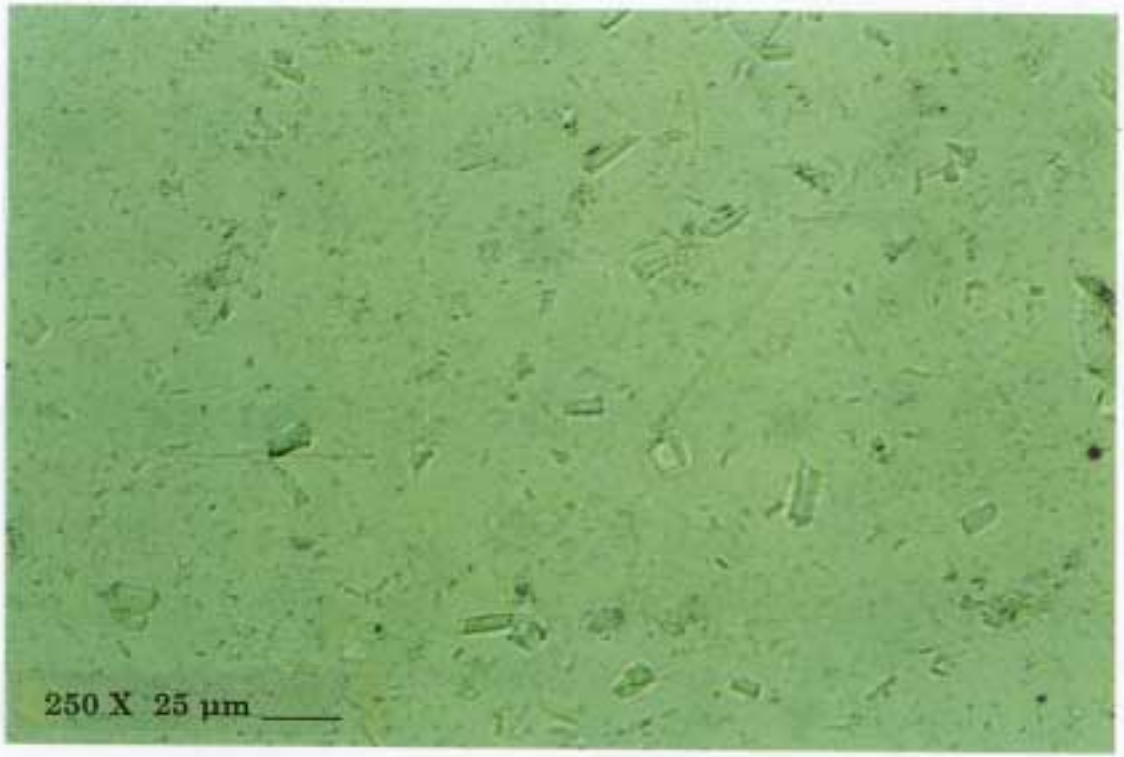

Fig. 5B. The same motive as above, but a droplet of immersion oil is now placed upon the filter. The refractive index of the oil is 1.600. The result is that transitional fibres of talc become nearly invisible.

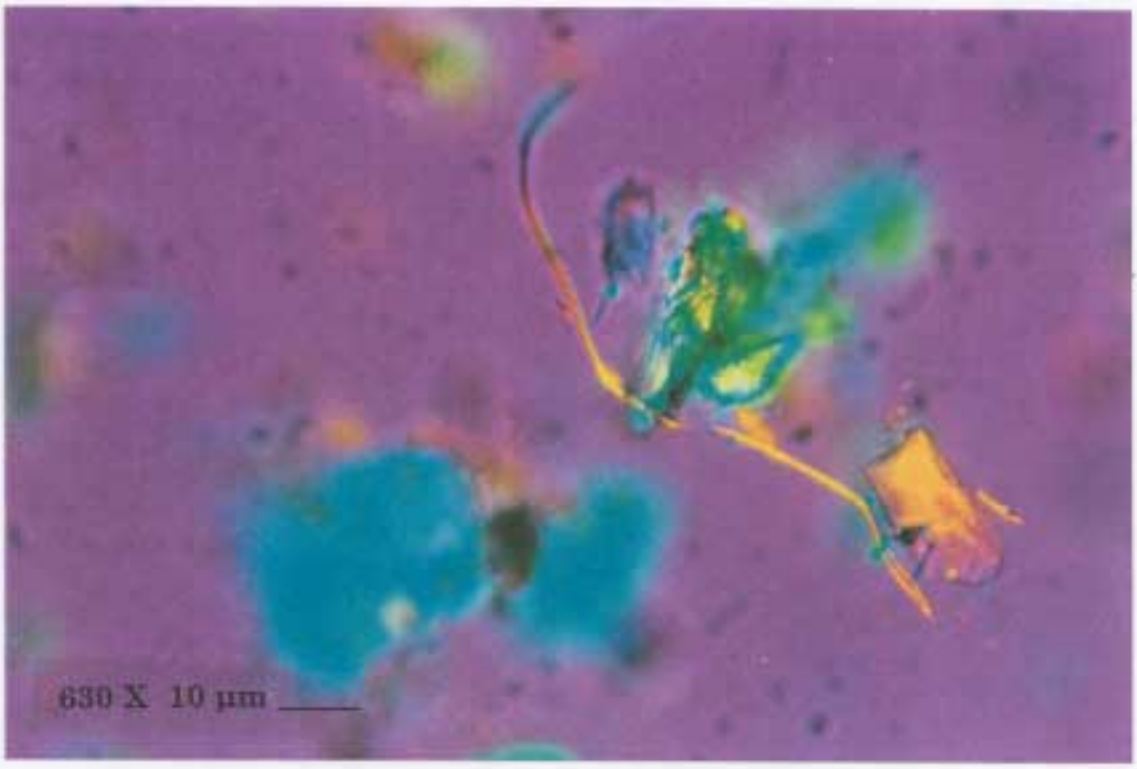

Fig. 6A. Sample 904-2. Fibre of sepiolite. PLM. Magnification 630 times.
Sepiolite has the same morphology as chrysotile, but the refractive index is less than that of chrysotile.

Fig. 6B. Sample no. 904-2. Fibres of sepiolite. SEM. Magnification 5000 times.

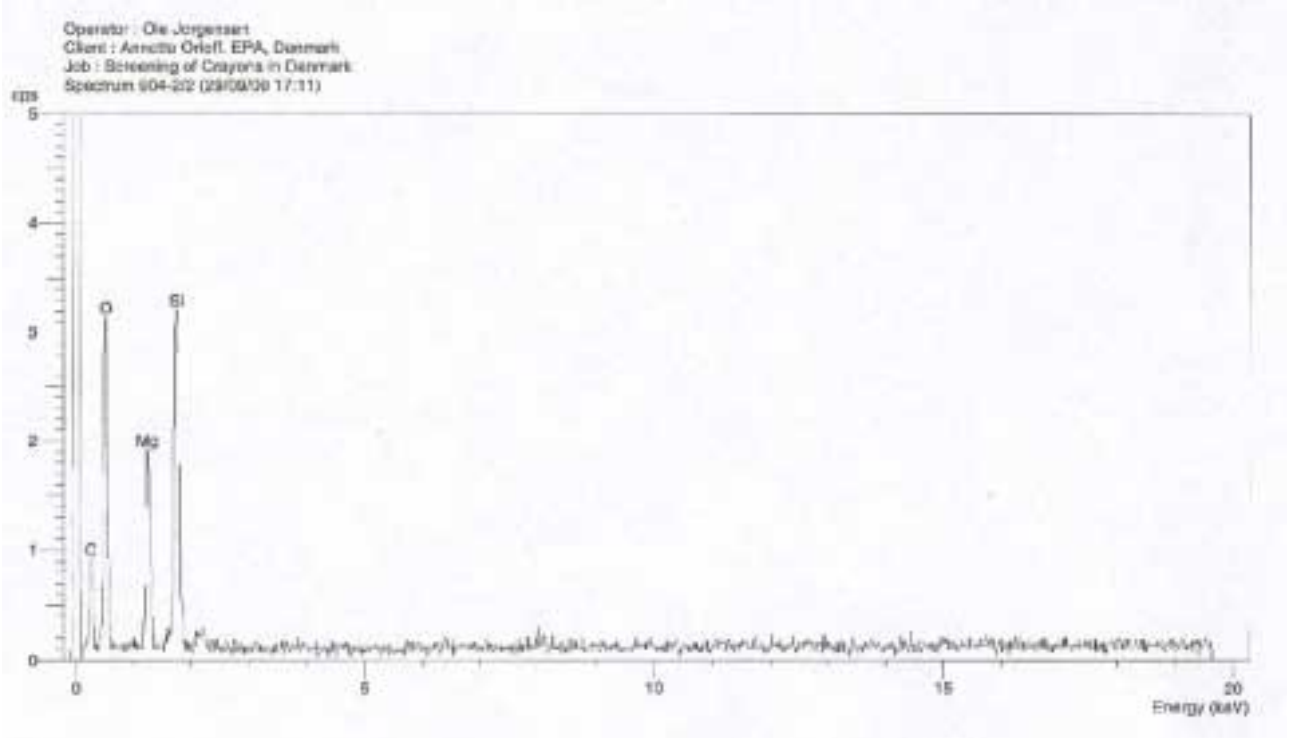

Fig. 6C. Sample no. 904-1. Element spectrum of sepiolite, $Mg_4\,(OH)_2\,[Si_6O_{15}]6H_2O$

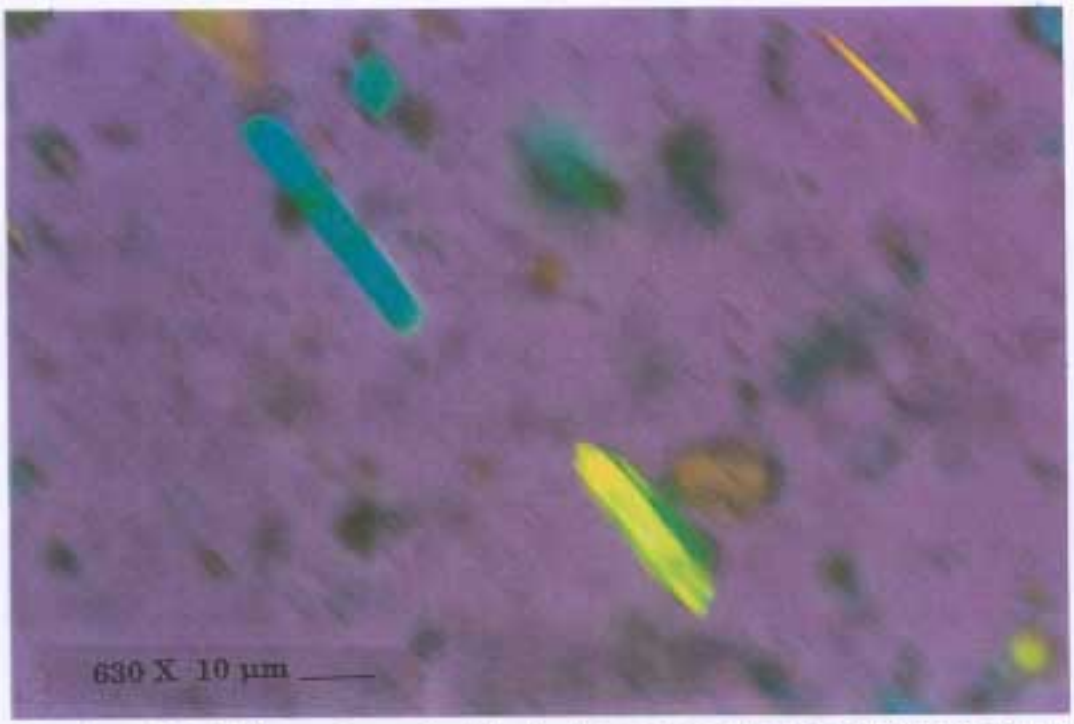

Fig. 7A. Sample no. 909-1. Fibres of nemalite (blue) and transitional fibre of talc (yellow) observed through a gypsum plate. PLM. Magnification 630 times.
The morphology and the refractive indices of nemalite and transitional fibre of talc are nearly identical, but the orientation of the vibration direction of light is different in the two minerals. The elongation of nemalite is negative (length fast) while it is positive (length slow) for transitional fibres. When the interference colour red I is added to the fibres in the same orientation, nemalite becomes blue and transitional fibre of talc yellow.

Fig. 7B Sample no. 909-1. Fibres of nemalite. SEM. Magnification 5000 times.

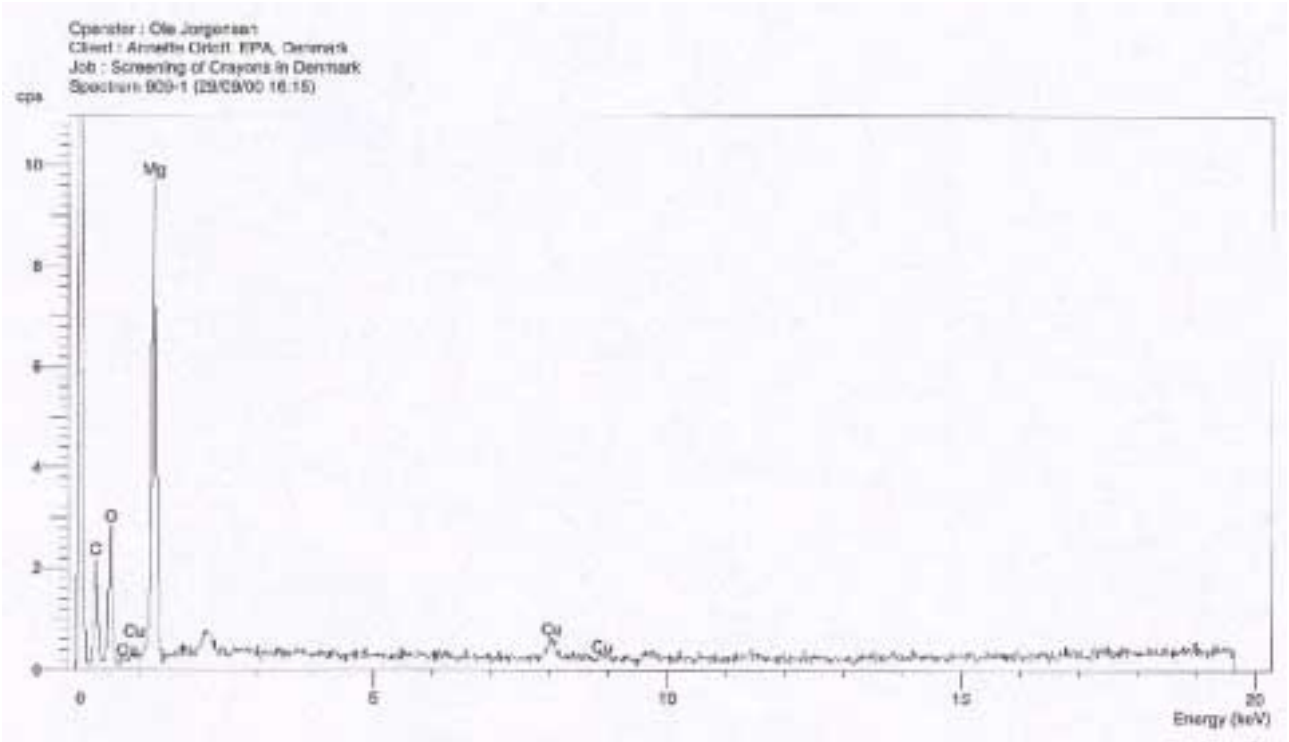

Fig. 7C. Element spectrum of nemalite, $Mg(OH)_2$.

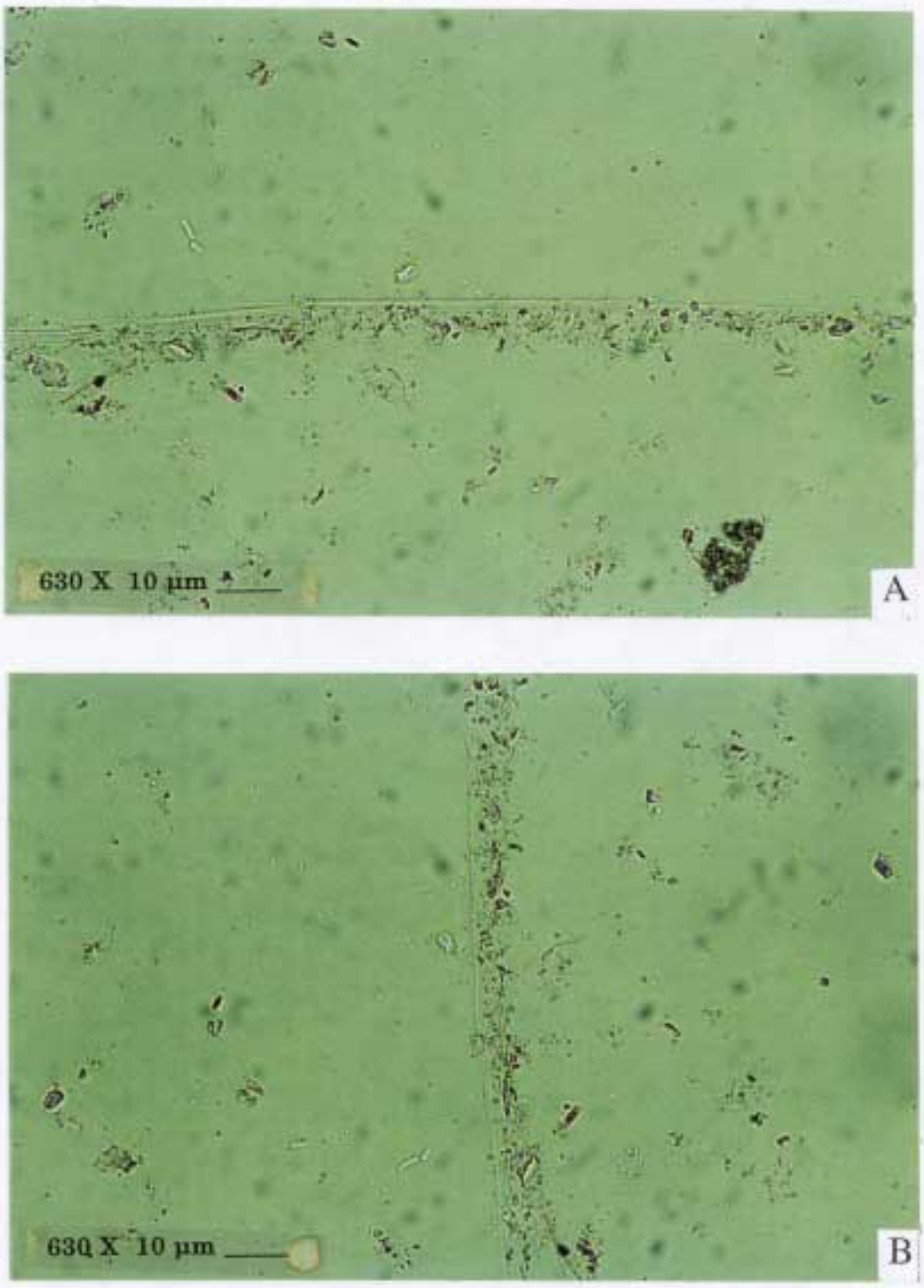

Fig.8. Sample no. 910-1. Tremolite asbestos observed by PLM. Magnification 640 times. N_{liq} = 1.600.

The highest refractive index of tremolite asbestos (n_γ = 1.622) is parallel to the length of the fibre. Perpendicular to the fibre direction the index of refraction is about 1.600. Consequently, when tremolite is immersed in a liquid with n = 1.600 and the fibres are oriented parallel to the direction of polarisation of the microscope the fibres are visible (A). Perpendicular to the length of the fibre, the fibres are invisible (B).

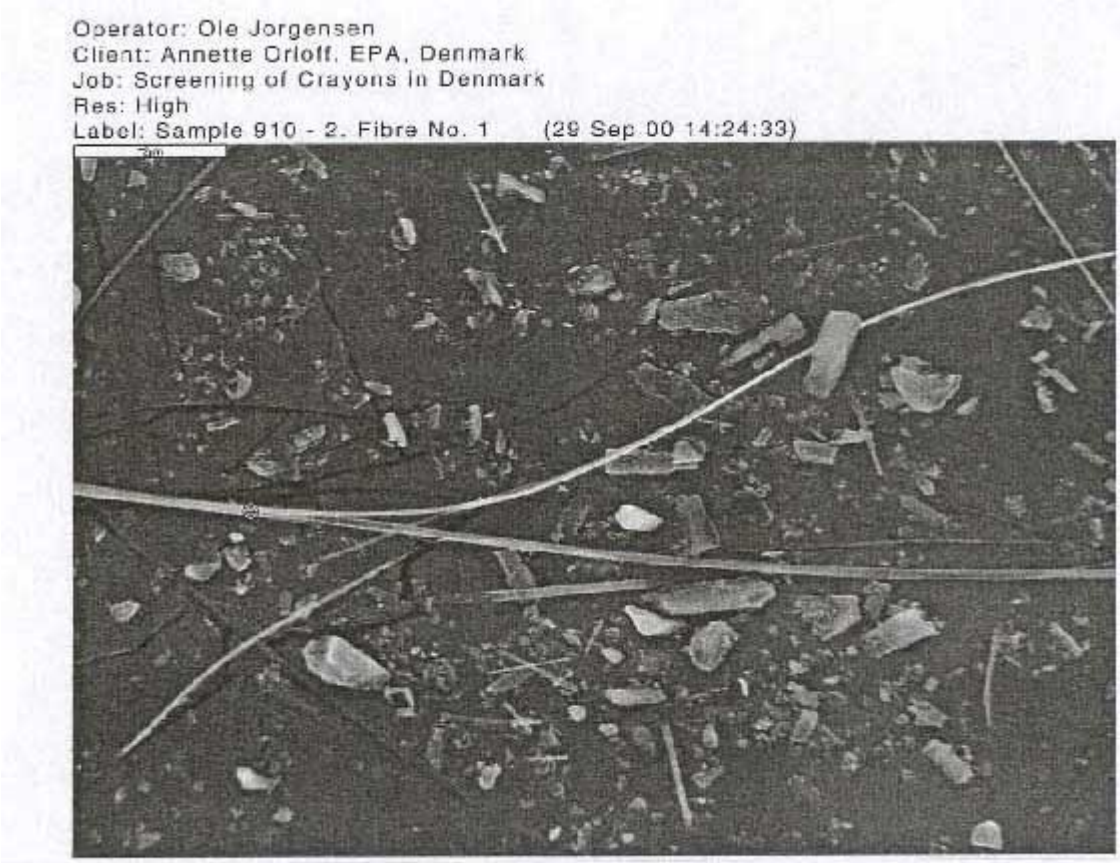

Fig. 9A. Sample no. 910-2. Fibres of tremolite asbestos. SEM. Magnification 10.000 times.

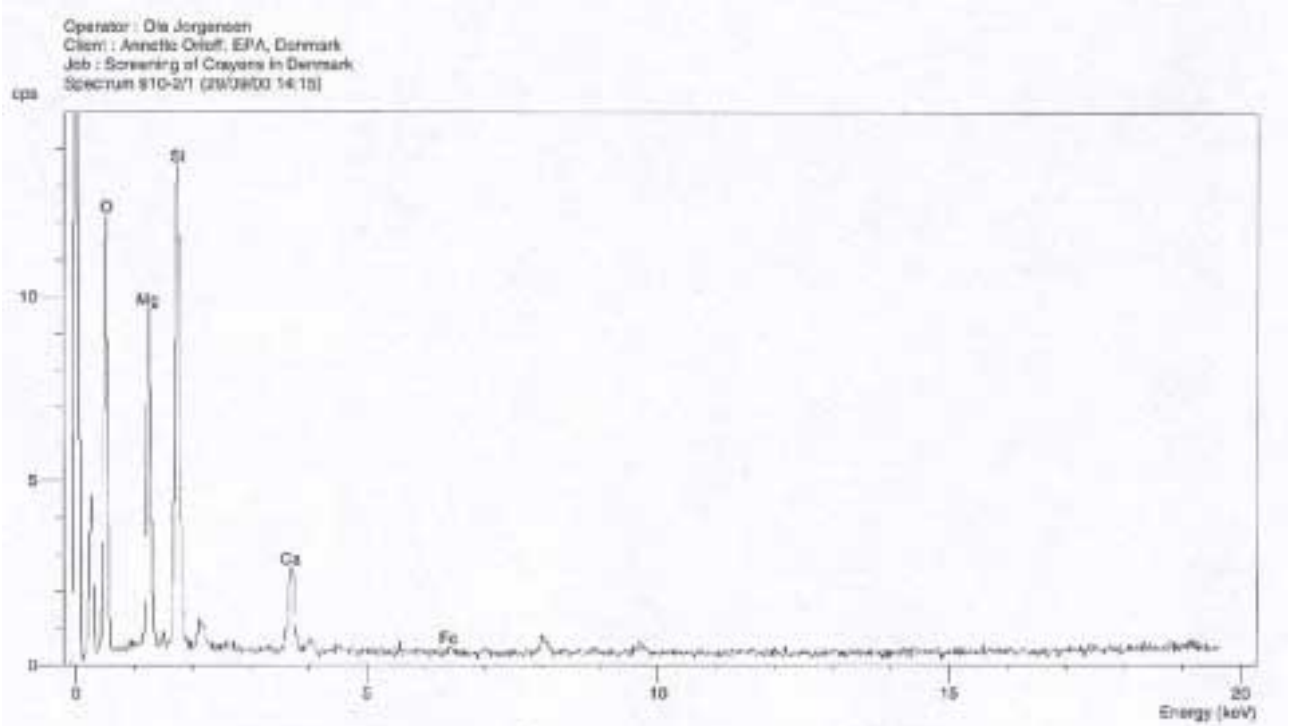

Fig. 9B. Sample no. 910-2. Element spectrum of tremolite, $Ca_2(Mg,Fe)_5[Si_8O_{22}](OH)_2$

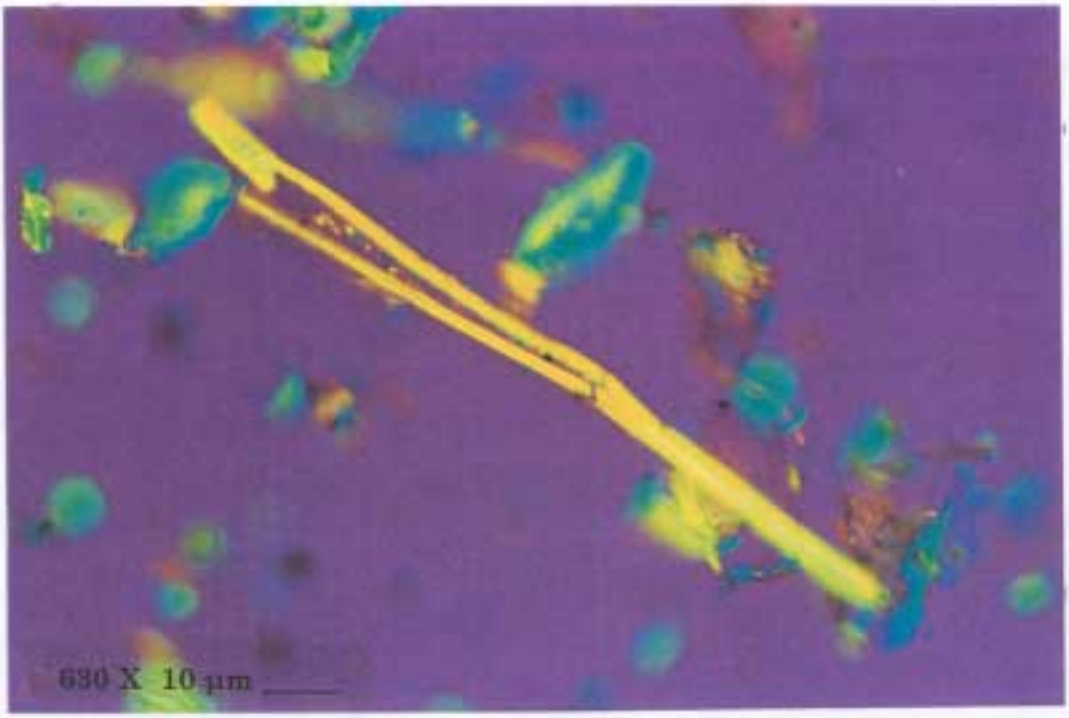

Fig. 10. Sample no. 910-3. Crysotile fibres observed by PLM. Magnification 640 times.

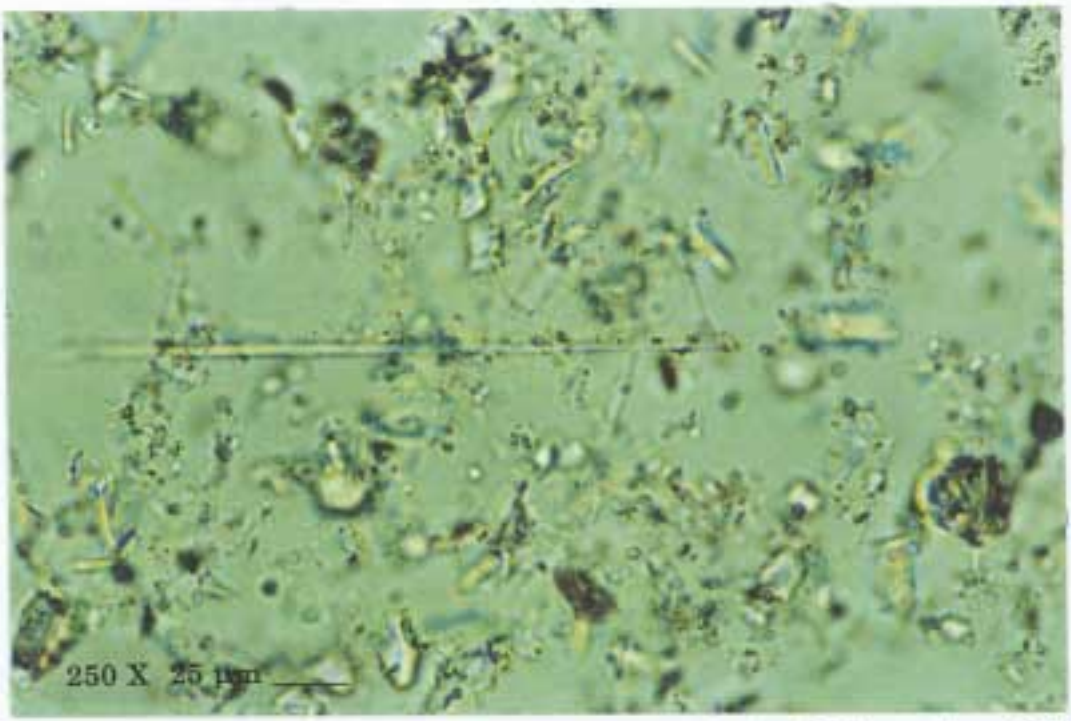

Fig. 11. Sample 910-3. Transitional fibre observed by PLM. Magnification 250 times. $N_{liq} = 1.600$.

Most transitional fibres become nearly invisible when the fibres are immersed in a liquid with a refractive index of 1.600. However, there exist some transitional fibres of which the refractive index varies along the direction of the fibre. When such a fibre is oriented parallel to the polarisation direction of the microscope, the fibre is visible at one end, but not in the other one.