Identification of asbestos in construction materials

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Abstract

Asbestos is the group name of six different fibrous minerals: chrysotile, amosite, anthophyllite asbestos, tremolite asbestos, actinolite asbestos and crocidolite. The nonfibrous forms of these minerals are not classified as asbestos. The asbestos minerals have a special kind of fibrous morphology called asbestiform. Asbestiform habit consists of fibres or bundles of fibres that easily split lengthwise into thinner fibres. The classification of a mineral as asbestos is based on a legal definition, not a mineralogical, as there exists other non-asbestos asbestiform minerals.

Asbestos was used in a wide variety of products and materials throughout the 20th century. Today, asbestos is mostly found in old buildings and building materials. With the demolition and renovation of these old buildings there is an increasing need to inspect the materials, as they might contain asbestos fibres. This inspection is done to prevent worker exposure and to limit the spread of asbestos fibres in the environment.

Most of the asbestos mined in the 20th century was chrysotile with some amosite, crocidolite and anthophyllite asbestos. The main producers of asbestos fibres were Canada, Russia, Australia and South Africa. Finland had the largest asbestos mine in Europe. It was situated in Paakkila, eastern Finland, and mined anthophyllite asbestos. It was active from 1904–1975.

Inhalation of asbestos fibres have been linked to several health issues. The asbestos fibres are deposited deep in the lungs and cannot be removed by the body. The body tries to expel the fibres but, due to their size and chemical resistance, the fibres cannot be removed naturally. Asbestosis occurs when the gas-exchange region of the lungs is blocked by asbestos fibres. The most common types of cancer associated with exposure to asbestos are lung cancer and mesothelioma. Mesothelioma is a cancer of the tissue lining the lung and the only known cause of mesothelioma is exposure to asbestos. Mesothelioma has an onset of up to 40 years after asbestos exposure.

In Finland, the two most common methods for asbestos identification and analysis in construction materials is polarized light microscopy (PLM) and scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS). In this thesis, these two methods were compared by analysing samples of construction materials. The samples were analysed for asbestos content and if asbestos was detected, the asbestos mineral was also determined.

The PLM method was developed from international standards and guides issued by USA and UK health authorities. The method was then compared to the SEM-EDS method. The SEM-EDS method has been in use at Top Analytica since 2016 and it is known to be accurate. Sample preparation is critical and was improved for both methods. In the analysis of samples, the PLM proved to be less accurate than the SEM-EDS method. The advantage of the PLM method is a faster and cheaper analysis. In the PLM method, the analyst's skill plays a significant role.

Swedish summary - Svensk sammanfattning

Identifiering av asbest i byggnadsmaterial

Asbest är en gemensam benämning för sex olika mineral: krysotil, amosit, antofyllitasbest, tremolitasbest, aktinolitasbest och krokidolit. Dessa mineral förekommer i en specifik fibrig form som kallas asbestiform. Asbestiformens habitus är speciell för att fibrerna klyvs betydlig lättare på längden än på bredden, vilket leder till att fibrerna kan fördela sig till submikroskopiskt tunna fiber. Asbestmineralen klassas som serpentin eller amfibol på basis av deras kristallstruktur. Krysotil är det enda asbestmineralet som tillhör serpentin-gruppen.

Den fibriga formen är orsaken till att mineralen klassificeras som asbest. Asbest klassificeras inte enbart på basis av mineralogi eftersom det finns andra mineral med liknande fibrighet, såsom erionit och fluoro-edenit. Dessa mineral klassas inte som asbest i vanliga fall. Den ursprungliga klassificeringen har uppkommit som följd av industriell användning av mineralfiber på 1900-talet. Definitionen av asbest är stadgad i lagen.

Asbestfibrerna har använts inom industrin på grund av deras fysiska och kemiska egenskaper. Asbestmineralen tål höga temperaturer, slitage och kemisk nedbrytning. Det är också dessa egenskaper som gör asbest vådligt för hälsan. Asbest har använts i allt från textil till fyllnadsmaterial i plaster och cementprodukter. Det största användningsområdet för asbest har varit inom byggnadsbranschen.

Asbest orsakar skada vid inandning av fibrerna. De långa och smala fibrerna fastnar i de djupaste delarna av lungorna. På grund av asbestfibrernas tålighet och längd kan kroppen inte bryta ner eller transportera dem. Detta leder till en kronisk inflammation då kroppen försöker bryta ner fibrerna. Till sist blir asbestfibrerna täckta av ärrvävnad och denna ärrvävnad minskar på lungornas kapacitet att uppta syre. Asbestos är en sjukdom som orsakas när asbestfibrerna förstört en stor andel av lungornas syreupptagningsförmåga. Cancer är också kopplat till inandning av asbestfiber. Lungcancer är den vanligaste cancern som uppstår på grund av asbestinandning. Mesoteliom är en ovanlig form av cancer som uppkommer i vävnaden kring lungorna. Den orsakas endast av asbest. Mesoteliom kan utvecklas upp till 40 år efter man blivit exponerad för asbest.

Det finns bevis på att asbest har använts småskaligt från och med ca 2000 f.Kr. Den industriella användningen av asbest började i Italien i slutet av 1800-talet, då främst textiler och rep tillverkades. Under första halvan av 1900-talet ökade användningen av asbest betydligt och allt fler användningsområden utvecklades. Det var speciellt inom byggnadsbranschen som man utvecklade nya användningsområden. Asbest användes också i bland annat bromsbelägg och cigarrettfilter. I Finland påträffas asbest i huvudsak i byggnader och byggnadsmaterial.

Över 90 % av all asbest som producerades under 1900-talet var krysotil. De största producenterna av asbestfiber var Kanada och Ryssland som producerade krysotilfiber. Australien och Sydafrika producerade stora mängder amosit- och krokidolitfiber. I Finland fanns Europas största asbestgruva i Paakkila, östra Finland. Den var aktiv mellan åren 1904–1975 och producerade ca 350 000 ton antofyllitasbest. I dag produceras asbest främst i Asien varav Kina, Ryssland och Kazakstan är huvudproducenter. Brasilien är den största asbestproducenten utanför Asien.

Produktionen och användningen av asbest ökade kraftigt från början av 1900-talet. Under 1970-talet insåg man hälsoriskerna med asbest och i Europa och Nordamerika infördes asbestförbud. I EU kom det slutliga förbudet på all försäljning, produktion och användning av asbest i kraft år 2005. Endast ett fåtal tillfälliga undantag från lagen beviljades. I Finland förbjöds asbest fullständigt år 1994. I både USA och Kanada är asbest bara delvis förbjudet men båda länderna planerar att förbjuda asbest i all form.

Asbest är fortfarande relevant idag, 20 år efter att det förbjudits, eftersom äldre byggnader har byggmaterial som innehåller asbest. Vid rivning eller renovering av dessa byggnader måste allt material analyseras som kan tänkas innehålla asbest. I analysen undersöker man om materialet innehåller asbest och om det innehåller asbest så identifieras asbestmineralet. Denna avhandling jämför de två vanligaste metoderna som används för denna typ av asbestanalys.

Metoderna som jämförs är polariserat ljus (PLM) och elektronmikroskop med grundämnesanalysator (SEM-EDS). Under denna avhandling utvecklades PLM-metoden som ett snabbare och billigare alternativ till SEM-EDS-metoden. SEM-EDS-metoden är den nuvarande metoden som används vid Top Analytica. PLM-metoden utvecklades på basis av internationella standarder (ISO 22262–1:2012) och instruktionsmaterial från USA:s och Storbritanniens hälsomyndigheter. Mikroskopet som användes för största delen av analyserna var ett

Leica 12 POL-mikroskop utan specialutrustning för asbestidentifikation. Specialutrustningen gör det möjligt att urskilja de olika asbestmineralen betydligt lättare. Senare användes ett Leica DM 2700 P-mikroskop med utrustning för asbestidentifikation. Raman-spektroskopi användes endast på referensprov.

Totalt undersöktes 193 prov och 24 kontrollprov. Proven bestod av olika sorters byggnadsmaterial som kan innehålla asbest. Om provet innehöll asbest bestämdes också asbestmineralet. Proven som undersöktes var byggnadsmaterial som Top Analyticas kunder beställt analys på. Kontrollprover från Health and Safety Executive (arbetshälsomyndigheten) i England användes för att bekräfta metodernas pålitlighet. PLM- och SEM-EDS-metoderna utfördes vid Top Analyticas laboratorium. SEM-EDS-metoden har varit i användning vid Top Analytica sedan 2016 och användes som en kontroll för den nyutvecklade PLM-metoden.

Båda metoderna visade sig vara användbara vid identifikation av asbestfibrer. PLM-metoden hade sämre noggrannhet vid identifikation, vilket i huvudsak berodde på bristfällande provpreparation. PLM-metoden kräver mycket noggrann förberedelse av provet. Prepareringen visade sig vara en mycket viktig del för båda metoderna.

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1 Introduction

Asbestos is the group name of six different silicate minerals. The six asbestos minerals are: chrysotile, amosite, anthophyllite asbestos, tremolite asbestos, actinolite asbestos and crocidolite. The asbestos minerals have a special form of fibrous habit called asbestiform, which is defined as a fibre or bundle of fibres that are easily separated lengthwise. The individual fibres consist of aggregates of even smaller fibres. The fibres are heat- and chemically resistant, non-conductive and have great tensile strength (Strohmeier et al., 2010). These physical properties together with inexpensive production have resulted in the widespread use of asbestos.

The classification of a mineral as asbestos is a legal definition, not a mineralogical. Several minerals exists that show asbestiform habit but are not classified as asbestos. This is due their lack of industrial use. The six regulated asbestos minerals have been in industrial use since the beginning of the 20$^{\text{th}}$ century. In Finland, a zeolite called erionite is also legally classified as asbestos (FINLEX, 2015b).

The asbestos minerals can also be classified by crystal structure into amphiboles or serpentines. Chrysotile is the only serpentine mineral while the rest belong to the amphibole group. The crystal structure and morphology are important, because only the asbestiform variants of the minerals are asbestos. Non-asbestiform variants exists of all six asbestos minerals. Sometimes the transition of a mineral from non-asbestos to asbestos is unclear, as these are naturally occurring materials (Virta, 2002; Gunter et al., 2007).

In the latter half of the 20$^{\text{th}}$ century, asbestos was shown to be a health hazard (Ross and Nolan, 2003). Asbestos is known to cause asbestosis, lung cancer and mesothelioma with prolonged exposure to airborne fibres. When inhaled, the fibres are deposited deep in the lungs and cannot be removed naturally by the body. With the accumulation of fibres, the gas-exchange region is blocked and the lung is in chronic inflammation. The smallest fibres can also enter the cells lining the lung and this eventually leads to cancer. This is known as mesothelioma and is always fatal. Smoking suppresses the lungs natural defences against dust and significantly increases the health risks associated with asbestos. Chrysotile asbestos is thought to be the least hazardous of the minerals but there is no safe limit for asbestos exposure (Ross and Nolan, 2003). Chrysotile is also the most commonly used asbestos in the 20$^{\text{th}}$ century (Virta, 2006).

The purpose of this master's thesis is to compare the detection and identification of asbestos fibres in construction materials with different methods. The two methods compared are polarized light microscopy (PLM) and scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS). The samples are different types of construction materials from buildings built before 1994. In Finland all production, sale and use of asbestos was completely banned in 1994 (FINLEX, 1992). From these materials a small sample is brought to the lab for analysis. The analysis looks for asbestos fibres in the sample and if such fibres are found, the type of asbestos mineral is identified. The amount or concentration of fibres in the sample is not part of the analysis. This thesis also compares the different preparation methods required for the two methods.

2 Asbestos minerals

2.1 Chrysotile

Chrysotile is the only non-amphibole of the six minerals that are regulated as asbestos (Ross and Nolan, 2003). The ideal chemical formula of chrysotile is: $Mg_3Si_2O_5(OH)_4$. Chrysotile is referred to as ”white asbestos” due to its white-greyish colour. Chrysotile belongs to the serpentine group of minerals and consists of layers that are rolled up to look like a scroll. The mineral structure of chrysotile is scroll-like due to length difference in the atomic layers. This causes the crystal structure to bend into rolls (Virta, 2002). The roll-like structure leads to chrysotile having a woolly and textile fibre-like look macroscopically.

Chrysotile consists of sheets of silicate tetrahedra with brucite layers in-between (Figure 1). This brucite layer makes chrysotile slightly soluble in acid (Virta, 2002). Chrysotile accounts for over 90% of all asbestos used worldwide and is still mined in large-scale operations today. Chrysotile is seen as potentially the least harmful of the asbestos minerals (Ross and Nolan, 2003).

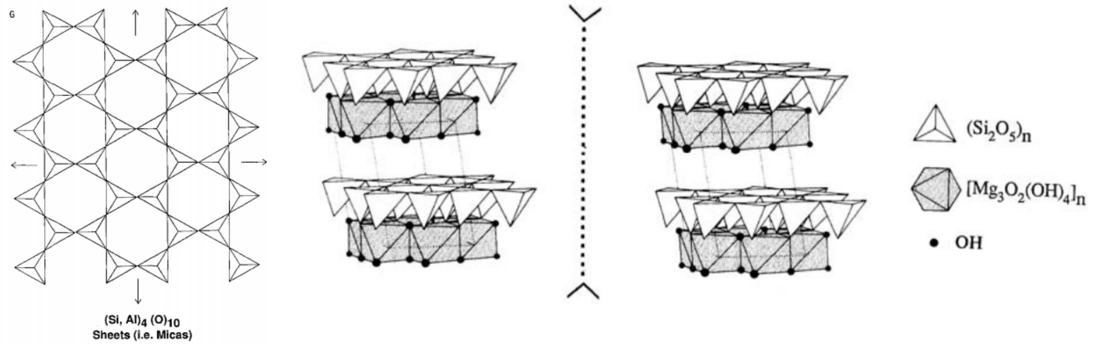


Figure 1: The crystal structure of chrysotile (Skinner et al., 1988).

2.2 Amosite

Amosite is an amphibole mineral and the fibrous form of the solid solution of the minerals grunerite and cummingtonite. Amosite is found in the iron-rich grunerite endmember and the ideal formula is: $Fe_7Si_8O_{22}(OH)_2$. Amosite has a brownish colour. Amosite is a double chain silicate like the other amphibole asbestos minerals (Figure 2).

The name amosite is an acronym for “Asbestos Mines of South Africa” and it was mostly mined in South Africa (Gunter et al., 2007).

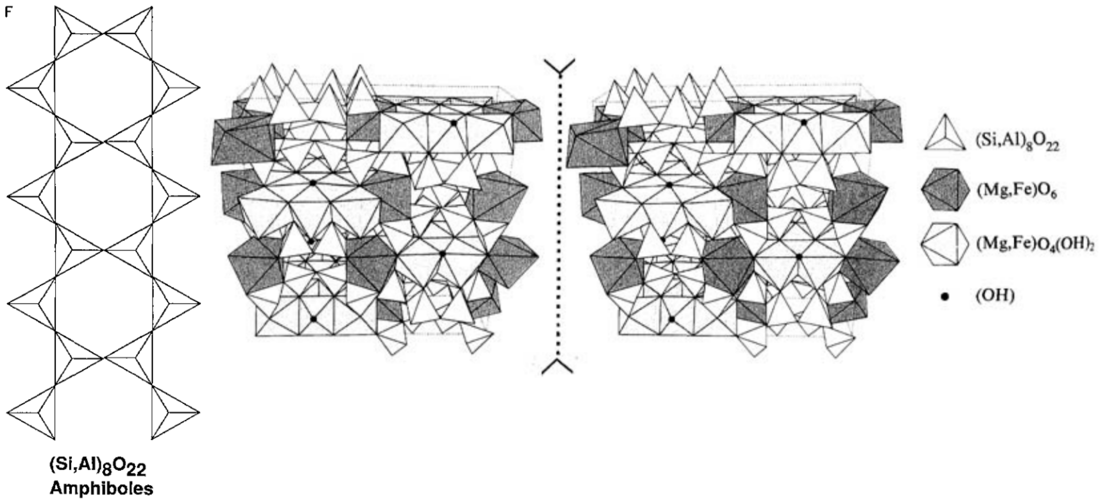


Figure 2: The general crystal structure of amphibole asbestos minerals (Skinner et al., 1988).

2.3 Anthophyllite asbestos

Anthophyllite asbestos is an amphibole and the asbestiform form of the mineral anthophyllite. The ideal formula is: $(Mg,Fe)_7Si_8O_{22}(OH)_2$. Anthophyllite has a grey-brown colour. Anthophyllite is chemically like the Mg-rich endmember cummingtonite in the cummingtonite-grunerite solid solution, but it has an orthorhombic crystal system. The other amphibole asbestos minerals have a monoclinic crystal system.

Anthophyllite was sparsely mined commercially and mostly occurs as a contaminant in other asbestos minerals and talc (Gaffney et al., 2017). Finland had a large anthophyllite mine at the beginning of the 20th century, but it was closed in 1975 (Nikkarinen et al., 2001; Virta, 2006).

Anthophyllite is considered potentially the least harmful of the amphibole asbestos minerals (Gaffney et al., 2017).

2.4 Tremolite and actinolite asbestos

Tremolite asbestos and actinolite asbestos have only been sparsely mined worldwide. They most commonly occurred as contaminants with other asbestos minerals (Gunter et al., 2007).

Tremolite asbestos is an amphibole and the fibrous form of the mineral tremolite. The ideal formula is: $Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$. Tremolite asbestos has a grey-brown colour. Canadian chrysotile has a small amount of tremolite asbestos and it is thought to be causing most of the asbestos-related health problems in the asbestos mining region (Gunter et al., 2007).

Actinolite asbestos is an amphibole and the fibrous form of the mineral actinolite. The ideal formula is: $Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$. Actinolite is also pleochroic with a grey-green colour.

Tremolite and actinolite are both part of the same solid solution between tremolite and ferro-actinolite where the magnesium rich end is tremolite. With increasing iron content, it goes to actinolite and lastly ferro-actinolite (Ross and Nolan, 2003; Verkouteren and Wylie, 2000).

Today, small-scale mining of tremolite and actinolite asbestos is still ongoing in India (Ramanathan and Subramanian, 2001).

2.5 Crocidolite

Crocidolite is an amphibole and the fibrous form of the mineral riebeckite. The ideal formula is: $Na_2(Fe_3^{2+}Fe_2^{3+})Si_8O_{22}(OH)_2$. Crocidolite is referred to as the ”blue asbestos” due to its deep blue colour. Crocidolite is pleochroic and shifts between blue and grey.

The largest producers of crocidolite were South Africa and Australia with small-scale mining in Bolivia. Crocidolite was used in many products such as cigarette filters, gas masks and fireproofing (Virta, 2002).

Crocidolite is considered the most harmful of the asbestos fibres (Gunter et al., 2007)

2.6 Other asbestiform minerals

The previously mentioned asbestos minerals are the most common and well-known naturally occurring mineral fibres. They are also the most extensively used natural mineral fibres, but there also exists other fibrous minerals that show the same kind of properties, such as strength and chemical resistance, as asbestos fibres. These non-asbestos asbestiform minerals have not been commercially exploited for their properties. They are not classified as asbestos legally but mineralogically they would be classed together with the asbestos minerals (Gunter et al., 2007). Known minerals with fibrous, asbestiform habit are for example: erionite (Dogan, 2003), fluoro-edenite (Gianfagna and Oberti, 2001), winchite and richterite (Gunter et al., 2007). Erionite is a zeolite while the others are amphiboles. Due to the chain silicate structure of amphiboles there might exist more asbestos-like minerals then are currently known. The health hazard of these minerals is like the regulated asbestos minerals (Gunter et al., 2007).

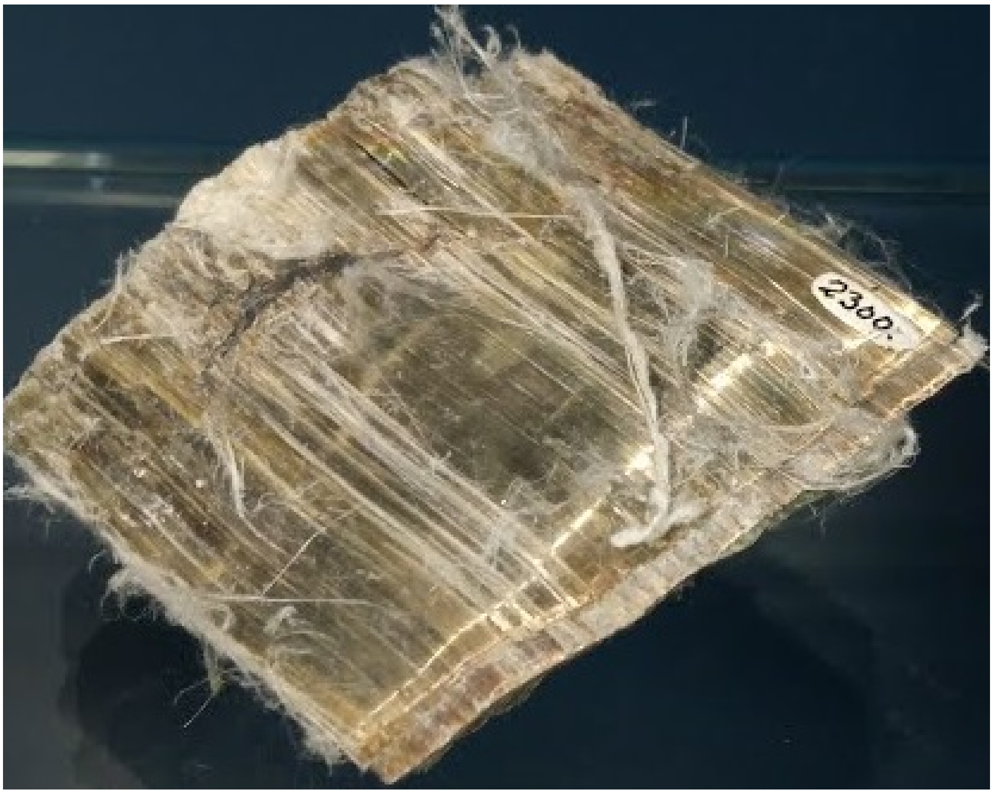
2.7 Morphology and occurrence of asbestiform fibres

Asbestos minerals have a unique fibrous morphology that is known as asbestiform. The fibrous classification is broader and includes all kinds of thin elongated particles, e.g. prismatic and acicular habits. The acicular habit consists of long, thin, needle-like mineral particles commonly found in the amphibole minerals. Some asbestos minerals look more acicular than fibrous in macroscopic scale. One example is the Finnish anthophyllite (Figure 3a).

Asbestiform habit shows excellent cleavage lengthwise while being highly resistant to sideways splitting. Lengthwise splitting into thinner fibres can occur down to a sub-microscopic level. This preference for lengthwise splitting is seen in macroscopic scale as bundles of small fibres, needles or a woolly appearance that characterizes some asbestos minerals (Figure 3b) (Strohmeier et al., 2010).

The asbestos minerals are white to brownish in colour except for crocidolite that has a dark blue colour (Strohmeier et al., 2010). High temperatures can change the colour of crocidolite to brownish.

(a) Finnish Anthophyllite



(b) Chrysotile from Russia

Figure 3: (a) Anthophyllite containing rock from the Paakkila mine, Finland. (b) Chrysotile has often a woolly appearance. Chrysotile ”ore” from the Urals, Russia. Pictures taken by author.

All asbestos minerals are naturally occurring mineral fibres. These fibres form in magnesium- and iron-rich bedrocks. The most common host-rock for large-scale asbestos deposits are ophiolites, i.e. old seafloor that has been uplifted onto continents (Strohmeier et al., 2010). Only crocidolite and amosite occur in other host-rocks, both are known to form in banded-iron formations (Ross and Nolan, 2003).

In addition to requiring a certain type of local geology to produce the minerals, the bedrock also needs to have been subjected to tectonic activity. The most common mechanism that produces asbestos is strike-slip movements. The fibres form within the fractures that occur in the rock. A rock can contain up to several percentage of asbestos fibres by mass. The fibres can also be produced in the contacts of magma intrusions. As amphiboles are generally common in bedrocks around the world, so are small amounts of asbestos. (Strohmeier et al., 2010).

The non-regulated asbestiform minerals form in similar tectonic environments as the six asbestos minerals. For example, the mineral erionite occurs in an ophiolite in central Turkey. Erionite has not been used commercially for its fibres, but erionite containing rock has unknowingly been used as construction material. As these rocks were used in construction, the fibres within were released and this is today seen in the local cancer statistics. Erionite consists of fibres with a woolly appearance and is known to have asbestos-like impact on human health (Dogan, 2003).

Another accidentally mined amphibole with an asbestiform habit is fluoro-edenite. Fluoro-edenite is a fluorine-rich amphibole that is known to occur in large quantities on the southern slopes of Mt. Etna. A local quarry mined the fluoro-edenite containing rock for use in construction (Gianfagna and Oberti, 2001).

2.8 Classification and production of asbestos

The classification of asbestos is a legal definition and not a mineralogical. The basis for this classification was the commercial use of these six minerals during the 20^{th} century. In large-scale production were chrysotile, amosite and crocidolite. The other regulated asbestos minerals were only in small-scale production or occurred as contamination in the commercial minerals.

This legal classification was intended to regulate the use and exposure to the fibres that were released by mining and processing (Gunter et al., 2007). In the beginning of the 20th century some health problems associated with asbestos fibres were known, but it was an occupational hazard in the asbestos trade. The grouping of these fibrous minerals became the basis for their later regulation (Gunter et al., 2007).

Asbestos has been in worldwide production and consumption since the beginning of the 20th century (Virta, 2006). The peak for production and consumption occurred in the 1970s (Figure 4) and the asbestos industry was growing until new legislation in Europe and USA restricted its use significantly. At its peak, the top producers of asbestos were Canada, South Africa and Russia. It is estimated that during the 20th century, roughly 166 million tonnes of asbestos was produced, of which over 90% was chrysotile (Virta, 2006).

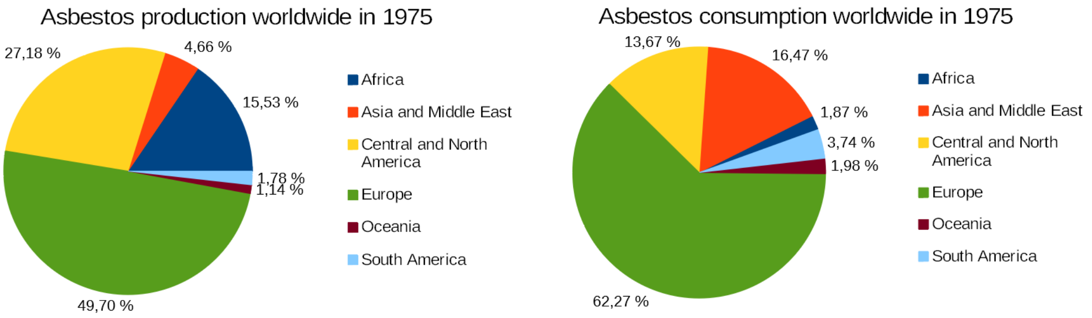


Figure 4: Asbestos use and consumption worldwide in 1975. The Soviet Union is included in Europe. Total tonnage for production: 4 212 740. Total tonnage for consumption: 4 331 210. (Virta, 2006).

Today, there is only significant asbestos mining in Russia, Brazil, China and Kazakhstan (Figure 5). Most asbestos is consumed locally in these countries but there is also some export to neighbouring countries. Today only chrysotile mines are in operation (Virta, 2006).

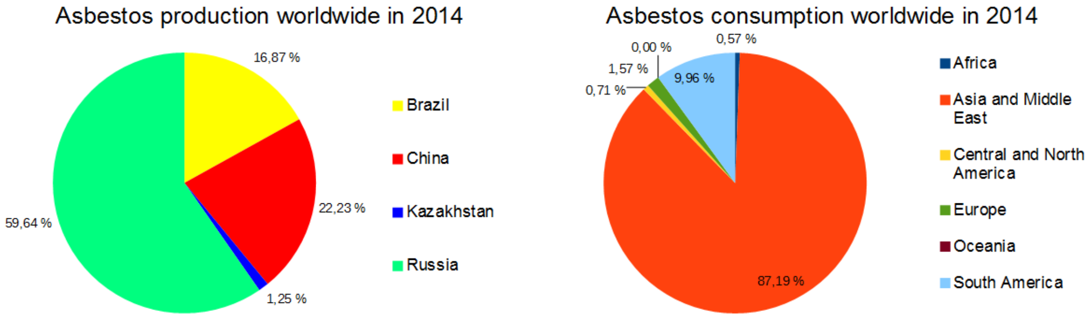


Figure 5: The main asbestos producers and consumers worldwide in 2014. Total tonnage for production: 2 018 530. Total tonnage for consumption: 1 844 330. (Flanagan, 2016)

In Finland, there has been two active asbestos mines, in Paakkila and Maljasalmi. Both are in eastern Finland, roughly 50 km east of Kuopio. The Paakkila mine was in operation from 1904 to 1975 while the Maljasalmi mine was only briefly in operation during the 1940s and 1950s. Both mines produced anthophyllite asbestos fibres. There was also several processing plants at the Paakkila mine that crushed, milled and processed the fibres from both mines (Nikkarinen et al., 2001). From 1918 to 1975 the Paakkila mine produced roughly 350 000 tons of anthophyllite asbestos fibres. The domestic consumption of this was 120 000 tons. The smaller Maljasalmi mine produced 61 000 tons of asbestos in its lifetime. At the time, the Paakkila mine was the biggest anthophyllite mine in the world (Nikkarinen et al., 2001).

2.9 Physical and chemical properties of asbestos minerals

Asbestos fibres are long, thin and flexible crystalline fibres that are heat, wear and chemically resistant. The fibres have a high tensile strength and large surface to mass ratio. A useful property of the fibres is that they can easily be bent without breaking and only split lengthwise into smaller fibres. This means they can be worked like other organic fibres and it is possible to make extremely durable textiles and ropes. The heat and wear resistance makes asbestos fibres ideal for fireproofing and reinforcement. The fibres are not electrically conductive and work well as both heat and electrical insulation in industrial applications (Virta, 2002).

The least chemically resistant of the asbestos fibres is chrysotile due to its different crystal structure compared to the amphibole minerals. The chrysotile fibre consist of sheets of silicon tetrahedra with layers of brucite (MgO) between the layers. Brucite is prone to dissolve from the fibre and this makes chrysotile fibres susceptible to acidic environments (Virta, 2002). As the human lungs can possibly dissolve the brucite layer, it is thought that chrysotile is the least harmful type of asbestos (Ross and Nolan, 2003). When the brucite layer leaves the crystal structure, all that remains is the hollow shell of silicon tetrahedras. This weakened structure easily falls apart, causing the fibres to disintegrate. Chrysotile also has the lowest heat resistance of all asbestos minerals and starts to convert to forsterite (Mg_2SiO_4) at 550°C (Virta, 2002).

The amphibole asbestos minerals consists of chains of silicon tetrahedra which is also referred to as inosilicates. These fibres are extremely resistant to chemical and high-temperature environments, which makes them useful in many industrial applications. The amphibole fibres are not susceptible to acidic environments like chrysotile is. The amphiboles also withstand higher temperatures and start to convert to pyroxenes at over 900°C (Gunter et al., 2007; Spasiano and Pirozzi, 2017). The amphibole minerals often occur as prismatic crystal shards and show a gradual transition from prismatic and acicular habit to asbestiform.

The amphibole group of minerals have a large variety in the chemical composition as several different elements can be substituted in the crystal lattice. Because of this, some of the asbestos minerals are formed from a solid solution series like for example the solid solution between cummingtonite and grunerite that forms amosite. The health impact of this chemical variation is unknown (Ross and Nolan, 2003).

The physical properties of the fibres vary somewhat with length and this has caused different lengths of asbestos fibres to be used for different applications. Longer fibres are possible to spin to textiles and ropes while medium to short fibres are used as reinforcement in cement, plastic and asphalt products. The shortest fibres and asbestos dust is used as filler in plastics, concrete and reinforcement applications (Virta, 2002).

3 Health impact of asbestos

3.1 Asbestos in the 20th century

Asbestos has been known to be hazardous since the beginning of the 20th century. In the beginning, it only affected the workers of the asbestos industry and was classified as an occupational hazard (Ross and Nolan, 2003). With the substantial increase in the use of asbestos in the latter half of the 20th century, more people outside the industry started to suffer from health problems due to asbestos. This was mostly due to the introduction of asbestos-containing products to the public (Virta, 2006). The conclusive studies of the harmful effects of asbestos fibres were published in the 1960s and 1970s which prompted the later asbestos regulations and bans (Strohmeier et al., 2010). Even without deeper research into the fibres themselves, a common understanding was that the serpentine asbestos, chrysotile, was the least harmful while the amphibole asbestos minerals were the most harmful. This led to the complete banning of all amphibole asbestos minerals (Gaffney et al., 2017). Crocidolite and amosite were the most used amphiboles and, thus, also believed to be the most harmful. The lack of large-scale use of the other amphibole asbestos fibres prevented more detailed research into the health hazards caused by them (Gaffney et al., 2017).

At the end of the 1970s, the first asbestos bans were introduced in the Western world due to health concerns (Kameda et al., 2014). Asbestos was replaced by synthetic and ceramic fibres in most materials, the health impact of these newer fibers is unknown. As the use of asbestos was phased out, the cases of asbestosis and lung cancer started to decrease. Mesothelioma has a significantly longer onset of up to 40 years, so the peak of asbestos-induced cancers in Europe and North America should be seen in the 2010s (Tossavainen, 2004).

3.2 Types of diseases

There are three main diseases associated with the inhalation of asbestos fibres: asbestosis, mesothelioma and lung cancer (Gunter et al., 2007). There is also some evidence that ingestion of asbestos fibres is linked to other forms of cancer (Kjærheim et al., 2005; Van Kesteren et al., 2004).

3.2.1 Asbestosis

Asbestosis is the scaring and inflammation of the lung tissue that occurs when large amounts of asbestos fibres are inhaled. As the lungs cannot break down the fibres, they are eventually covered with scar tissue. Within the lungs, the asbestos fibres can split to several thinner fibres, exacerbating the problem. With continued exposure to asbestos, the gas-exchange ability is reduced and chest pain is common. Asbestosis can eventually lead to death. Asbestosis was known quite early in the 20th century, as many workers and their family members died of asbestosis and related pneumonia (Virta, 2002). This made it more difficult to find direct evidence that asbestos caused lung cancer and mesothelioma (Strohmeier et al., 2010). The onset of asbestosis is around 15 years, but depends on the amount of asbestos exposure (Prazakova et al., 2014).

3.2.2 Lung cancer

Lung cancer is a less common disease caused by asbestos exposure. The link between asbestos and lung cancer was much more difficult to detect in the 20th century as smoking was common and, thus, obscured asbestos-caused cancer. It is now known that smoking significantly increases the risk of lung cancer when also exposed to asbestos fibres (Gunter et al., 2007). The latency period for developing lung cancer from asbestos is around 15–20 years (Prazakova et al., 2014).

3.2.3 Mesothelioma

Mesothelioma is a cancer of the tissue that surrounds the lungs. Mesothelioma is extremely rare and is only found in those exposed to asbestos. It is thought that the amphibole asbestos minerals almost exclusively cause mesothelioma. It took until the 1970s to link mesothelioma with asbestos and a big reason for this was that mesothelioma has a latency period of up to 40 years. This means it was impossible to know all the health risks of asbestos when it was first used in industrial scale (Prazakova et al., 2014).

3.3 Mechanism for disease

The natural defences of the lungs expel much of the asbestos fibres, when inhaled. A small but significant amount travel deep into the lungs and are lodged there permanently. Due to the fibres being permanently stuck in the lungs, there is no known safe limit of asbestos exposure (Lippmann, 1990).

The harmful effect of asbestos is closely linked to the physical properties of the fibres. The long and thin fibres easily travel deep into the lungs and get trapped. The thinner the fibre the more strongly it is linked to disease (Wylie et al., 1993). Most dust that gets lodged in the lungs are removed by macrophages and the mucociliary escalator, but as the asbestos fibres are very long compared to most dust, they cannot be readily removed by the macrophages (Lippmann, 1990). As the macrophages try to "eat" the fibres, they are unable due to the length of the fibre. This causes the macrophage to eventually die and this leads to an inflammatory reaction. This chronic inflammation causes fibrosis of the lungs (Blake et al., 1998).

In the lungs there is a mucociliary escalator which transports away particles trapped in the upper lungs. This prevents dirt, dust and large asbestos fibres from entering the gas-exchange region of the lungs. Some short fibres are also trapped on the way to the lungs and are removed by the mucociliary escalator (Lippmann, 1990). The function of this escalator is greatly diminished by smoking and thus smokers are much more susceptible to asbestos-related diseases such as asbestosis (Gunter et al., 2007; Prazakova et al., 2014). As the asbestiform fibres can easily be separated into thinner fibres, they can be reduced to such a small size that they can enter the cells surrounding the lungs. As these small fibres enter the cells surrounding the lungs, they stay there and are thought to eventually lead to mesothelioma. Small asbestos fibres can also be detected in a person's urine if the person has recently been exposed to asbestos (Gunter et al., 2007).

Chrysotile is thought to be partly soluble in the lung fluids and, thus, is more seldom seen in the lungs of asbestosis patients (Gunter et al., 2007). It is thought that the brucite layer is dissolved, causing the hollow silica structure collapses to smaller pieces and these fragments can then be transported out of the lungs naturally. Chrysotile is also linked to the growth of large hydroxyapatite (bone) crystals that are too large to have entered the lungs as dust particles (Gunter et al., 2007). The amphibole fibres are resistant to the conditions in the lung and once inhaled, the fibres are almost impossible to remove. Fibrosis eventually takes over the lung and the risk of lung cancer and mesothelioma are considerably increased. Other minerals such as quartz are also persistent in the lungs and are known to cause lung diseases (Gunter et al., 2007).

The danger of asbestos comes mainly from the respiration of the fibres, although ingestion of the fibres have shown to increase the risk of cancer (Kjærheim et al., 2005; Van Kesteren et al., 2004).

3.4 Environmental exposure to asbestos fibres

Environmental exposure to asbestos happens when humans or animals are exposed to significant quantities of airborne asbestos fibres. This can be due to the local geology or asbestos-containing products. As an example, when old buildings are demolished, the removal of asbestos-containing materials can release fibres in the air. Another known problem is when asbestos occurs in the bedrock. This can cause the air and soil in the area to be contaminated with asbestos fibres. Sometimes human activities such as quarrying or other utilization of local rock, can significantly and unintentionally increase the amount of asbestos fibres in the air.

Large deposits of asbestos are rare, and have often been exploited commercially, but asbestos occurs in low amounts everywhere where the geologic conditions are right. This means people can be exposed to asbestos without being close to a mine or working with asbestos-containing products. Especially the amphibole varieties of asbestos are common throughout the world. The environmental exposure to asbestos fibres is an increasing concern worldwide (Ross and Nolan, 2003).

In the asbestos mining areas and other areas where larger concentrations of fibres are found naturally, there is a statistical increase in the asbestos related diseases (Dogan, 2003; Gianfagna, Andreozzi, et al., 2007).

It is not only the six asbestos minerals that warrant concern as there are other naturally forming mineral fibres that have shown to have asbestos-like impact on health. One such is erionite in central Turkey. The occurrence of erionite has been linked to a significant amount of mesothelioma cases in nearby villages (Dogan, 2003). There has not been any mining or production of asbestos nearby, all exposure and subsequent cancer is from environmentally occurring erionite.

Another non-asbestos fibrous mineral is fluoro-edenite that is a fluoride containing amphibole. It has been linked to asbestos diseases in Sicily (Gianfagna and Oberti, 2001; Gianfagna, Andreozzi, et al., 2007).

Southwestern USA has a relatively large amount of chrysotile fibres occurring in the environment, for example, in the El Dorado Hills in California (Ross and Nolan, 2003; Gunter et al., 2007). In California, this has raised some concern as urban development increasingly occur on rock and soil which has a naturally high amount of asbestos fibres. There has been no significant increases in asbestos-related diseases in the areas affected and much of the concern is unfounded (Gunter et al., 2007).

In Finland, the areas around the old asbestos mines at Paakkila and Maljasalmi show a significant amount of asbestos fibres in the soil. This is due to the recent ice age, as it grated the local bedrock causing the fibres to be spread in the environment. It is estimated that the soil around the old mines can have up to 120 000 fibres per gram of soil. The baseline amount of expected asbestos fibres in soil is less than 3 000. Outside the mine itself there has been found outcrops and glacial erratic boulders that contain anthophyllite. Recent sediments analysed from ponds near the Paakkila mine show that asbestos fibres are still today spread throughout the area, while the mine and processing plants closed in 1975. Finnish anthophyllite is not linked to mesothelioma and this explains the lack of widespread mesothelioma cases in the region (Nikkarinen et al., 2001).

Environmental exposure to unhealthy levels of asbestos fibres is rare. Up to the 1970s, asbestos was commonly used in a wide variety of applications and this sometimes significantly increased the amount of asbestos fibres in the local environment (Gunter et al., 2007). As an example, the brake-pads of large vehicles were made of chrysotile and this released fibres into the air in normal use of the vehicle. Some professions that heavily used asbestos in the workplace, e.g. spraying of fire insulation and installing brake-pads, as well as those that worked directly in the asbestos industry, were by far the ones most significantly impacted by asbestos (Virta, 2002).

3.5 Occupational exposure and asbestos abatement

Throughout the 19th and 20th century, asbestos diseases mostly affected those in the asbestos industry. As many Western countries banned the use of asbestos starting in the 1970s, the principle exposure has been asbestos abatement work (Strohmeier et al., 2010). This abatement work consists of the removal of asbestos containing products or sealing them to prevent dust from being released. The most common asbestos containing materials are in buildings, vehicles and industrial settings. As these old materials are removed, often by sawing, grinding or tearing, the fibres are potentially released into the air. The dust created by this is the most common way to be exposed to asbestos today (Strohmeier et al., 2010). This has caused a requirement for special measures and a licence is required for the removal of asbestos containing products (FINLEX, 2015a) As asbestos was used in a wide variety of products, it is often required to do an asbestos mapping before the commencement of any demolition. The materials from this mapping are analysed by laboratories to find any potential asbestos in the old construction materials.

In Finland, the law has required a mandatory asbestos mapping of buildings since 2016 (FINLEX, 2015b). As all use of asbestos was completely banned in Finland in 1994 (FINLEX, 1992) the new law requires all buildings older than 1994 to be mapped before renovation or demolishing (FINLEX, 2015b). The law does not specify any methods in analysing asbestos, only that they need to reliably find and determine asbestos content. A requirement in Finland is also to take an air sample after asbestos abatement to ensure that asbestos containing dust is not allowed to spread outside the work area (FINLEX, 2015b). In Finland, two of the most common methods for asbestos identification are SEM-EDS and PLM based (SAP, 2018).

4 History of asbestos

4.1 Asbestos use and production

The first known small-scale asbestos mining and use is dated to 3000–2500 BC in Cyprus and Finland (Ross and Nolan, 2003; Virta, 2002). It was mostly used for reinforcement of pottery, fireproof textiles and clothing. The word asbestos comes from Greek and means 'unquenchable' or 'indestructible' (Ross and Nolan, 2003).

The large-scale use of asbestos started in the 1800s, as the need for heat and fire-resistant materials grew with the rise of the industrialization (Ross and Nolan, 2003). The industrial production of asbestos started in Italy but by the beginning of the 20th century, Canada and Russia had taken over as the world producers of asbestos fibres. The most commonly mined asbestos mineral was chrysotile with large mines in Canada and Russia. Other commercially significant asbestos minerals were crocidolite and amosite, mostly mined in South Africa and Australia (Virta, 2002). These minerals account for over 95% of the asbestos used in the world.

4.2 EU asbestos ban

In the EU, the definition of asbestos is according to the Chemical Abstract Service that has mineralogical and morphological definitions of asbestos (EUR-Lex, 2009). The definition includes all six known asbestos minerals. The first major ban of asbestos use in the EU was in 1976 with the 76/769/EEC Directive (EUR-Lex, 1976). This started the banning of various asbestos products and completely banned the amphibole asbestos minerals. With various amendments to the law, the bans became stricter until 1997 when a new amendment banned all new applications for asbestos from 1. January 2005. In 2009, a new directive for the exposure to asbestos was introduced and it banned all use, extraction and manufacture of asbestos and asbestos containing products in the EU (EUR-Lex, 2009). It also mandated the replacement of all products that currently use asbestos for non-asbestos containing products. Only a few exceptions were made, and the exceptions were temporary (REACH, 2006).

The use of asbestos sharply declined after the first bans of asbestos and became almost non-existent by 1990s (Virta, 2006). Today, asbestos remains mostly in old construction materials and old spare parts for the automotive industry. In the EU, most applications that used asbestos can be used until the end of their service life or until suitable substitutes are found (REACH, 2006). The largest usage of asbestos after the ban has been in specific industrial applications, the largest of which is diaphragms for the production of chlorine by electrolysis. As of 2006, no suitable alternative to chrysotile had been found and its usage will continue until a replacement is found (REACH, 2006). The REACH regulation specifies exceptions in the asbestos ban, but many of these exemptions have already expired. Examples of items containing asbestos that are allowed to be used if there is nothing to substitute them are: spare part, vehicle parts (often brakes or gaskets), diaphragms of chrysotile for electrolysis plants and acetylene cylinders containing chrysotile (REACH, 2006).

4.3 US and Canadian asbestos bans

In the United States, the definition of asbestos varies depending on whether it is used in a legal or mineralogical setting (Virta, 2002; Gunter et al., 2007). The United States Geological Survey issues the official definition of asbestos. In the US, asbestos containing dust has been regulated since the 1920s. The regulations concerning asbestos became more stringent in the latter parts of the 20th century. In the 1970s, the first bans of asbestos use were introduced but they applied only to specific applications, such as asbestos pipes and spray-applied surfacing. After the 1970s, only some application-specific bans of asbestos have been issued. In 1989, a law banned all new uses of asbestos in products but many legacy products remain legal to use (Lemen and Landrigan, 2017). Today, asbestos is restricted to niche applications in the US due to the risk of health-related lawsuits. There is no official ban on the usage of asbestos. The most common use of asbestos in the US today is in the electrolysis industry.

In Canada, there has been mining and processing of asbestos for over 130 years. Canada has long been one of the largest producers of asbestos and promoted worldwide the use of asbestos with the help of the Chrysotile Institute. The collapse of the Canadian asbestos industry started in 2011 with the closure of the last asbestos mines in Quebec (CBC, 2011). In 2012, Canada stopped supporting the industry completely and cut funding for the Chrysotile Institute (Tim

Povtak, 2012). The processing and use of asbestos is still allowed in Canada, but a complete ban is expected in the future (Government of Canada, 2018).

5 Materials and methods

5.1 Methods for asbestos analysis

The most common methods in use for commercial asbestos analysis are scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS), transmission electron microscopy (TEM-EDS), X-ray diffraction (XRD) and polarized light microscopy (PLM) (Strohmeier et al., 2010). Other methods such as Raman and micro-Raman spectroscopy are used for asbestos analysis in research (Rinaudo et al., 2004).

The oldest method for identifying asbestos minerals is the PLM method. It has been in use since 1920s and is based on identifying the fibres with optical characteristics (OSHA, 1995). The SEM-EDS and TEM-EDS methods became more common in the latter half of the 20th century. Both SEM-EDS and TEM-EDS use the much greater magnification allowed by the electron beam, together with elemental analysis to identify asbestos fibres. XRD and Raman are based on identifying the molecular bonds of the mineral and therefore it is possible to differentiate between asbestos and non-asbestos forms of the same mineral. All methods have their limitation and most commonly it is the small size of the fibres that make it hard to do a proper identification. The TEM has the largest magnification and has the possibility to identify the smallest of asbestos fibres. In France, dust samples must be analysed with ATEM (analytical transmission electron microscopy) (ANSES, 2016). Most countries in Europe do not have a regulatory requirement for a specific type of analysis method.

In Finland, the most common methods for asbestos analysis are SEM-EDS, TEM-EDS and PLM (SAP, 2018).

5.2 Background

In this thesis, two methods were used to identify asbestos from construction materials. These methods were SEM-EDS and PLM. Both the SEM-EDS and PLM analyses were conducted at Top Analytica in Turku. The SEM-EDS method is based on the international standard for identifying asbestos in bulk materials: ISO 22262-1:2012 (International Organization for Standardization, 2012). The PLM method is based on guides for asbestos analysis released by the health authorities in the US and the UK (EPA, 1993; NIOSH, 1994; OSHA, 1995; HSE, 2006) and the same ISO standard as the SEM-EDS method. The PLM method was developed and adapted to suit the equipment and methods already in use at Top Analytica. A reliable SEM-EDS method has been in use at Top Analytica since 2016. Raman-spectrometry was also used on reference samples and they were successfully identified. This thesis focuses mainly on the strengths and weaknesses of the PLM and SEM-EDS methods.

5.3 Preparation of samples and sample types

For this thesis, 192 samples were analysed by both PLM and SEM (Appendix A). The samples were submitted to Top Analytica as construction materials to be analysed for asbestos content. The samples were a wide range of different types from plastic carpets to bitumen to concrete. The sample preparation technique evolved significantly during the work on this thesis. In Figure 6 is given a flowchart of the general process of sample preparation for the PLM method. In the PLM method, liquids with different refractive indexes (R.I) are needed. the liquids allow the different asbestos minerals to be identified. The SEM-EDS method requires much less preparation as the sample only needs to be broken to smaller pieces and added to a copper tape.

The AIMS samples are control samples issued by the Health and Safety Executive in the UK. The samples are construction materials similar to the regularly submitted samples. It is part of a voluntary accreditation program that asbestos laboratories can participate in. The AIMS samples come in a batch of four samples which are analysed for asbestos content. If asbestos is found the asbestos type or types are identified. AIMS samples can contain any of the six asbestos minerals and often there is more than one asbestos type in the sample. Samples without asbestos are also possible. A table of the AIMS samples is found in Appendix B.

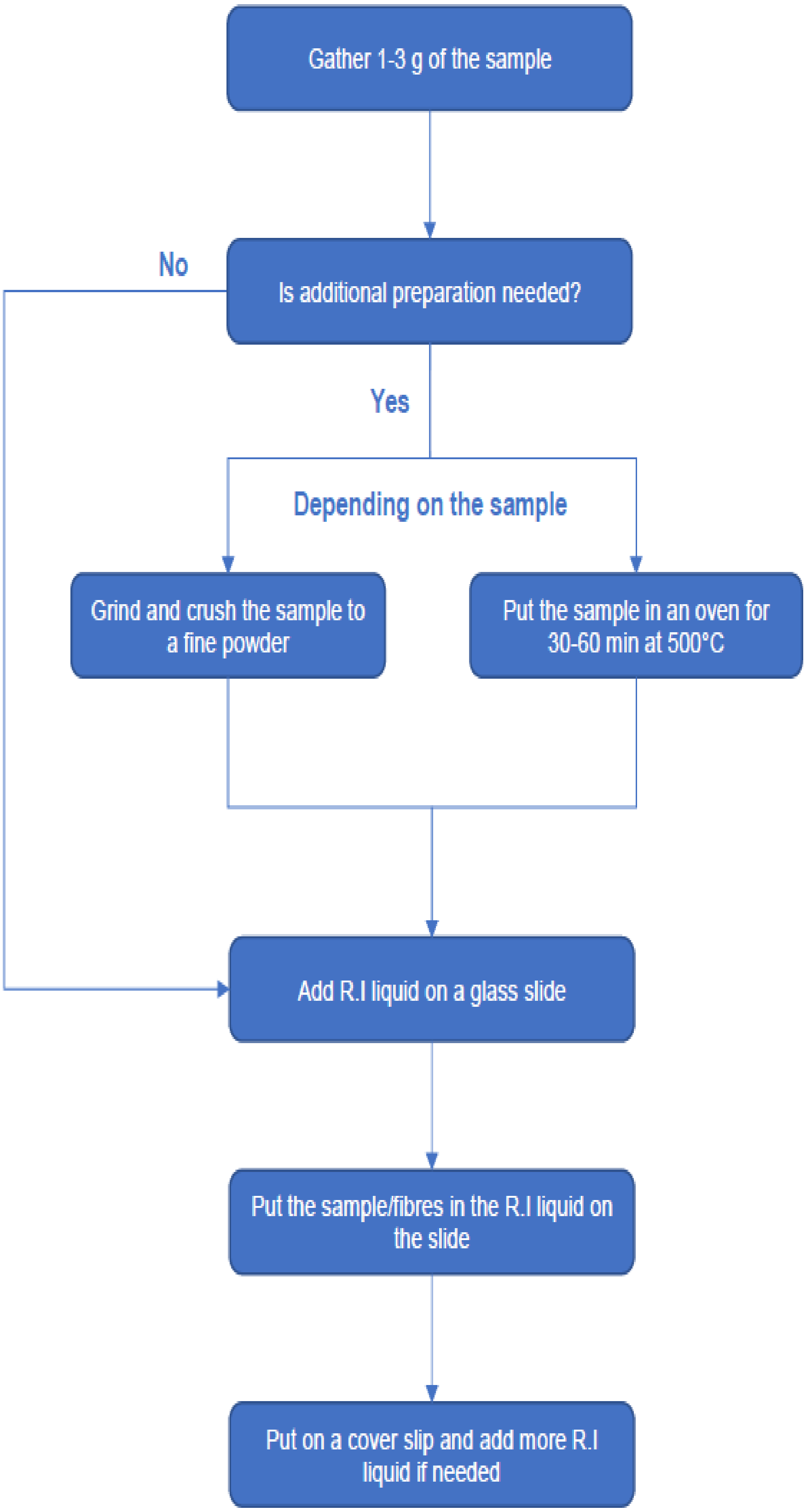


Figure 6: A simple overview of the steps required for sample preparation for the PLM method.

The first step in the preparation of a sample is a visual inspection for fibres. As the fibre content in the materials range from zero to over 30%, the fibres can sometimes be seen by eye. An important part of the visual inspection is to identify all the different parts of the sample, for example a tile might include the tile itself together with plaster and filler. Once all the different materials and layers are identified, it is important to get a representative sample of each material. The asbestos fibres might be heterogeneously distributed in the sample, so it is important to reduce the particle size and free any fibres that might be trapped in the matrix of the material. The prepared samples are then inspected by stereo microscope for any possible fibres. If fibres can be seen with the naked eye, no further preparation is necessary.

For the PLM method, the samples are prepared by crushing, grinding and tearing the material to as small pieces as possible. Samples with an organic matrix like rubber or bitumen are heated to 500°C before they can be properly prepared. Ashing also removes any possible organic coating on the asbestos fibres and other non-asbestos fibres that can interfere with identification. Inspection of the sample with a stereo microscope before and after ashing is important to ensure that any fibres that are in the sample are found. In the PLM method, it is also crucial to get the prepared sample as small and even-grained as possible. This ensures the best possible analysis. The prepared samples are then put on a glass slide in a R.I liquid (Figure 7). To prevent contamination of the microscope, the sample is covered with a cover slip. If the prepared sample is of varying particle sizes, it causes the cover slip to be uneven and this makes analysing the sample much harder.

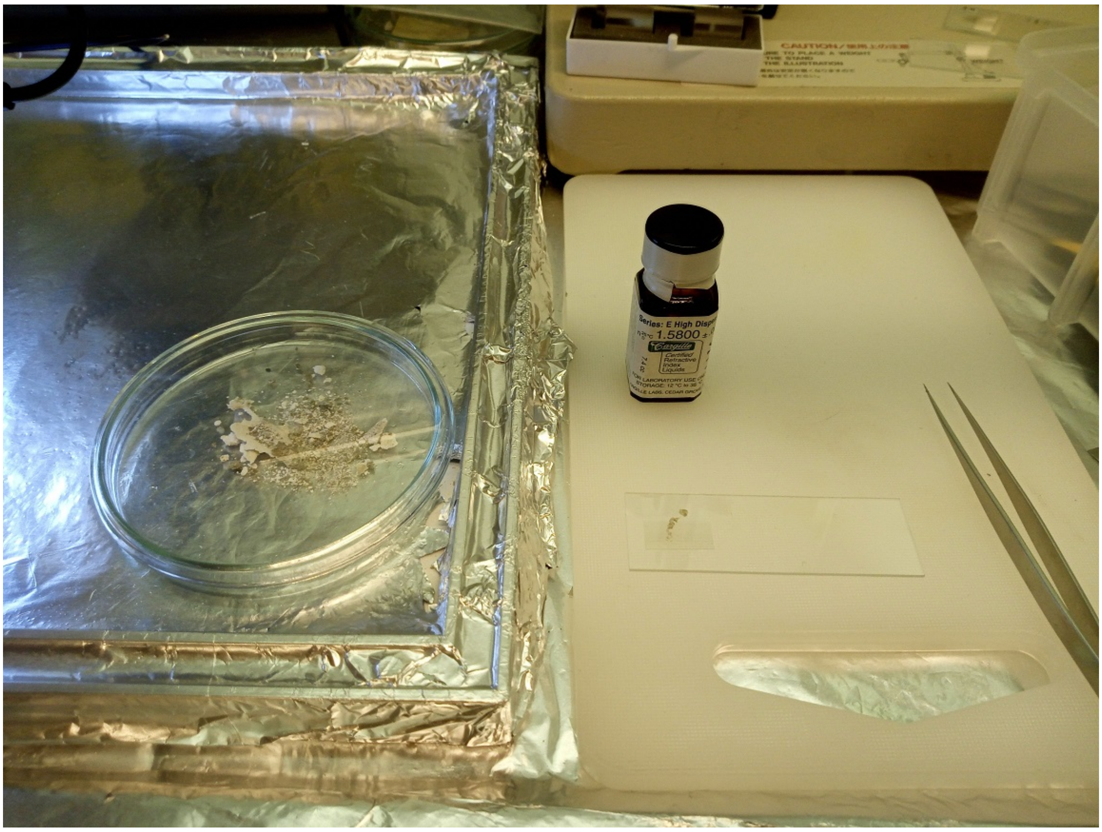


Figure 7: The prepared samples are added to a glass slide that has a drop of R.I liquid. The R.I liquid is chosen based on suspected asbestos type or if none are suspected the 1.5800 liquid is used.

For the SEM-EDS method the samples also require some preparation. First all materials and layers in the sample need to be identified and prepared. The sample is then crushed, ground and teared in the same way as in the PLM method. The samples do not need to be reduced to as small a size due to the different operating principle of SEM. Ashing is also often not needed, it is only done in cases where the fibres are thought to be completely trapped inside the matrix. After the samples have been prepared, they are added to a double-sided copper tape. The SEM-EDS method allows much larger and uneven-sized particles to be analysed. The preparation of samples is still important for the SEM-EDS, as the fibres might be heterogeneously spread in the sample. Both a visual and stereo microscope inspection is done before adding the prepared sample to the copper tape (Figure 8).

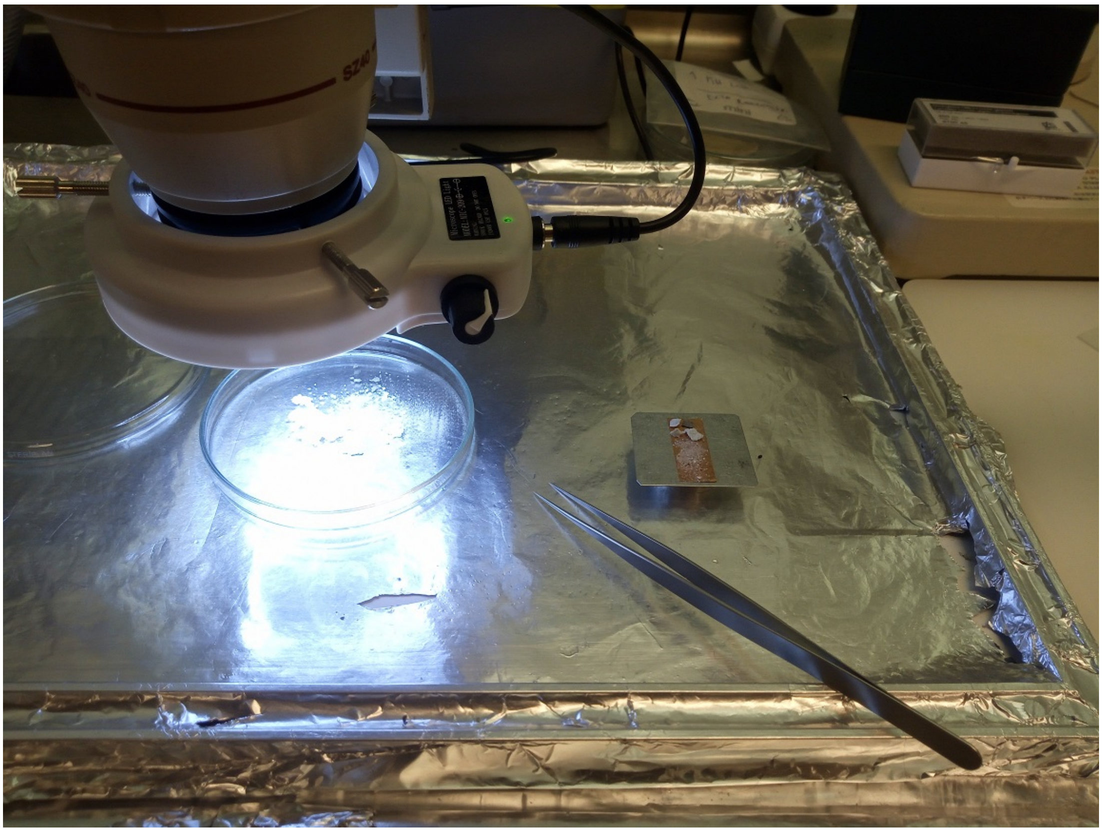


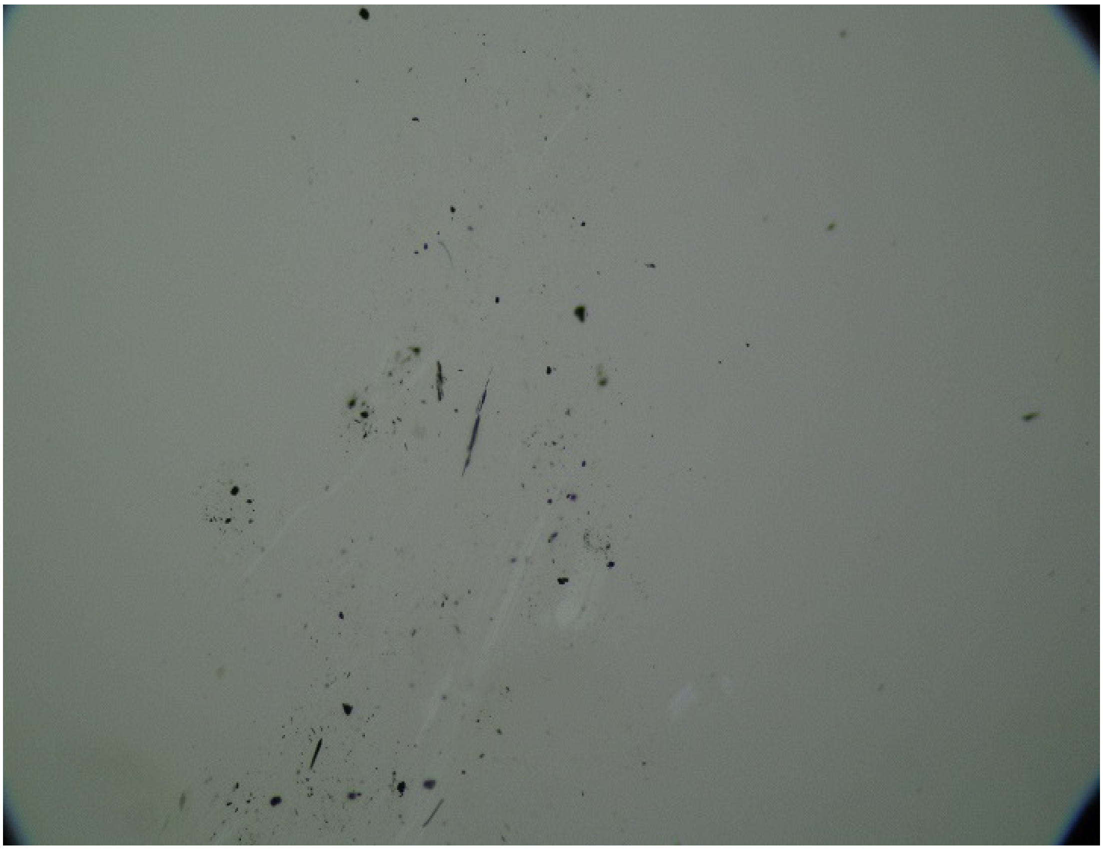
Figure 8: For the SEM-EDS method the prepared samples are added to a copper tape.

5.4 Polarized light microscopy (PLM)

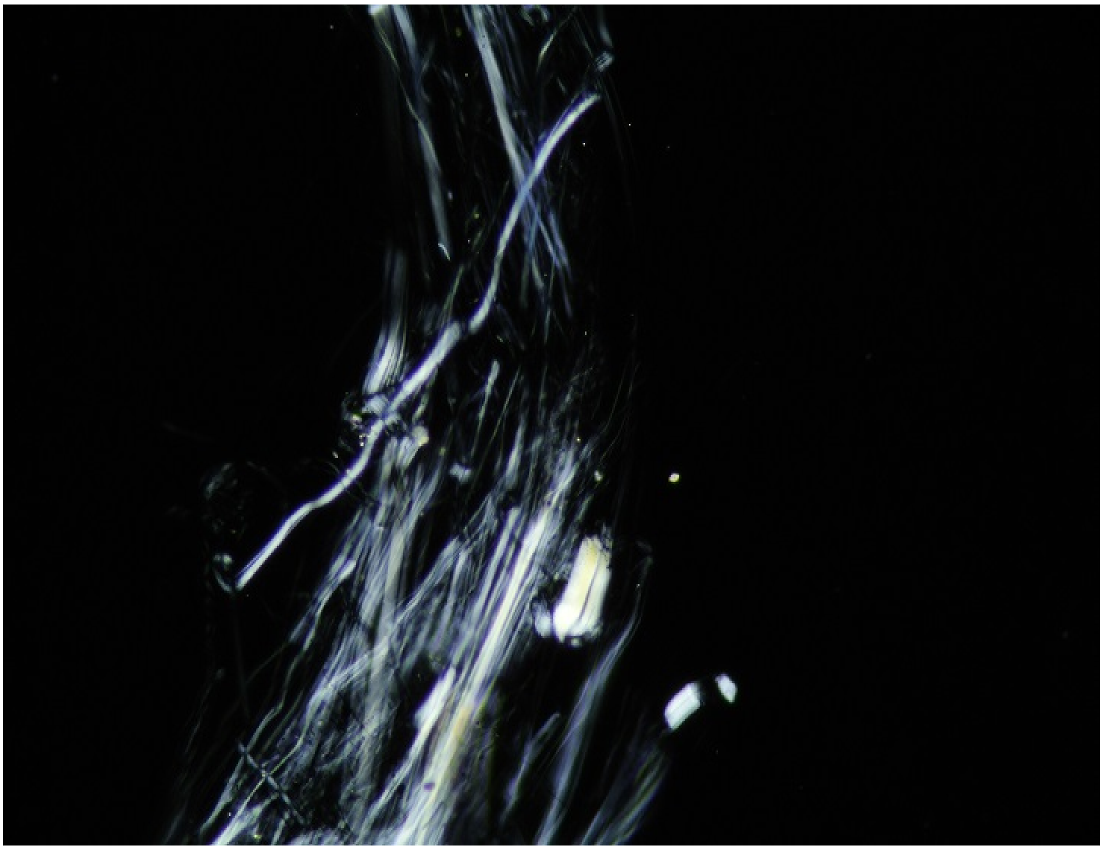
Most of the PLM analyses were carried out on a Leica 12 POL microscope with 40x, 100x, 600x magnification. The microscope is an ordinary petrographic microscope with no special modifications for asbestos identification. Towards the end of the thesis the analyses were done on a Leica DM 2700 P with 125x and 500x magnification. The DM 2700 P is equipped with dispersion staining capabilities for asbestos identification.

In a polarizing light microscope, the light shines from below, through the sample and up to the ocular. The light is modified in different ways to achieve desired optical characteristics in the sample. The first modification happens above the light in the polarizer. This makes the light plain-polarized. Next comes an condenser to focus the light more intensely on the sample. The sample needs to be such that light can pass through it, most minerals pass light through when thin enough. After the sample the light goes to an objective and this determines the magnification. Above the objective there is another polarizer perpendicular to the polarizer below. It is referred to as the analyser. If both polarizers are in the lights path, the light is cross-polarized. If the analyser is not inserted the light is plane-polarized. Lastly there is a gypsum plate to shift the light 550 nm. Both polarizers need to be in use for the gypsum plate to have an visual effect.

The identification of asbestos fibres is based on several different optical characteristics. The most important are morphology, anisotropic crystal structure, comparison of refractive index (R.I) in plane-polarized light, pleochroism and dispersion staining colour. For asbestos the morphology needs to be long, thin, almost hairlike with a length-to-width ratio of over 20:1. The fibres also must be longer than 5 μm and thinner than 3 μm. The anisotropic crystal structure can be seen in cross-polarized light as the fibres show an even colour throughout the fibre. This even diffracting of light is only seen in minerals, this helps in differentiating between asbestos and non-asbestos fibres. Pleochroism can be seen in two of the asbestos minerals, actinolite and crocidolite, as green-grey and blue-grey respectively. This changing of colour is seen in plane-polarized light when the sample is rotated. If the mounting liquid and fibre have the same R.I, no clear fibre edges can be seen in plane-polarized light (Figure 9a). The same fibres are still clearly seen in cross-polarized light (Figure 9b). These characteristics work as a crude way to identify asbestos, dispersion staining is required to get a reliable final analysis. A flowchart of the identification process for the PLM method is found in Figure 10.



(a) Chrysotile in plane-polarized light



(b) The same chrysotile fibres in cross-polarized light

Figure 9: In (a) the chrysotile fibres are barely seen as they are in a liquid with the same R.I. (b) is the same fibres in cross-polarized light. The fibres show a even colouring in cross-polarized light that is only seen in mineral fibres. Taken with 125x magnification.

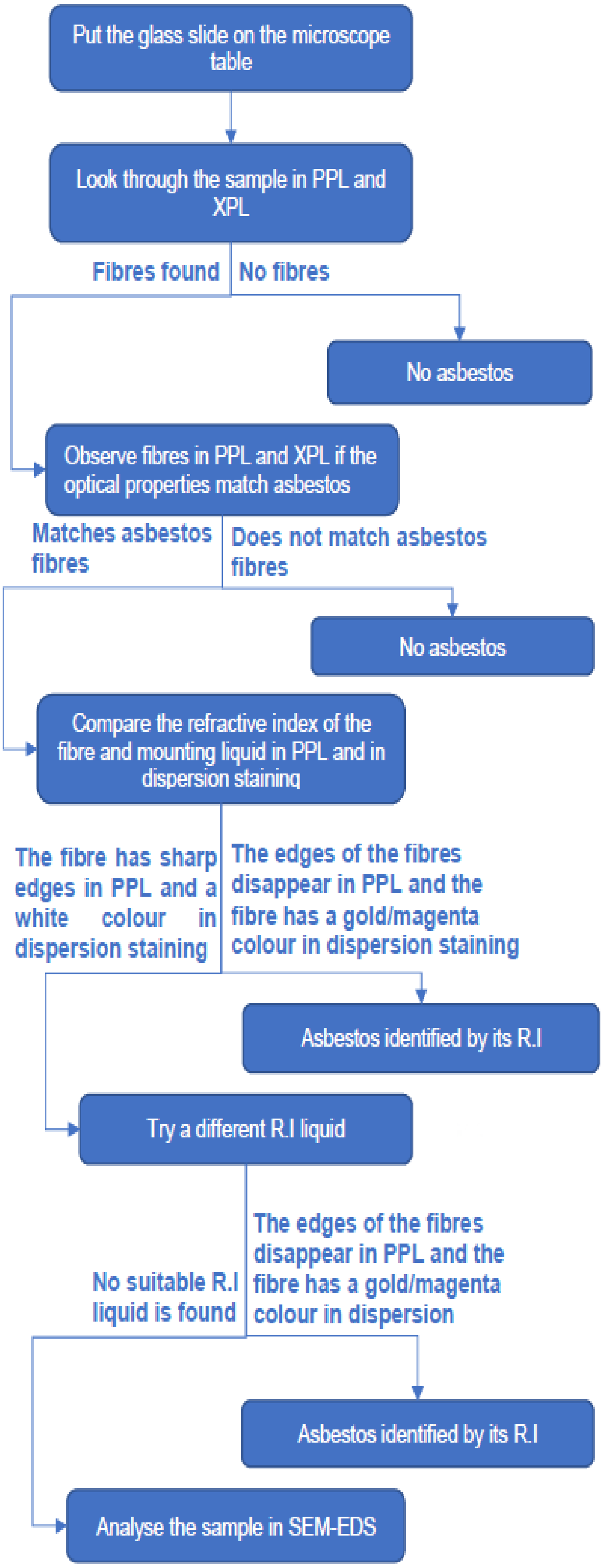
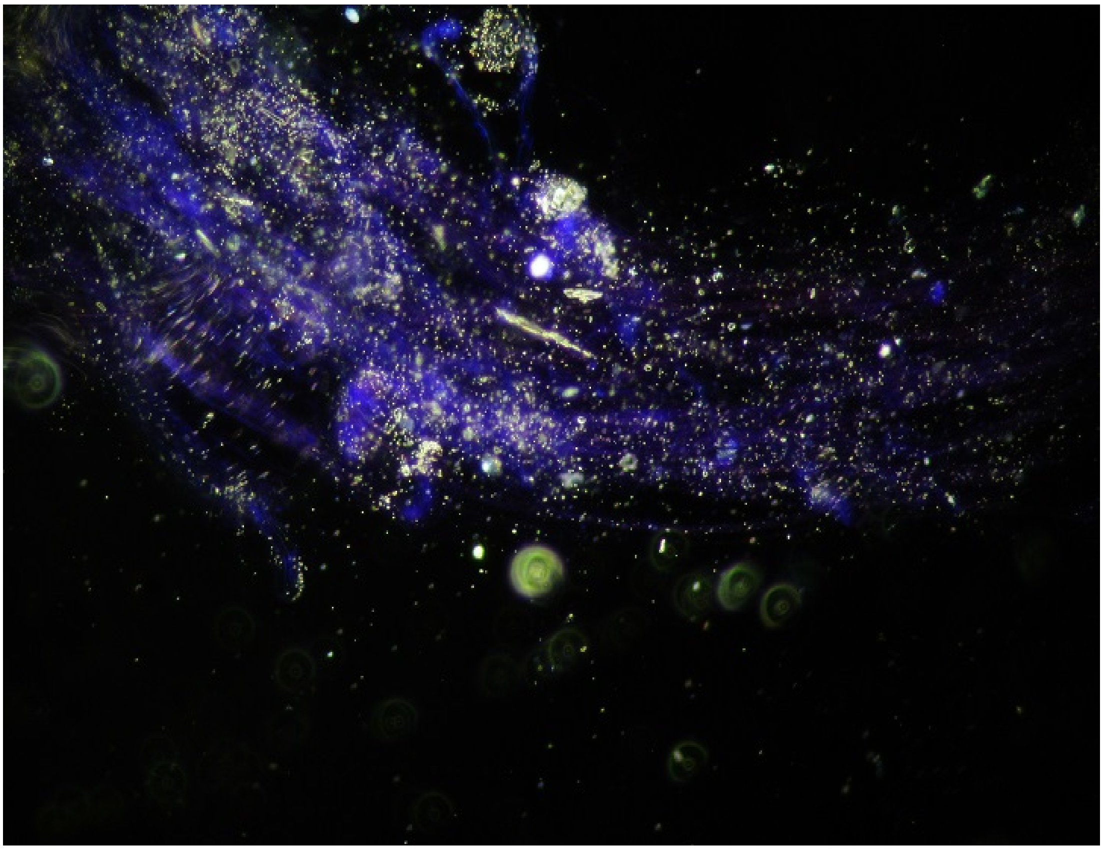


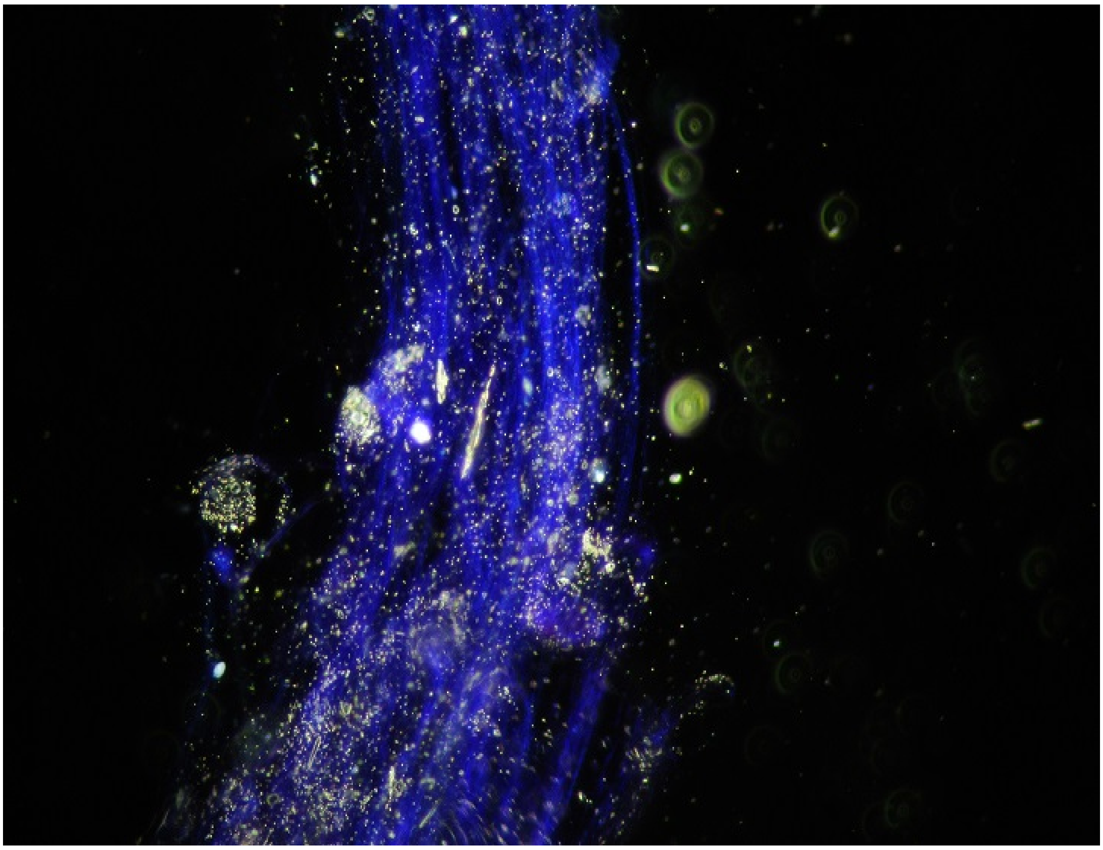
Figure 10: A flowchart of the steps done to identify asbestos with the PLM method.

For proper and accurate identification of asbestos, the microscope needs to be equipped with a darkfield condenser or a dispersion staining objective. The darkfield condenser works with any objective and its function is to block direct light from reaching the objective. Only dispersed light reaches the objective. The dispersion staining objective works in a similar way by having a central stop within the objective to block direct light. With a dispersion staining objective the dispersion staining colouring is seen when a the condensers iris is closed. The dispersion staining objective works with the darkfield condenser but if the iris is also closed, no light reaches the ocular.

As the asbestos minerals have different refractive indexes, the dispersion staining effect can be used to identify individual asbestos minerals. When the liquid and the fibre have the same refractive index, the light is dispersed to the visible spectrum and is seen as a colour. If the refractive indexes differ, the fibres appear white. Both the darkfield condenser and the dispersion staining objective can be used to view the dispersion staining colours. As an example, the chrysotile fibres show a magenta-purple colour in the correct R.I liquid (Figure 11) while the amphibole asbestos fibres show a golden-blue colour. The appendices Appendix C and Appendix D show the relevant optical characteristics for the identification of asbestos minerals with the PLM method.



(a) Chrysotile has a magenta colour in dispersion staining



(b) When the fibres are rotated they show a bluer colouring

Figure 11: Chrysotile in the correct R.I liquid shows a magenta-blue colouring. If the R.I liquid is not correct the fibres appear white. Taken with 125x magnification.

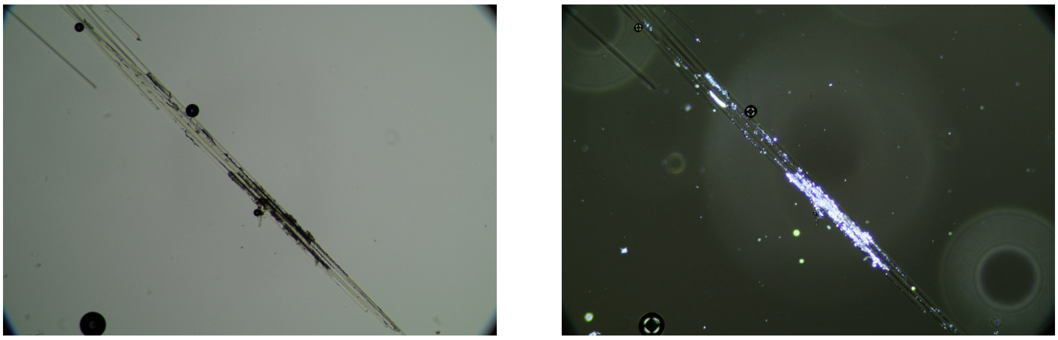
For most of the work in this thesis, a microscope equipped with dispersion staining was not available and this reduced the accuracy of the analyses. Especially when a material containing several asbestos minerals was analysed.

The R.I liquids used were the Cargille HD 6 from Agar Scientific. The series includes six liquids: 1.5500, 1.5800, 1.6050, 1.6400, 1.6700 and 1.7000. All these liquids correspond to a specific asbestos mineral except for 1.5800 and 1.6050. The 1.6050 is the refractive index of both anthophyllite and tremolite and 1.5800 is a middle ground between chrysotile and the amphibole asbestos minerals. 1.5800 is a good starting point for determining unknown fibres, as it allows all fibres to be seen in plane-polarized light. Due to tremolite and anthophyllite having the same R.I they cannot be distinguished by dispersion staining. Table 1 shows the R.I values of all asbestos minerals.

Table 1: Table of R.I values for the different asbestos minerals

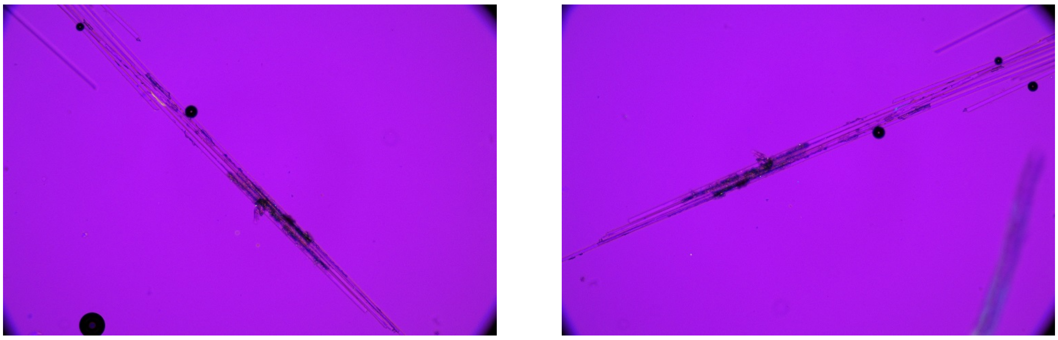
Chrysotile	Amosite	Anthophyllite	Tremolite	Actinolite	Crocidolite
1.5500	1.6700	1.6050	1.6050	1.6400	1.7000

In the PLM method there is a risk of misidentifying non-asbestos fibres as asbestos. The most common asbestos-like fibres are glass fibres, elongated wollastonite particles and organic fibres such as paper or polyethylene fibres. Glass fibres are easy to distinguish from minerals, as they are invisible in cross-polarized light due to not being anisotropic (Figure 11). Wollastonite particles have an asbestos-like morphology and are somewhat transparent. As they are mineral particles they show anisotropy in cross-polarized light (Figure 12). Polyethylene and paper fibres are quite similar to asbestos but differ most prominently in their uneven interference colours. They also show a variable R.I value in the fibre (Figure 13 and Figure 14). All these potential sources of error can be significantly reduced by heating the sample to 500 °C.



(a) Glass fibres in plane-polarized light

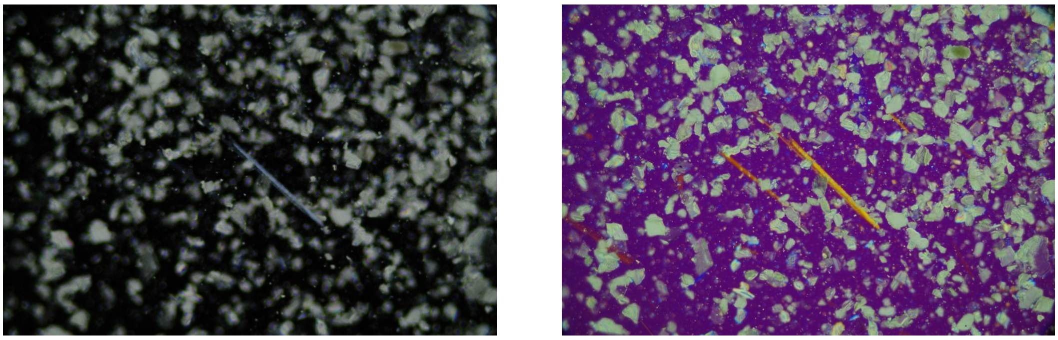
(b) Glass fibres in cross-polarized light



(c) Glass fibres in cross-polarized light with the gypsum plate inserted

(d) Rotating does not produce colours

Figure 11: In (a) the glass fibres look somewhat like asbestos fibres in plane-polarized light but become black in cross-polarized light (b). In (c) and (d) the glass fibres show no interference colours with the gypsum plate inserted. Taken with 125x magnification.



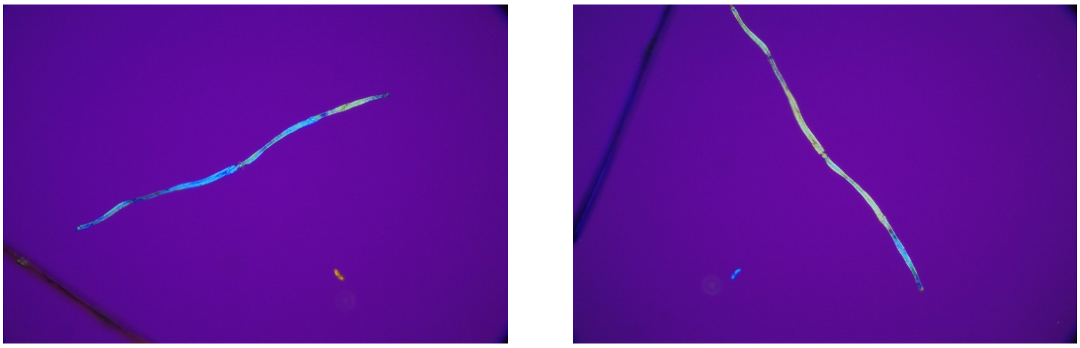
(a) Wollastonite in cross-polarized light

(b) Wollastonite in cross-polarized light with gypsum plate inserted

Figure 12: Wollastonite can easily be mistaken for asbestos fibres due to morphology. The refractive index of wollastonite is close to the amphibole asbestos fibres. Closer examination shows that wollastonite particles are mineral fragments rather than fibres. Taken with 125x magnification.

(a) Paper fibre in plane-polarized light

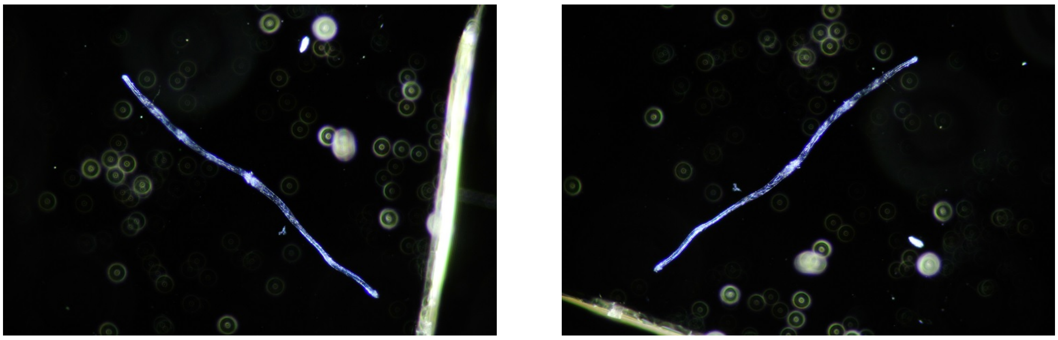
(b) Paper fibre in cross-polarized light.



(c) Paper fibre with the gypsum plate inserted

(d) The fibre shows a uneven sign of elongation

Figure 13: Paper fibres are a common contaminant in construction materials. They are usually coloured and show an uneven anisotropy. They have many similarities to asbestos fibres. Compared to asbestos, the fibres look more ragged and show uneven colours in cross-polarized light. Taken with 125x magnification.



(a) Paper fibre viewed in dispersion staining

(b) Paper fibre rotated

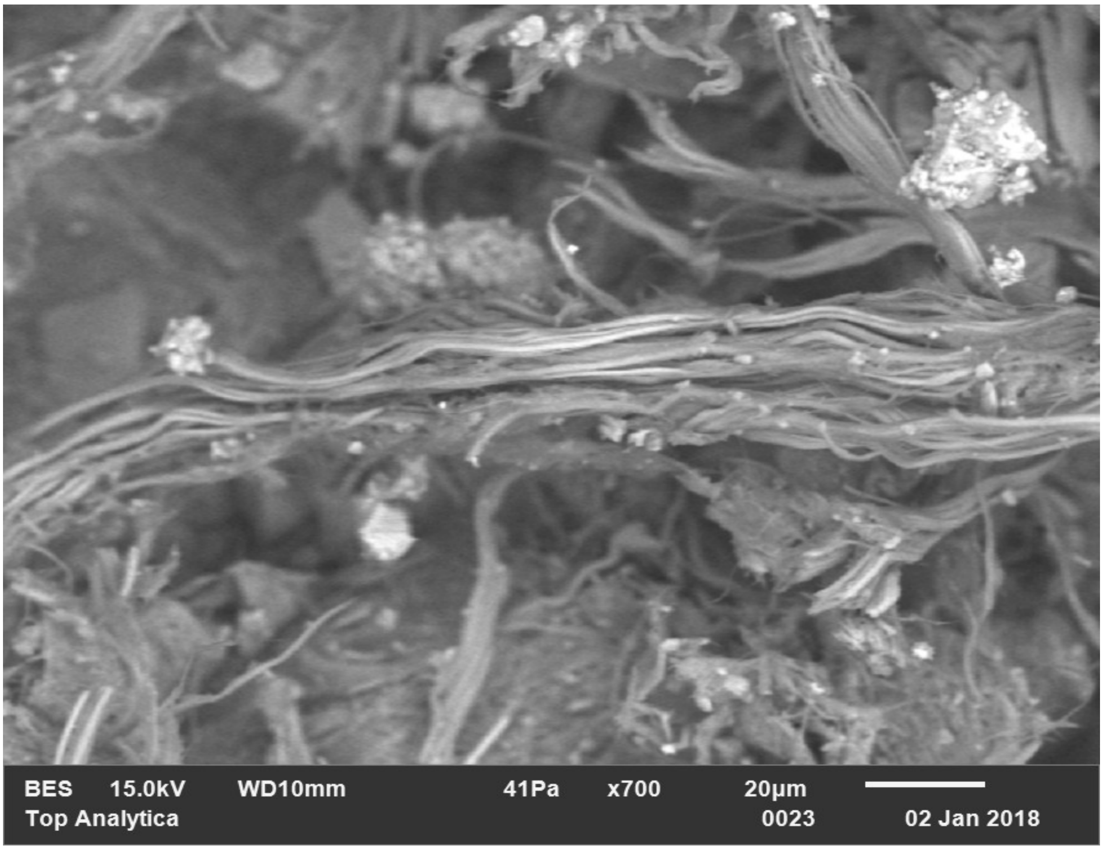
Figure 14: Paper fibres show a white or uneven colouring when viewed in dispersion staining. Rotating the fibre does not change its colour. Taken with 125x magnification.

5.5 Scanning Electron Microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS)

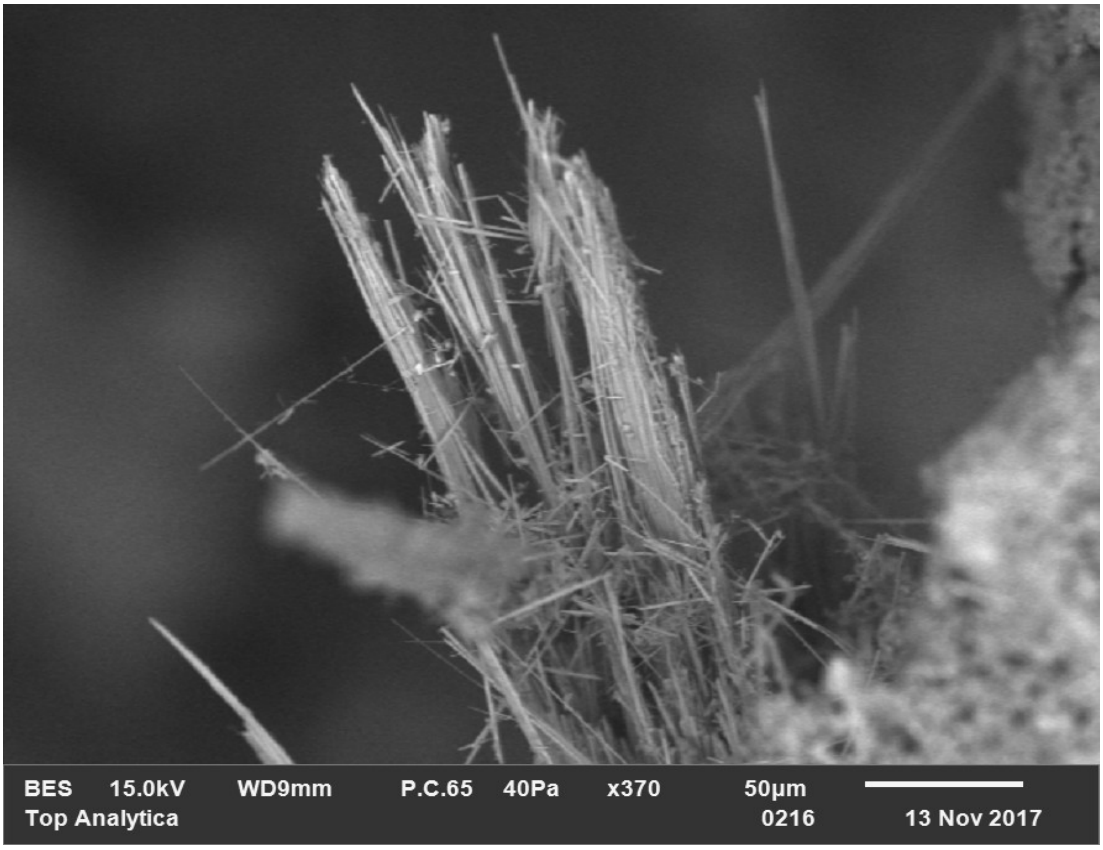
The SEM used in the SEM-EDS method was a JEOL IT-100 equipped with an EDS detector. The EDS detector used was a JEOL DPP5. The SEM was used in low-vacuum mode because it offered a significant speedup in analysis time. This sacrificed some imaging and analysis resolution. The low-vacuum mode has the advantage of not having to coat the sample with a conductive material. In low-vacuum mode, only backscatter image mode is available and that was used for imaging. The EDS detector allowed determination of the composition of the fibres. This made it possible to differentiate between the different asbestos minerals and other types of fibres. With an EDS it is possible to reliably identify all the different asbestos minerals.

The SEM-EDS method is based on bombarding the sample with electrons in a vacuum atmosphere. A detector is configured to receive electrons emitted by the sample as external electrons bombard the sample. As the detector receives electrons, it produces a signal and this signal is converted to black-and-white images seen in the SEM. In backscatter imagining the contrast in the image is due to atomic mass, where heavier elements are seen in a lighter colour and lighter elements in a darker colour. The use of electrons in imaging produces images of greater magnification than would be possible with light-based microscopes. This is a great advantage in asbestos identification, as asbestos fibres are often microscopic to sub-microscopic in size. SEM instruments can come with a variety of different detectors for the analysis of elemental composition. In this thesis an EDS was used. This detector measures the energy produced by X-rays in the material as it is bombarded with electrons. This allows for qualitative and quantitative analyses of the elemental composition in the fibres.

Asbestos fibres are mostly identified by morphology and composition in SEM-EDS. The fibres are either curvy, almost textile-like looking or sharp needles depending on the mineral. Chrysotile has a wavy and bendy look while the amphiboles look like needles (Figure 15). Morphology alone is not enough to reliably identify asbestos, as there exists similar looking fibres. The spectra given by the fibre also needs to be examined.



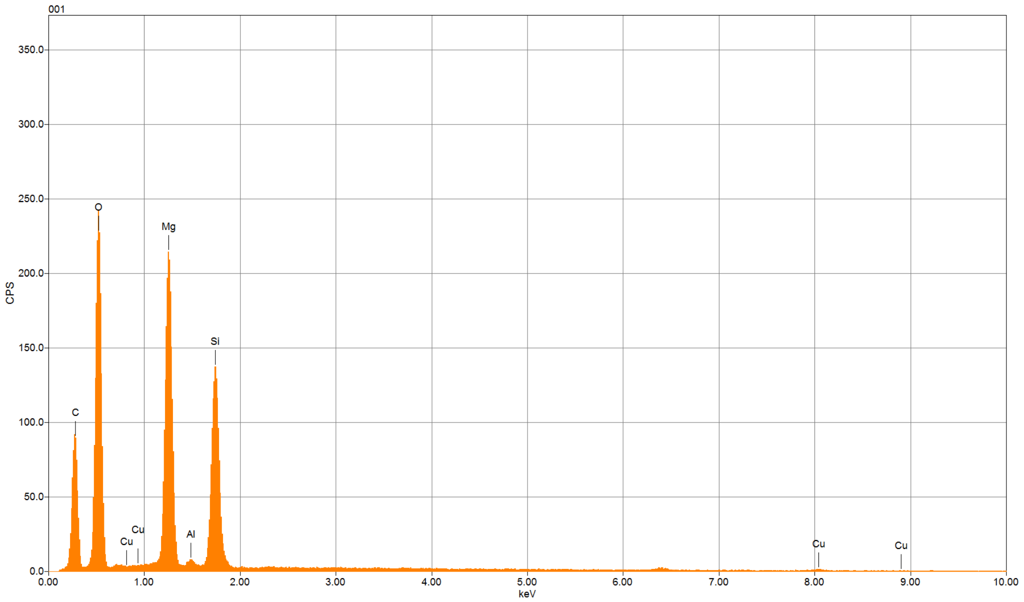
(a) Chrysotile in SEM



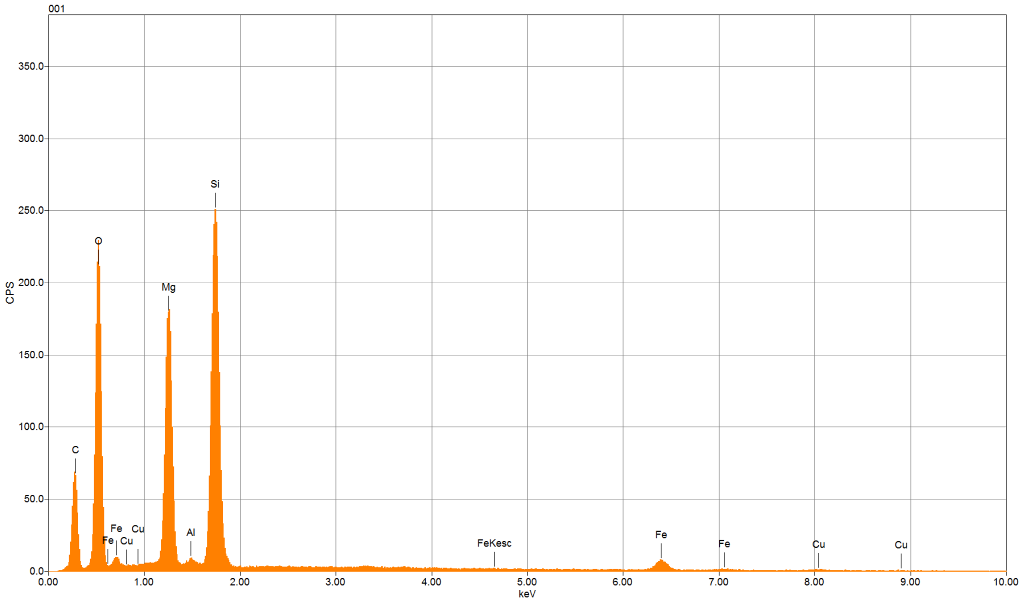
(b) Anthophyllite in SEM

Figure 15: Chrysotile has a wavy or textile-like look while the amphibole minerals look more like sharp needles.

For identification of asbestos fibres with SEM-EDS, the elemental spectra needs to be compared. When a fibre is analysed, a spectrum is produced and this is then compared to known spectra from reference samples (Figure 16, Figure 17 and Figure 18). The spectra in figures 16–18 are of reference asbestos minerals done in low-vacuum with EDS at Top Analytica. The low-vacuum analysis introduces some interference, but the minerals can be clearly identified. The most important part of the spectra is the ratio between elements. This ratio is then compared to the ideal chemical formula of the minerals. As asbestos minerals are natural materials, they can vary somewhat in their actual composition. This is especially true for the amphibole asbestos minerals, as they have several elements that can substitute each other in the crystal lattice. Often there is also some interference from the matrix material in which the asbestos fibres are embedded. Because of this several fibres needs to be analysed.

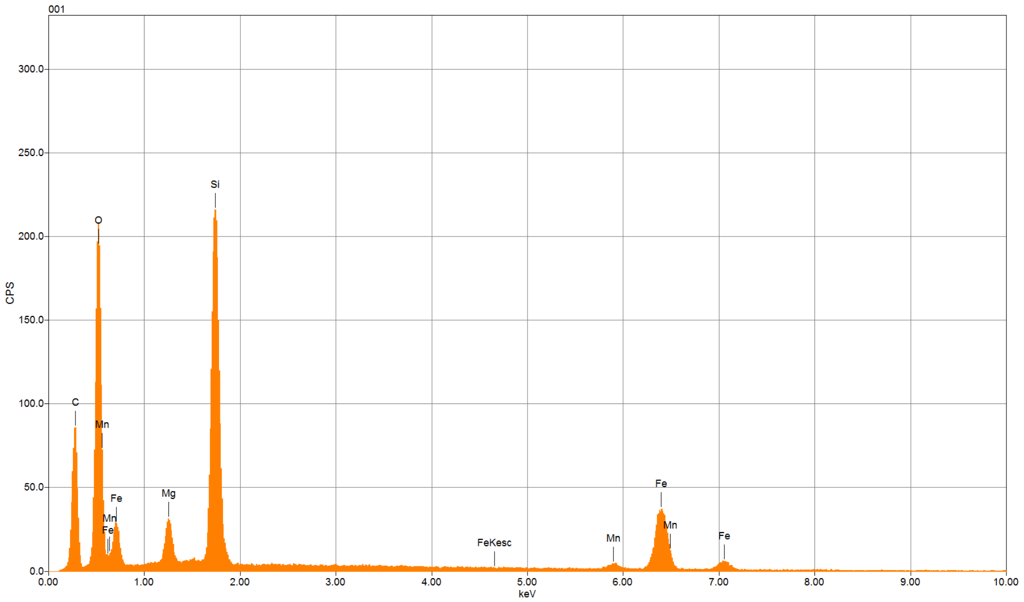


(a) Chrysotile: $Mg_3Si_2O_5(OH)_4$

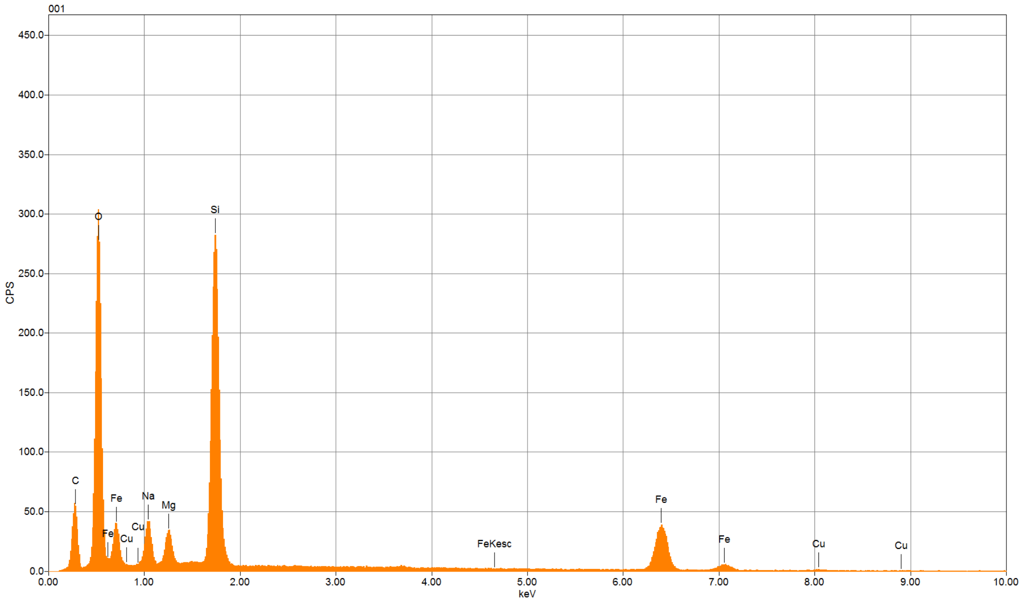


(b) Anthophyllite asbestos: $(Mg,Fe)_7Si_8O_{22}(OH)_2$

Figure 16: The EDS spectra for a reference sample of chrysotile and anthophyllite asbestos. The carbon peak is caused by the low-vacuum atmosphere and copper comes from the sample holder. The oxygen peak is also slightly elevated due to the low-vacuum atmosphere.

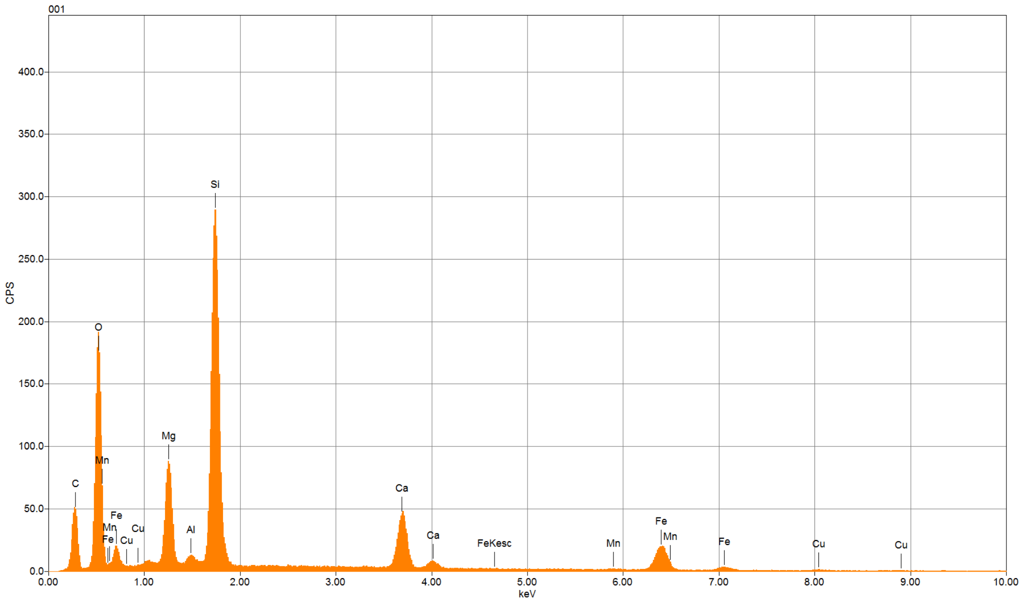


(a) Amosite: $Fe_7Si_8O_{22}(OH)_2$

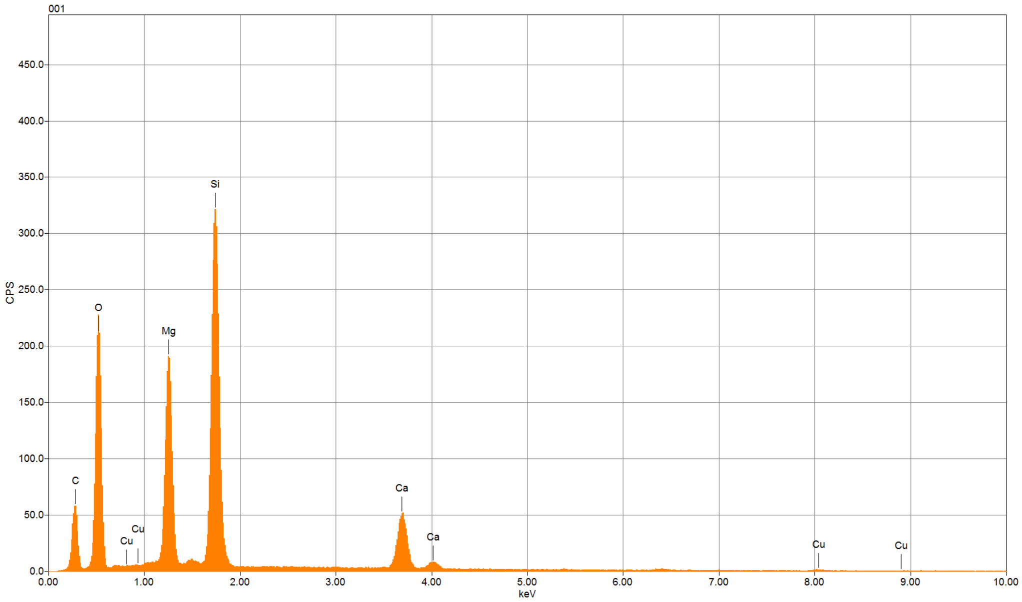


(b) Crocidolite: $Na_2(Fe_3^{2+}Fe_2^{3+})Si_8O_{22}(OH)_2$

Figure 17: The EDS spectra for a reference sample of amosite and crocidolite. The main difference is the significant peak in Na for the crocidolite.



(a) Actinolite asbestos: $Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$



(b) Tremolite asbestos: $Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$

Figure 18: The EDS spectra for a reference sample of actinolite asbestos and tremolite asbestos. All asbestos minerals are mostly made of the same components but in different proportions.

When analysing with the SEM-EDS method, there are several fibres that can look like asbestos fibres. These can erroneously be identified as asbestos. The most common fibres and fibre-like materials that are encountered in construction materials are glass fibres and gypsum crystals (Figure 19). Both can look like asbestos fibres but can clearly be differentiated from asbestos by EDS analysis.

(a) Glass fibres seen in SEM

(b) Gypsum crystals: $CaSO_4 \cdot 2H_2O$

Figure 19: (a) Glass fibres have a tubular morphology and often do not contain iron or magnesium. (b) Gypsum crystals are common in construction materials and can look somewhat like asbestos fibres. Elemental analysis is important for identification of asbestos

5.6 Raman-spectroscopy

The Raman-spectrometer used in this thesis was a Renishaw InVia Qontor confocal Raman microscope equipped with a 532-wavelength laser at the Physics department, University of Turku. All asbestos minerals except crocidolite was analysed at the same power settings, for crocidolite the power output had to be lowered as the laser burned through the thin asbestos fibres.

Raman-spectroscopy is based on a laser hitting the sample and a detector measuring the signal that results. This signal is based on the crystal structure of the sample. Different wavelength lasers are suitable for different materials, as characteristics such as fluorescence hamper detection in some materials. As the laser hits the sample, the detector measures signals at different wavelengths, called Raman shift, and the signal peaks that form allow for very accurate differentiation between many kinds of materials. It is also possible to detect the crystal structure of a mineral and this allows for accurate identification of different minerals that have the same composition. Examples of this are the minerals chrysotile, antigorite and lizardite, which only differ in crystal form. Of these minerals only, chrysotile is classified as asbestos. The use of the Raman method for asbestos analysis is mostly confined to research, as it is too expensive and cumbersome for typical asbestos identification (Rinaudo et al., 2004).

The Raman method allows for the proper identification of asbestos based on the crystal structure. This makes it possible to differentiate between crystal fragments and proper asbestiform minerals. The method does not require coating, vacuum or special liquids in preparation. The measurements are also fast and non-destructive.

A big drawback for the Raman method is that it is hard to get a good analysis of a single fibre; often bundles of fibres are required for identification. As the Raman method is based on light, it cannot detect the smallest asbestos fibres. The equipment is also expensive and it needs to be equipped with a laser that does not induce fluorescence in asbestos fibres.

6 Results

The PLM method was successful in identifying 42 of the 192 samples as containing asbestos and the type of asbestos mineral. It was estimated that 25% of all the samples would contain asbestos based on the previous samples received at the Top Analytica laboratory. Samples 44, 70, 149, 163, 164 and 189 were identified as containing asbestos with the SEM-EDS method but not with the PLM method. In all samples except 149, 163 and 164, the difference between the results were resolved with reanalysis of the samples. The preparation process was improved after each differing result. Sample 149 was identified as anthophyllite in the SEM and was not found in PLM even after reanalysis. This is most likely due to the small size of the fibres. Samples 163 and 164 contained black opaque fibres that were identified as chrysotile asbestos in the SEM but due to the opaque nature, they were impossible to properly identify with the PLM method. Ashing the sample did not remove the opaque layer on the fibres. It is most probable that they were asbestos fibres but coated with some fireproof material. In general, the results might be affected by the limited type of asbestos fibres used in the construction materials. In Finland, only chrysotile, anthophyllite asbestos and crocidolite have been used in significant amounts. Amosite is rarely found and actinolite asbestos and tremolite asbestos has not been used in Finnish construction materials.

The AIMS samples contained a wider variety of asbestos minerals and are more representative of the capabilities of both PLM and SEM-EDS methods. Without dispersion staining it was extremely difficult to differentiate between the different asbestos minerals with the PLM method. This resulted in not being able to identify all asbestos minerals when more than one was present. Dispersion staining would have improved the accuracy of the AIMS results.

With the SEM-EDS method, 47 of 191 samples were identified as containing asbestos. Only in sample 38 was asbestos not detected in SEM-EDS at first. After ashing the sample anthophyllite was detected. With improved sample preparation both methods were able to detect asbestos with greater accuracy.

SEM-EDS was used as a control for the PLM results, as the SEM-EDS method has been in use at Top Analytica since 2016. Sample analysis was conducted simultaneously on both PLM and SEM-EDS as far as it was possible. The results were then compared in batches. The accuracy of SEM-EDS was known, as Top Analytica has participated in several rounds of AIMS testing. The SEM-EDS has passed all rounds of testing with the best possible score.

In both SEM-EDS and PLM, it was possible to differentiate between proper asbestos fibres and other fibrous materials. Glass fibre, wollastonite and organic fibres are the most common source of error in analysis. In the PLM method, the optical characteristics of these fibres are different and in SEM-EDS the morphology and elemental analysis is used to separate asbestos from non-asbestos fibres.

Both methods are suitable for detection of asbestos in bulk materials. In the samples analysed in this thesis, both failed to identify at least one sample which the other identified. This shows that combined the two methods produce the best result. The SEM-EDS is superior overall in the analysis as seen in these results, but it comes at the expense of analysis time and equipment cost. The PLM method can be improved upon to reach the accuracy of SEM-EDS in most materials.

7 Discussion

Three different methods were used to successfully identify asbestos fibres. These three methods were polarized light microscopy (PLM), secondary electron microscopy with EDS (SEM-EDS) and Raman-spectroscopy.

In total 193 unknown samples were compared, approximately 25% of which contained asbestos. The samples were construction materials that were sent to Top Analytica to be analysed for asbestos content. Due to the type of samples, only three types of asbestos minerals were found.

Because the samples were of man-made materials the identification was much easier than natural rock samples would be. This is because the fibres have been deliberately added and are of known type and quantity. In construction materials, the fibres were often found in significant quantities and homogeneously spread. This contrasts with natural samples where the fibres might occur in trace amounts that are heterogeneously spread in the sample. Natural samples might also contain a mix of many different fibrous minerals, whereas construction materials usually contained only one.

The method for PLM identification of asbestos was developed with the help of existing methods and standards. As these methods varied, the goal was to develop a method that could be used at Top Analytica.

The developed PLM method was then compared to the SEM-EDS method and both successfully identified almost all samples with asbestos and the correct asbestos mineral. There were some cases where the PLM method could not adequately identify the asbestos fibres. This was mostly due to sample preparation. The SEM-EDS method missed only one sample. The combination of both PLM and SEM-EDS achieved the best possible accuracy.

7.1 Strengths and weaknesses of PLM

The strength of the PLM method is that it is both fast and does not require expensive equipment. The equipment used is simple and only requires moderate training to use effectively. The identification of fibres in the microscope is quick due to not requiring a vacuum atmosphere like the SEM-EDS. The magnification in a petrographic microscope also covers most use-cases in asbestos identification.

The biggest weakness of the PLM method is the limited magnification. The reliable identification of fibres goes down to 5 μm and shorter fibres cannot be properly identified. There also exists long but extremely thin fibres that cannot be identified in PLM. This is due to the thin fibres not dispersing enough light to be identified. Another issue is small fibres within the sample matrix; the matrix needs to be completely removed to be able to do a proper identification of the fibres. This can sometimes be challenging, as the fibres are easily split into even smaller and thinner fibres in the removal process.

The preparation of samples is also often slower and more laborious for PLM, as the samples need to be broken to smaller pieces than with SEM-EDS. This is due to light needing to pass through the fibres. They must also be free from interference that can be caused by the matrix materials. The fibres also need to be in contact with a refractive index liquid, otherwise proper identification is impossible.

There is also no direct chemical data in PLM, which makes it impossible to identify the fibres in some cases. By refractive index alone it is impossible to tell tremolite asbestos from anthophyllite asbestos as both have the same R.I. The PLM method also requires more training of the analyst and courses and certification are mandatory in some parts of the world (HSE, 2006).

7.2 Strengths and weaknesses of SEM-EDS

A great strength of the SEM-EDS method is the greater magnification produced by electron microscopy as compared to PLM. This allows much easier morphological identification and a possibility to accurately detect fibres less than 5 μm in length. The use of a detector such as the EDS is needed to determine the composition. This allows fibres to be identified with great accuracy. This combination of greater magnification and knowledge of chemical composition makes it easy to differentiate between asbestos fibres and other types of fibres. With a SEM-EDS analysis, only a few fibres are needed for a good analysis.

A weakness of the SEM-EDS method is the significantly more expensive equipment and the need to analyse the sample in vacuum. This poses problems as SEMs without low-vacuum require the sample to be coated. However, even with low-vacuum it takes time to change between samples. This makes it slow to change samples, as the chamber must be brought to atmospheric pressure in-between uses. The matrix that asbestos fibres are found in can also hamper chemical identification. The matrix can have a similar composition to asbestos and if non-asbestos fibres are found, they can be misidentified as asbestos.

8 Conclusion

For the identification of asbestos fibres, there are several possible methods that can be used. In this thesis PLM, SEM-EDS and Raman-spectroscopy were successfully used. Of these methods PLM and SEM-EDS are best suited for rapid, commercial analysis.

During the work on this thesis the importance of sample preparation became apparent. It is impossible to identify fibres if they are still trapped within the sample matrix. The sample preparation methods in use at Top Analytica were improved for both the PLM and SEM-EDS methods.

Of the two methods, the SEM-EDS method has greater magnification and accuracy at the expense of longer analysis time. There is also a higher equipment cost. This better accuracy is crucial in some samples, but most often it is not needed. The PLM is much quicker and with proper training gives almost equal accuracy to the SEM-EDS method. The PLM also has significantly cheaper equipment costs. Both methods have limitations and the best analysis is achieved when both methods are used. The PLM method developed together with this thesis is a general way of doing the analysis. The most important factor in the accuracy of the results is the skill of the analyst.

Raman-spectroscopy was used to successfully identify all six asbestos minerals from bundles of fibres. Raman was also able to identify the different crystal structures of chrysotile which neither PLM or SEM-EDS can do. The use of Raman-spectroscopy was not evaluated for use in commercial bulk material analysis.

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Appendices

Appendix A	Results of PLM and SEM-EDS analysis
Appendix B	AIMS samples
Appendix C	Optical characteristics of chrysotile
Appendix D	Optical characteristics of amphibole asbestos

A Results of PLM and SEM-EDS analysis

The exception column has information if the PLM and SEM-EDS results differed in some way. Otherwise both methods reached the same conclusion.

Table 2: Construction materials analysed with PLM and SEM-EDS

Sample	Material	Detected in PLM	Exception
1	plastic carpet + glue + filler + bonding agent + grout	No	
2	bonding agent + grout	No	
3	floor tile + plaster	No	
4	wall tile + glue	No	
5	plastic carpet + linoleum + glue + filler	No	
6	filler	No	
7	filler	No	
8	linoleum + bonding agent	No	
9	linoleum + glue	No	
10	linoleum + glue	No	
11	grout + bonding glue	No	
12	grout	No	
13	plastic carpet + glue	No	
14	roof filler	No	
15	bitumen felt	Anthophyllite	
16	bitumen felt	Anthophyllite	
17	bonding agent + grout	No	
18	linoleum + glue	No	
19	plastic carpet + glue + glue	Anthophyllite, Chrysotile	

Table 2: **Construction materials analysed with PLM and SEM-EDS**

Sample	Material	Detected in PLM	Exception
20	plastic carpet	No	
21	filler + plaster	No	
22	plastic carpet + glue + filler	No	
23	bonding glue	No	
24	wall paint	No	
25	glue	No	
26	roofing underlay	Anthophyllite	
27	wall panel	No	
28	filler + glue + cardboard	Chrysotile	
29	wall filler	Anthophyllite	
30	plaster + paint	No	
31	plaster + filler	No	
32	jute felt carpet + bonding agent	No	
33	tile bonding agent	No	
34	mortar	Anthophyllite	
35	wall plaster	No	
36	carpet (PVC)	Chrysotile	
37	roofing felt	No	
38	roof material	Anthophyllite	Anthophyllite was not detected in SEM until the sample was ashed
39	bitumen roofing	No	
40	facade panel	Chrysotile	
41	bonding agent + grout	No	
42	bitumen roofing	No	

Table 2: Construction materials analysed with PLM and SEM-EDS

Sample	Material	Detected in PLM	Exception
43	fibreboard	No	
44	bonding agent + grout + plastic carpet + glue + filler	No	Chrysotile was detected in SEM
45	bonding agent + grout + filler	No	
46	roof paint	Chrysotile	
47	plaster	No	
48	plaster	No	
49	bonding agent + grout	No	
50	plaster + paint	No	
51	cement board	No	
52	paint + plaster	No	
53	grout	No	
54	filler	No	
55	bonding glue + grout + bonding agent + filler	Anthophyllite	
56	plastic carpet + glue	No	
57	filler	No	
58	bonding agent + grout + filler	Anthophyllite	
59	bonding agent + grout	No	
60	bonding agent + grout	Anthophyllite	
61	plastic carpet + glue	No	
62	filler + plaster	No	
63	vinyl tile + glue + filler	No	
64	paint + plaster	No	
65	plastic carpet + filler + glue	Anthophyllite	

Table 2: Construction materials analysed with PLM and SEM-EDS

Sample	Material	Detected in PLM	Exception
66	cement board	Chrysotile	Chrysotile, Amosite, Crocidolite detected in SEM
67	wall tile + bonding agent	No	
68	floor tile + bonding agent	No	
69	wall material	No	
70	vinyl floor carpet	No	Chrysotile was detected in SEM
71	plaster	No	
72	plaster	No	
73	wall filler	No	
74	grout + bonding agent	No	
75	plaster	No	
76	glue + plaster	No	
77	floor tile + glue	No	
78	plaster	No	
79	dust sample (textile)	No	
80	stucco/plaster	No	
81	cement board	No	
82	fire resistant mortar	No	
83	vinyl floor tile	No	
84	fibre cement board	Anthophyllite	
85	bonding agent	Anthophyllite, Chrysotile	
86	roof filler	No	
87	roof filler	No	

Table 2: Construction materials analysed with PLM and SEM-EDS

Sample	Material	Detected in PLM	Exception
88	tile + grout + bonding agent + water insulation	No	
89	floor tile + grout + bonding agent + water insulation + filler	No	
90	floor paint + filler	No	
91	filler + plastic carpet	No	
92	grout + bonding agent + filler	No	
93	plastic carpet + glue + floor filler + wall filler + roof filler	No	
94	bonding agent + grout + filler	No	
95	bitumen waterproofing	No	
96	older bitumen waterproofing	No	
97	newer bitumen waterproofing	No	
98	bitumen waterproofing	No	
99	bitumen waterproofing	No	
100	sealing paper	No	
101	cement board	Chrysotile, Anthophyllite	
102	acoustic board	Anthophyllite	
103	wall plaster	No	
104	bonding agent + grout	No	
105	bonding agent + grout	No	
106	floor tie + bonding agent	No	
107	wall plaster	Anthophyllite	
108	wall plaster	No	

Table 2: Construction materials analysed with PLM and SEM-EDS

Sample	Material	Detected in PLM	Exception
109	cement board	Anthophyllite, Crocidolite	
110	wall plaster	Chrysotile	
111	cement board	Chrysotile, Anthophyllite	
112	aerated concrete	No	
113	floor paint	No	
114	bonding agent + grout + filler	Anthophyllite	
115	wall plaster	Anthophyllite	
116	plastic carpet + filler + glue	Chrysotile, Anthophyllite	
117	bitumen felt	No	
118	bitumen felts	No	
119	plastic carpet + bonding agent + grout + filler	No	
120	bonding agent + grout + filler	No	
121	floor filler	No	
122	tile + bonding agent + filler	No	
123	floor tile + bonding agent + casting surface	No	
124	plastic carpet + bonding agent + plaster	No	
125	plastic carpet + jute carpet + glue + filler	Anthophyllite	
126	wall plaster	No	
127	bonding agent + grout + filler	Anthophyllite	
128	wall material	No	

Table 2: Construction materials analysed with PLM and SEM-EDS

Sample	Material	Detected in PLM	Exception
129	cement board	No	
130	bonding agent + grout + filler	No	
131	pipe insulator mass	Anthophyllite, Crocidolite	
132	pipe insulator + smoothing mass	Anthophyllite	
133	pipe insulator mass	Anthophyllite, Crocidolite	
134	plaster	No	
135	plaster	No	
136	bonding agent + grout + filler + gypsum board	No	
137	bonding agent + grout + filler	No	
138	floor carpet + glue + filler	No	
139	plastic carpet + glue + filler	No	
140	bonding agent + grout	No	
141	plastic carpet + glue	No	
142	bonding agent + grout + filler	No	
143	bonding agent + grout + filler	Anthophyllite	
144	bonding agent + grout + filler	Anthophyllite	
145	bonding agent + grout + filler	No	
146	bonding agent + grout + filler	No	
147	bonding agent + grout + filler	No	
148	bonding agent + grout + filler	Anthophyllite	
149	tile + grout + bonding agent	No	Too small fibres to identify in PLM. Anthophyllite detected in SEM
150	plastic carpet + glue + filler	No	

Table 2: Construction materials analysed with PLM and SEM-EDS

Sample	Material	Detected in PLM	Exception
151	filler	No	
152	vinyl carpet + filler	No	
153	plastic carpet + glue	No	
154	plastic carpet + glue	No	
155	pipe insulator cardboard + black paper	No	
156	filler	No	
157	bonding agent + grout + filler	No	
158	linoleum + glue + filler	No	
159	jute felt carpet + glue	No	
160	hovi tile + glue + filler + mass layer	Anthophyllite	
161	plastic carpet + filler + mass layer	Anthophyllite	
162	vinyl carpet + black glue	Chrysotile	
163	jute felt carpet + black glue	No	Opaque fibres, unable to detect properly in PLM. Chrysotile detected in SEM
164	vinyl carpet	No	Opaque fibres, unable to detect properly in PLM. Chrysotile detected in SEM
165	linoleum + glue + filler	No	
166	plastic carpet + black glue	No	
167	plastic carpet + glue	No	
168	acoustic wool bonding agent	No	
169	linoleum + glue	Chrysotile	
170	plastic carpet + black glue	Anthophyllite	
171	linoleum	No	

Table 2: Construction materials analysed with PLM and SEM-EDS

Sample	Material	Detected in PLM	Exception
172	linoleum + felt layer	No	
173	linoleum	No	
174	linoleum	Anthophyllite	
175	wall paper + bonding agent	No	
176	filler	No	
177	plastic carpet + glue + filler + mass layer	Anthophyllite	
178	bonding agent + grout + filler	No	
179	plastic wall paper + glue + filler	No	
180	wall paper + filler	No	
181	pipe insulator	No	
182	plastic carpet + glue	No	
183	fillers	No	
184	plastic carpet + glue + filler	No	
185	filler	No	
186	plastic carpet + glue + filler	No	
187	bonding agent + grout + filler	No	
188	filler	No	
189	plastic carpet + glue + filler	No	Chrysotile detected in SEM
190	plaster + paint	No	
191	plaster + paint	No	

B AIMS samples

Table 3: AIMS samples in PLM and SEM-EDS

Sample number	Sample description	Detected in PLM	Detected in SEM
R60/1	Paper	None	None
R60/2	Painted Board	None	None
R60/3	Cement	Crocidolite	Crocidolite
R60/4	Plaster	Chrysotile[1]	Chrysotile, Amosite
R61/1	Plaster	Chrysotile	Chrysotile
R61/2	Felt & Bituminous Paint	Amosite	Amosite
R61/3	Painted Board	Chrysotile	Chrysotile
R61/4	Cement	None	None
R62/1	Textile	None	None
R62/2	Plaster	None	None
R62/3	Cement	Tremolite/Anthophyllite[2]	Chrysotile, Anthophyllite
R62/4	Powder	Tremolite/Anthophyllite[3]	None
R62A/1	Lagging	Amosite	Amosite
R62A/2	Insulation	None	None
R62A/3	Fibreboard	Chrysotile, Crocidolite	Chrysotile, Crocidolite
R62A/4	Plaster	Chrysotile[4]	Actinolite
R63/1	Grout	Tremolite/Anthophyllite	Anthophyllite
R63/2	Board	Crocidolite	Crocidolite
R63/3	Cement	None	None
R63/4	Mixed Debris	None[5]	Chrysotile
R64/1	Board	Chrysotile	Chrysotile
R64/2	Cement	Chrysotile, Crocidolite[6]	Chrysotile, Crocidolite, Amosite
R64/3	Textured Coating	Anthophyllite	Amosite
R64/4	String	Chrysotile	Chrysotile

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[1] Amosite was not detected due to lack of dispersion staining

[2] Anthophyllite was not detected due to lack of dispersion staining

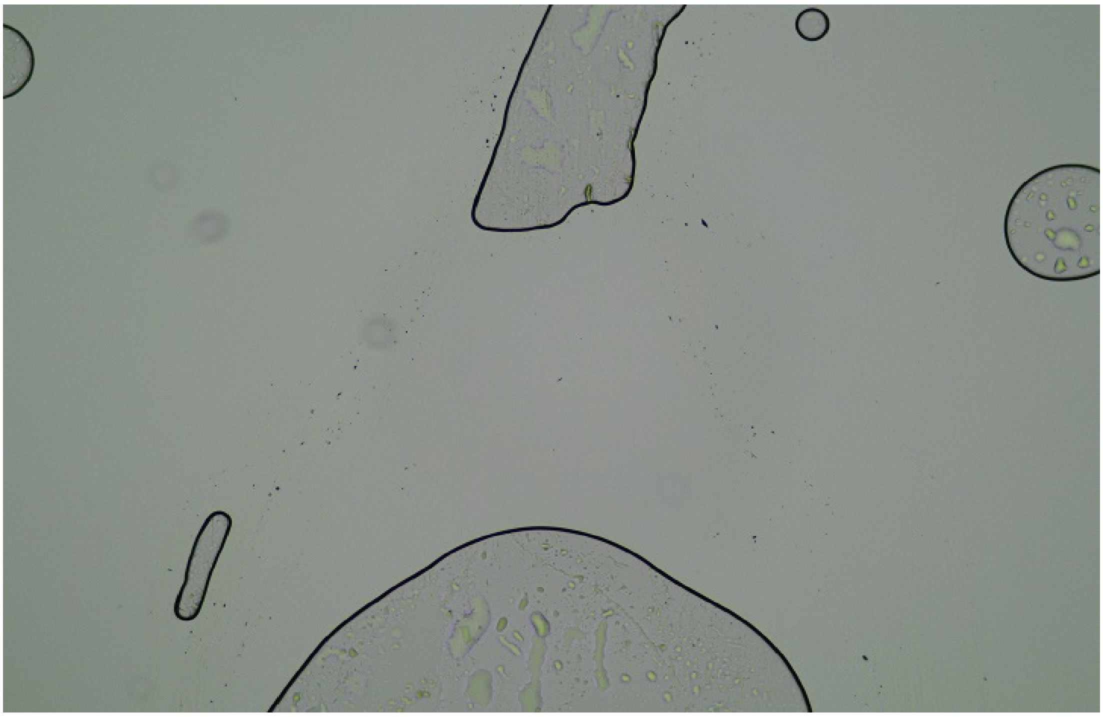
[3] A reanalysis of the sample revealed the detected asbestos to be wollastonite

[4] Asbestos content was very low, a reanalysis of the sample identified the asbestos as actinolite

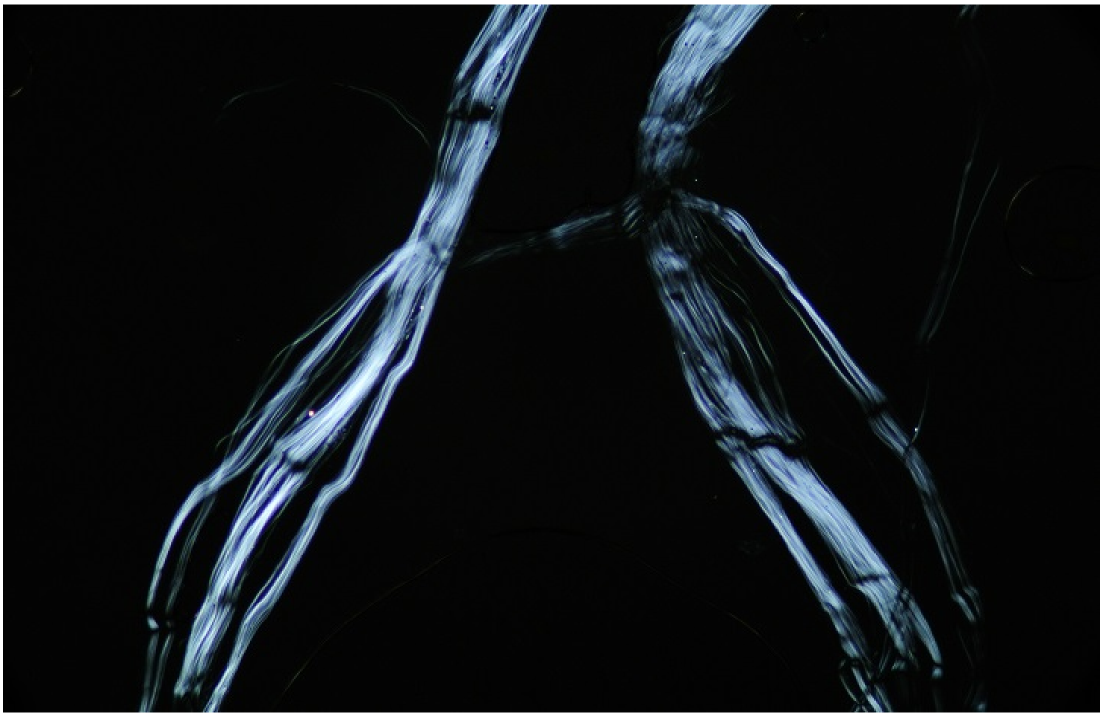
[5] Due to interfering organic fibres the sample needed to be ashed before asbestos was detected in PLM

[6] Amosite could not be separated from the other asbestos minerals without dispersion staining

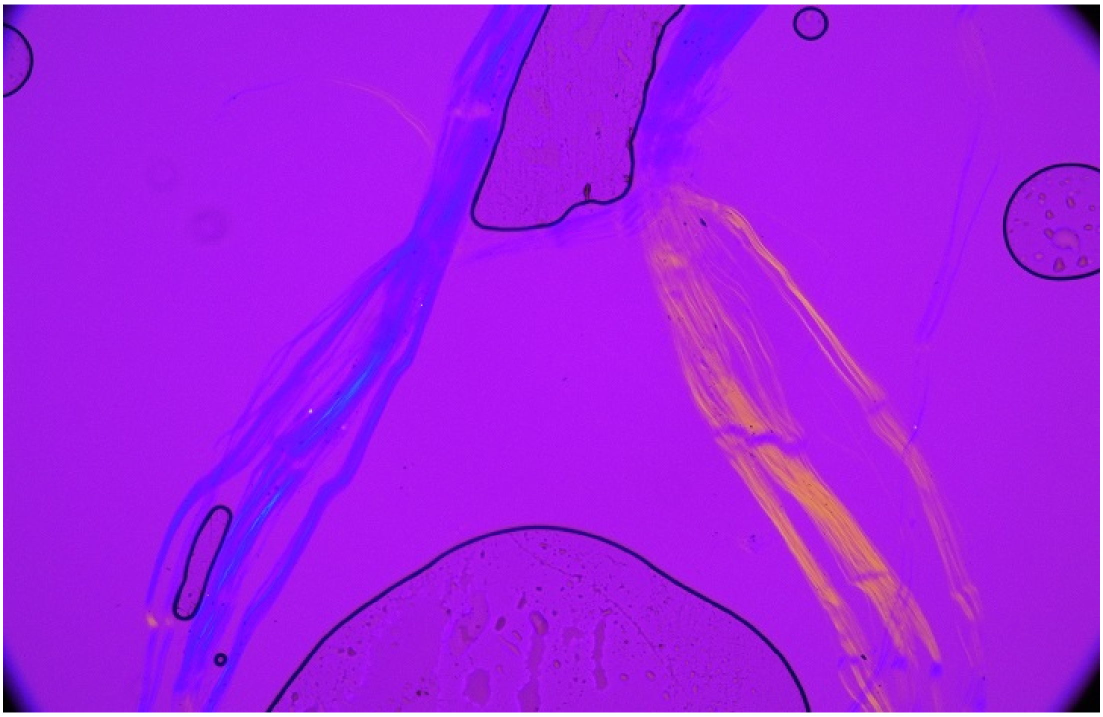
C Optical characteristics of chrysotile



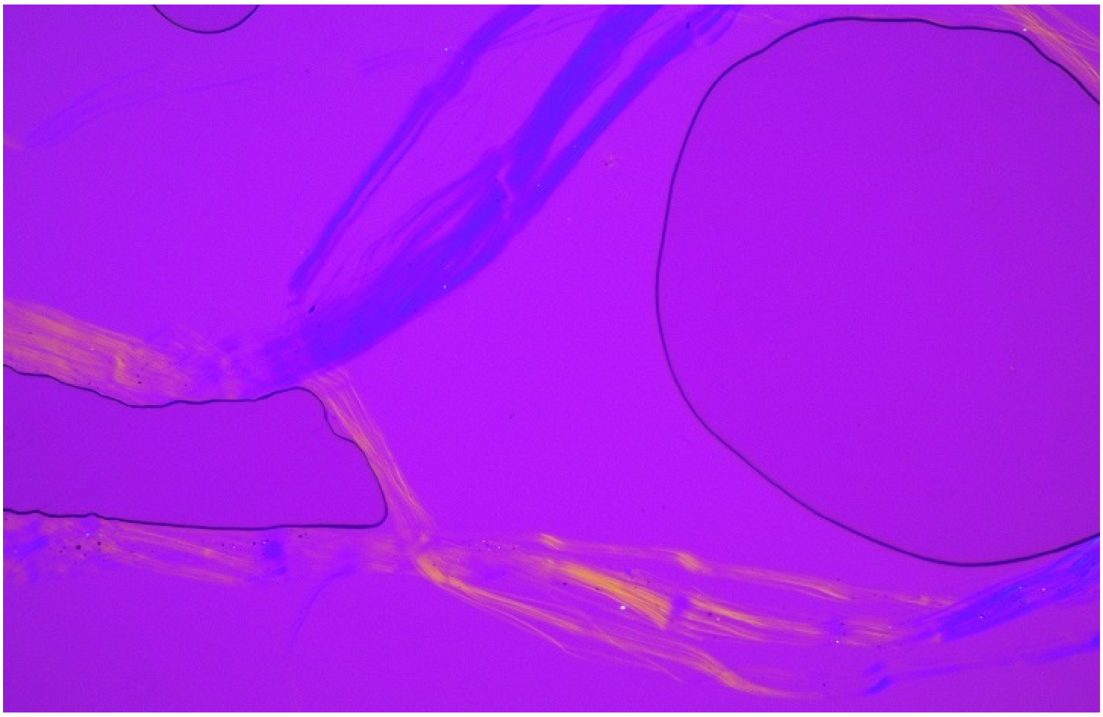
(a) Chrysotile in plane-polarized light. The chrysotile fibres are barely seen as they are in a liquid with the same refractive index. Taken with 125x magnification.



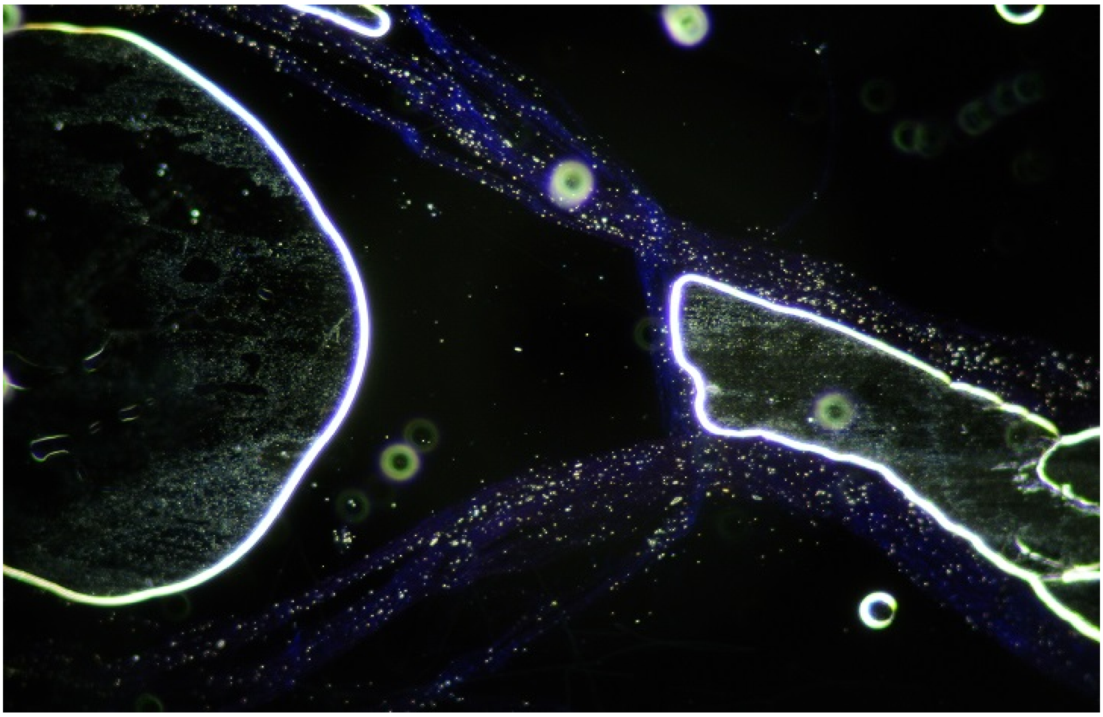
(b) In cross-polarized light the same chrysotile fibres are clearly visible. They also show an even colouring that is only seen in mineral fibres. Taken with 125x magnification.



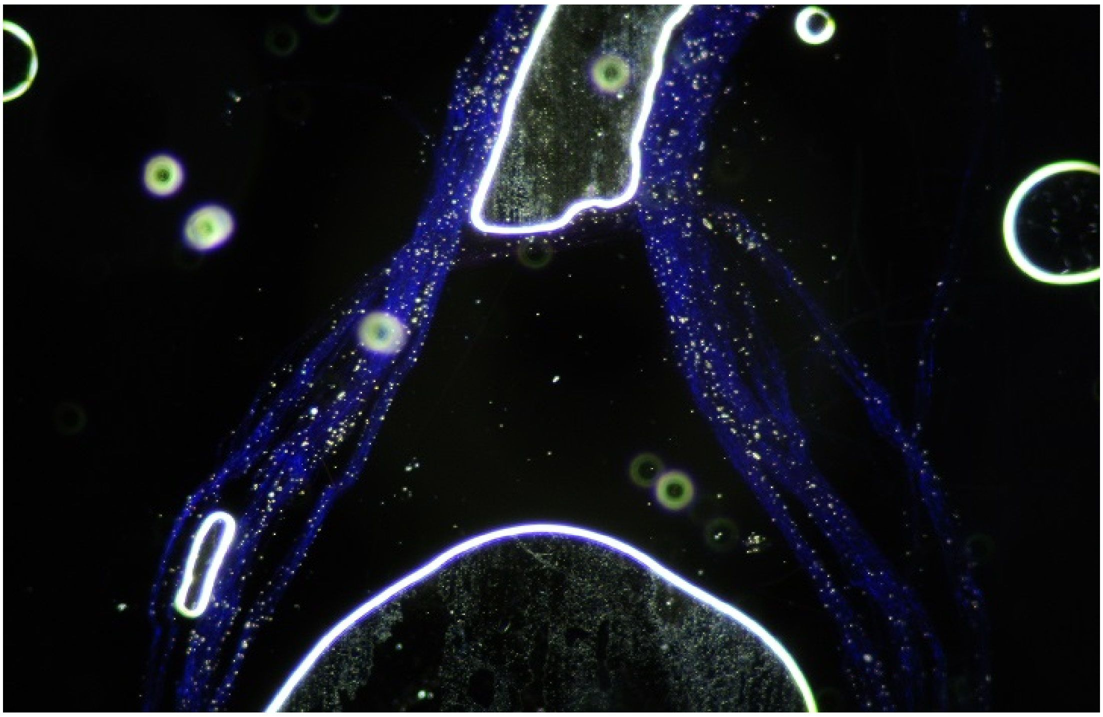
(c) When the gypsum plate is inserted in cross-polarized light the fibres show the sign of elongation. All asbestos fibres except crocidolite have the same sign of elongation. Taken with 125x magnification.



(d) When the fibre is rotated 90° degrees the colour changes. The easiest way to find asbestos fibres in the sample is with cross-polarized light with the gypsum plate inserted. Taken with 125x magnification.



(e) When chrysotile is in the correct R.I it shows blue-magenta colours in dispersion staining. Taken with 125x magnification.

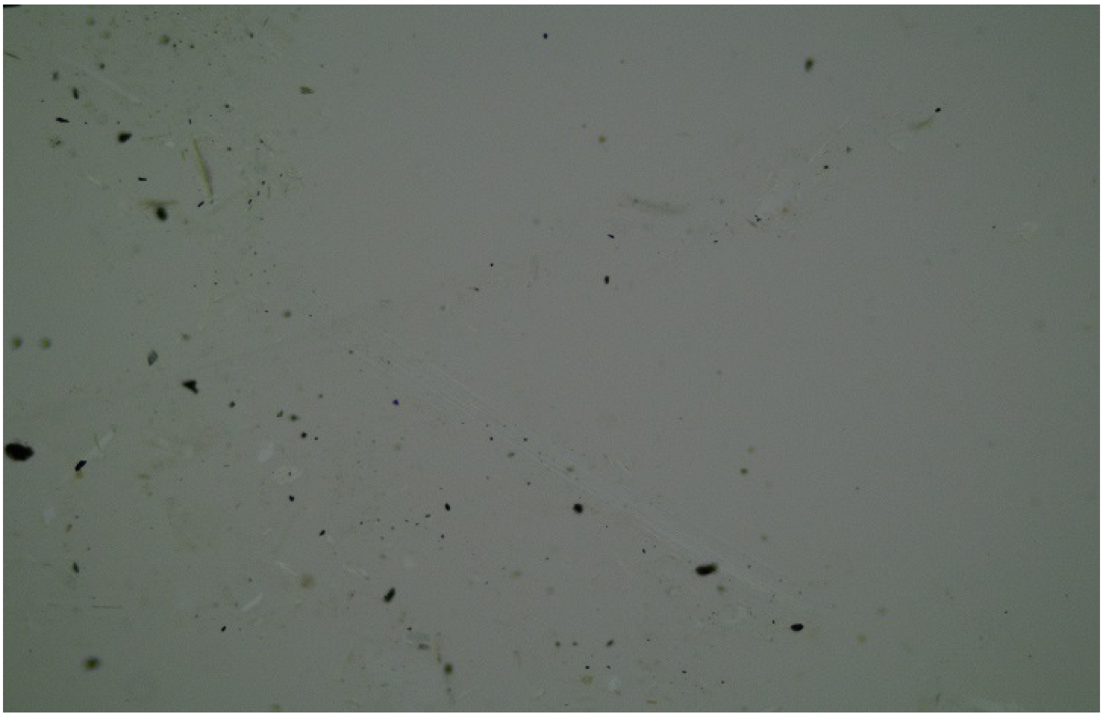


(f) By rotating the sample, the colour changes between blue and magenta. Taken with 125x magnification.

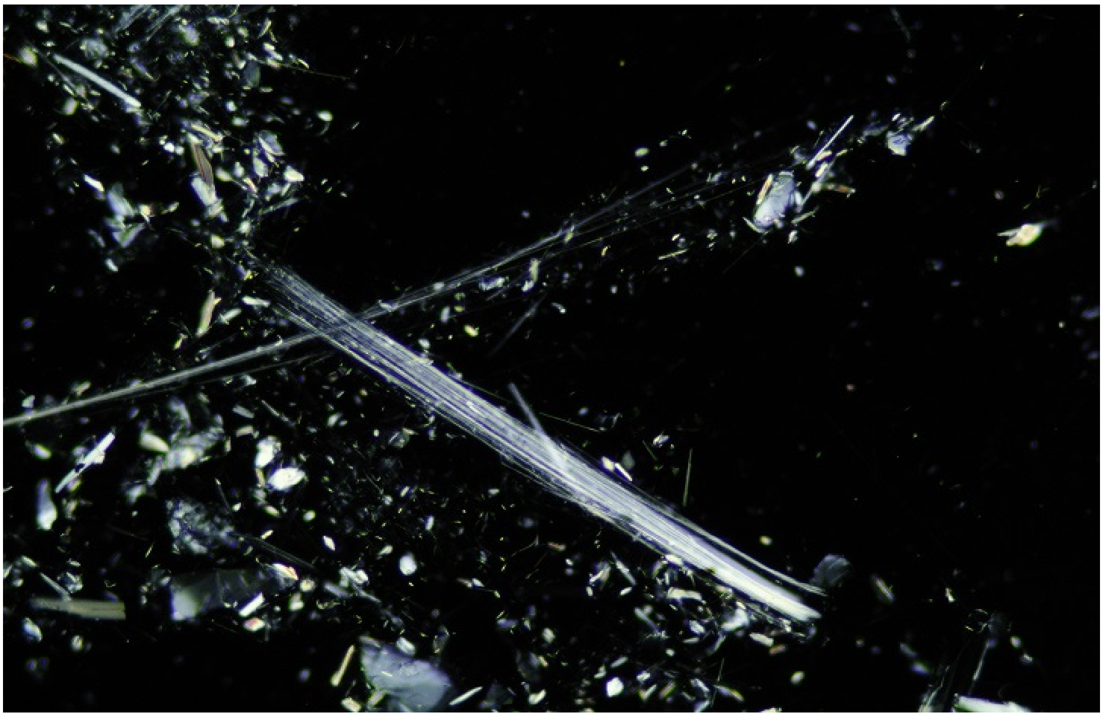
(a) When an asbestos fibre is in the wrong R.I it can clearly be seen in plane-polarized light. Taken with 125x magnification.

(b) In dispersion staining the fibres look white. In this case the asbestos is chrysotile in a liquid with a R.I of 1.6700. Taken with 125x magnification.

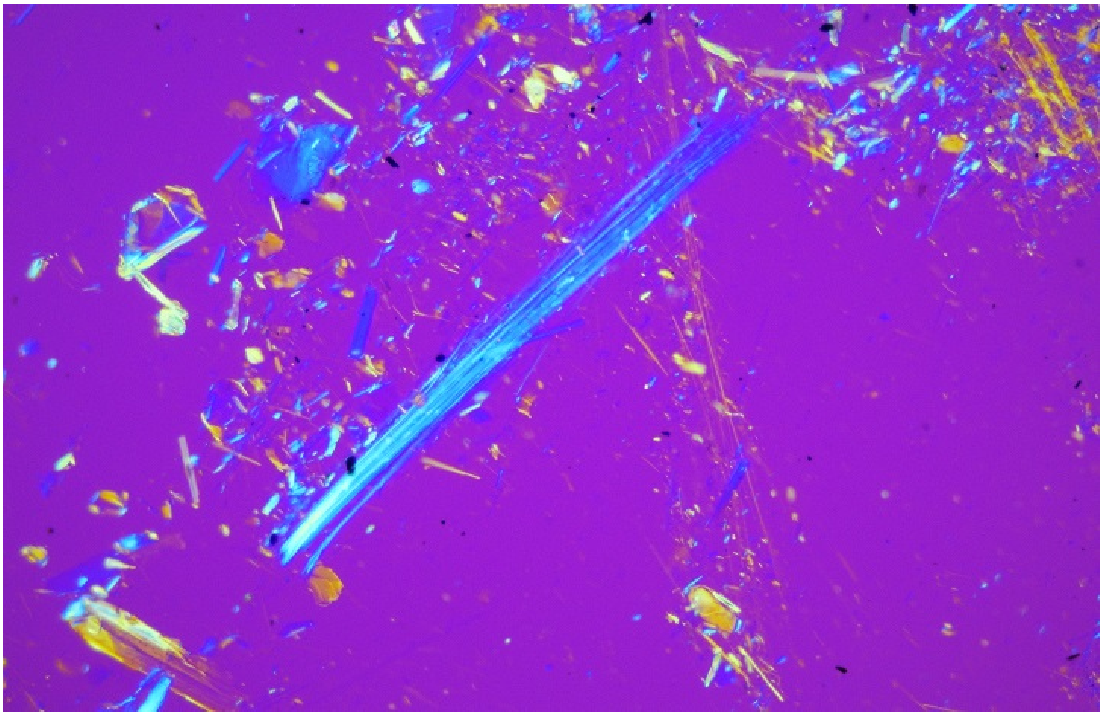
D Optical characteristics of amphibole asbestos



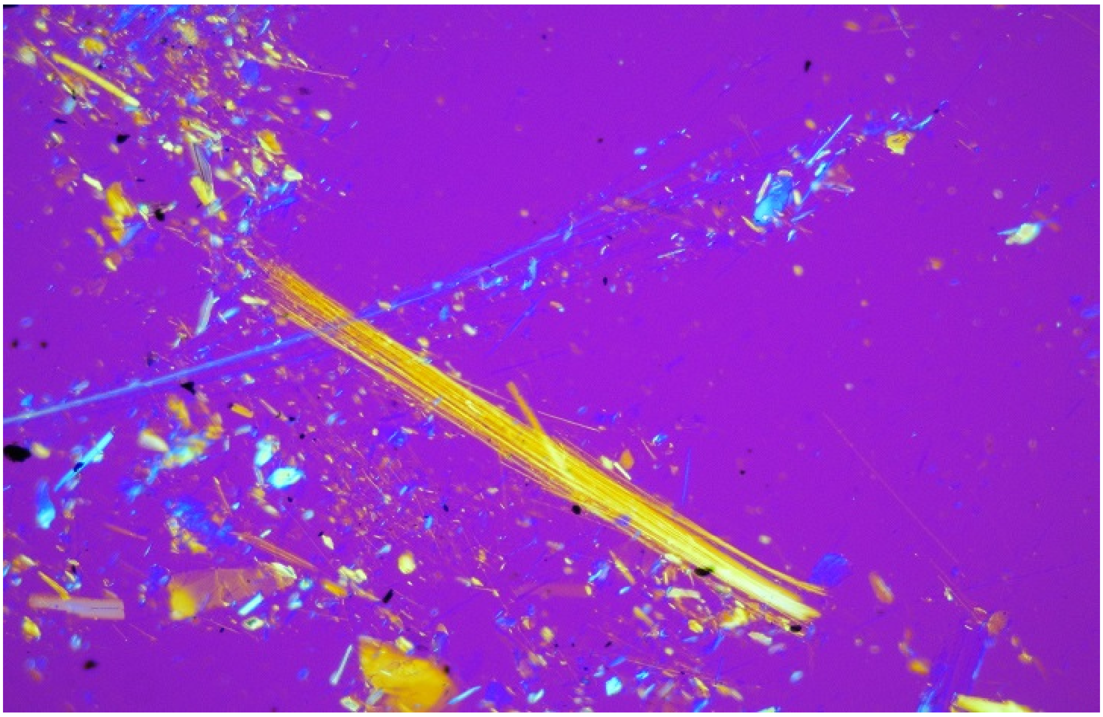
(a) Anthophyllite asbestos fibres are almost invisible in plane-polarized light when in the correct R.I. Taken with 125x magnification.



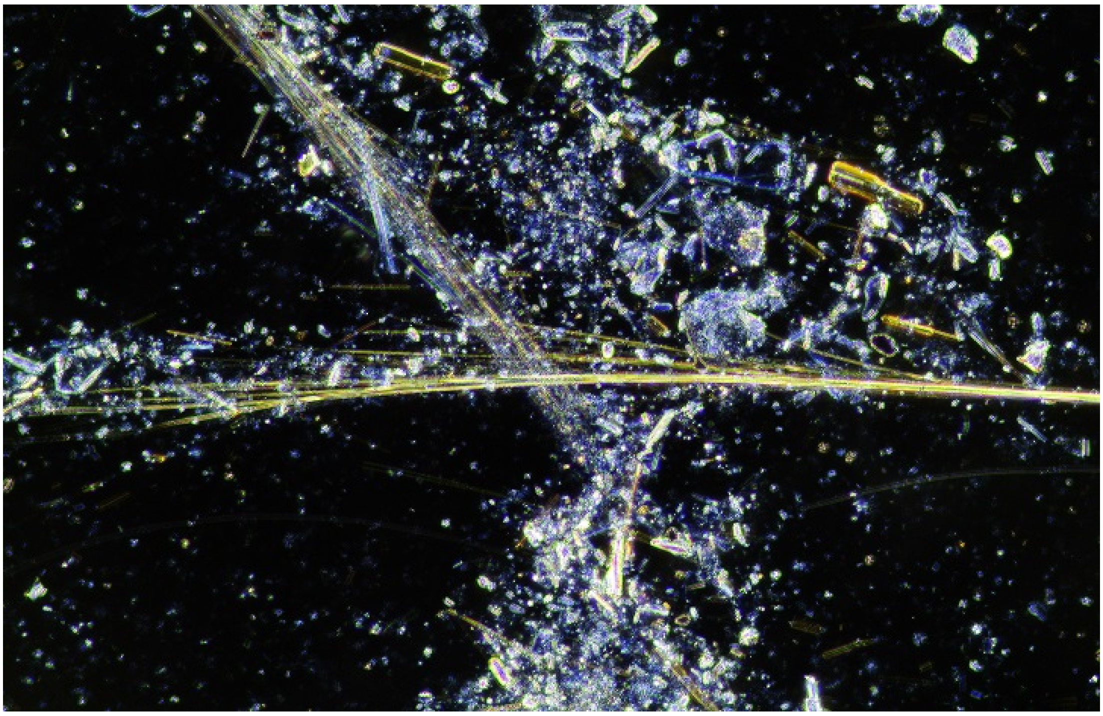
(b) When viewed in cross-polarized light the fibres are clearly visible. The fibres show even colouration typical for mineral fibres. Taken with 125x magnification.



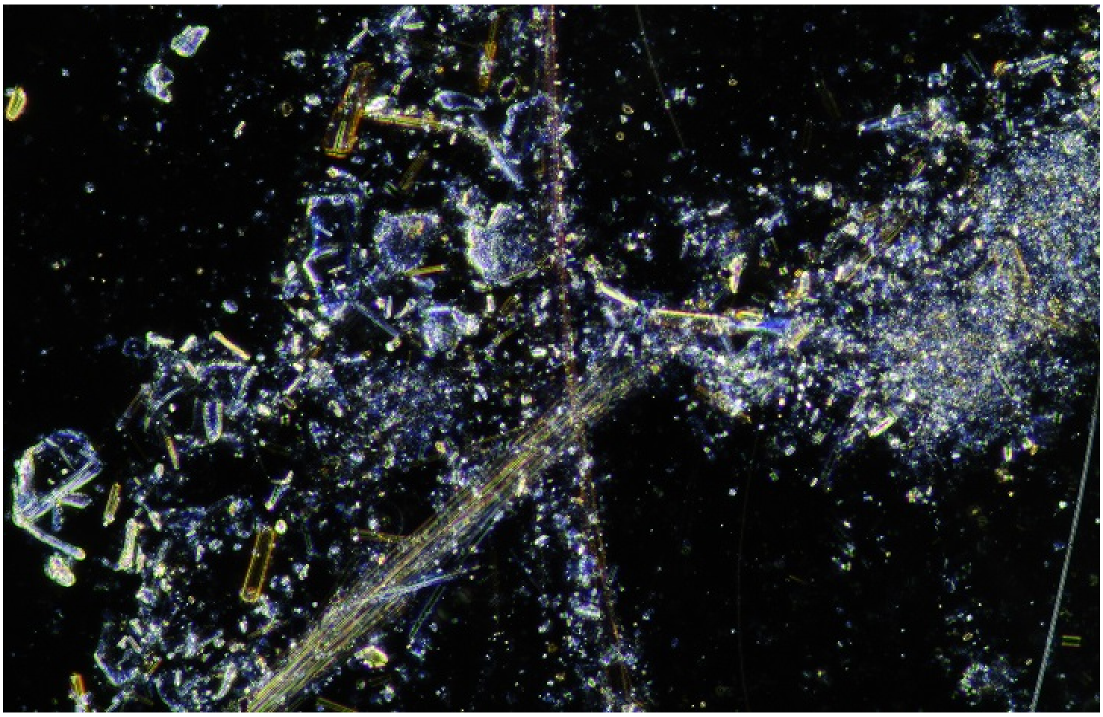
(c) Asbestos fibres show clear colours in cross-polarized light with the gypsum plate inserted. This shows the sign of elongation. Taken with 125x magnification.



(d) The same fibres rotated. Taken with 125x magnification.



(e) Anthophyllite shows a golden and blue colour in the correct R.I liquid when viewed in dispersion staining. Taken with 125x magnification.



(f) Rotating the sample shows both colours. Taken with 125x magnification.