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Detection and Measurement of the Concentration of Asbestos in Air*

Bepaling van de asbestconcentratie in lucht

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Summary

The attractive mechanical and chemical properties of asbestos have resulted in a steep rise in asbestos consumption since the 50's. Increasing evidence indicates that inhalation of even small quantities of asbestos can be detrimental to health.

Surveillance for the concentration of asbestos in air is therefore highly desirable.

As investigations of ambient air indicate that asbestos is mainly present as sub-microscopic fibers, it is necessary to determine these small particles.

The qualitative and quantitative aspects of the detection of asbestos fibers are treated and the determination of higher asbestos concentrations, as found during the manufacturing of asbestos containing materials, has been taken into consideration.

Samenvatting

Door de aantrekkelijke mechanische en chemische eigenschappen van asbest is het gebruik van dit materiaal de laatste decennia sterk toegenomen. Tevens nemen sedert de jaren zestig de aanwijzingen toe dat het inademen van zelfs zeer kleine hoeveelheden asbest, zoals die in omgevingslucht voorkomt, schadelijk kan zijn. Onderzoek naar de concentratie asbest in omgevingslucht is daarom gewenst.

Uit tot nu toe verricht werk is gebleken dat de in de omgevingslucht voorkomende asbestvezels overwegend submicroscopische afmetingen hebben.

Ingegaan wordt op de kwalitatieve en kwantitatieve detectie van sub-microscopische asbestvezels.

De bepaling van asbestconcentraties optredend tijdens het fabriceren en verwerken van asbest en asbesthoudende materialen, wordt eveneens behandeld.

Introduction

„Heatproof" and „shockproof" are words which are frequently used in advertising and, though advertising is not entirely objective, it indicates that both are highly valued properties of materials. Asbestos combines the properties of both heat and shock resistance.

Asbestos is the term applied to a group of natural occurring inorganic fibers belonging to two groups of silicate minerals known as amphiboles and serpentines.

Their discovery dates back to prehistoric times. They were first described by Plutarch who introduced the name „αοβεστα", meaning indestructible. Later, Charles V amazed and amused his guests by throwing tablecloths into a fire, from which they usually emerged whiter than when they went in [2]. Up to the end of the last century, asbestos remained a curiosity with very few applications. The discovery of large asbestos deposits in Canada gave a great impetus to its applications. Due to its unique mechanical and chemical properties, its use grew very rapidly as shown in figure 2 [3].

Table 1 [4] gives an estimate of the asbestos consumption in some European countries, while in table 2 [4], an estimate of its use on a world wide basis is shown.

Table 1. *Estimated asbestos consumption in a number of highly industrialised European countries in 1970.*

	tons per jaar
Belgium	59.000
France	167.000
Italy	109.000
Netherlands	24.000
U.K.	170.000
Western Germany	192.000

Table 2. *Estimate of major uses of asbestos. Breakdown in % of the total asbestos production.*

asbestos textiles	2
asbestos cement	66
friction materials and gaskets	4
asbestos paper	7
floor tiles	10
paints, roof coatings, caulks, etc.	3
plastics	1
miscellaneous	7
	100%

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Fig. 1. Chrysotile asbestos fibers (2750 × C.E.M. Central Lab. TNO).

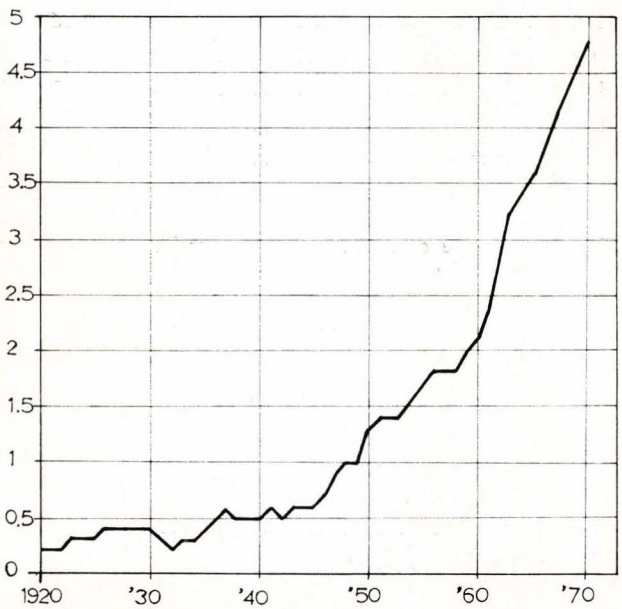

Fig. 2. Production of asbestos in 10^6 ton per year

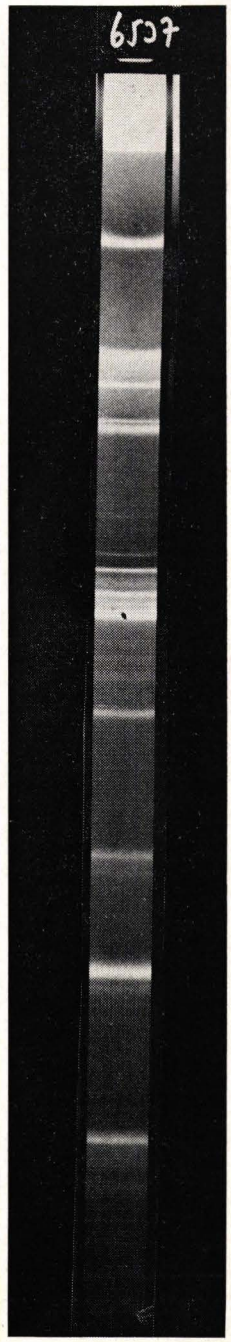

Fig. 3. X-ray diffraction of chrysotile asbestos (T.P.D. - T.H. - TNO).

Unfortunately, evidence becomes more and more convincing that asbestos, which from a technological point of view is an outstanding material, represents a serious health hazard — vide the article of Planteijdt in this same issue of „TNO-Nieuws". It is not surprising that attempts are being made to reduce its use: (a) by safety regulations which govern the precautions which have to be taken during the handling of asbestos [5, 6] and (b) by application of substitute products [7, 8].
Vigilance as to its concentrations in air is a necessity. The measurement problems arising from this need can be divided into two parts:

1. Determination of asbestos fibers in air under industrial conditions (e.g. work on asbestos containing materials);
2. Measurements of asbestos concentrations in ambient air (e.g. in buildings made of asbestos-containing construction materials, and in outdoor air).

In The Netherlands not much is known of the asbestos concentration of ambient air. Indications are that the concentrations are low (a few fibers per 1000 cm^3) and consist of submicroscopic fibers (diameter smaller than 0.1 μm, length in the order of 0.1 μm). Quantitative determinations of the number of fibers are difficult to make. The evaluation of methods to obtain reliable data is one of the projects of the Project group on Asbestos Exposure which was established by the Organization for Health Research TNO.
Methods pertaining to both problems are discussed in the subsequent paragraphs.

Qualitative and quantitative detection of asbestos in air under industrial conditions [9]

When selecting methods of investigation for the determination of asbestos fibres in dust samples precipitated from the air, the concentration to be expected is an important criterion.
Outside the asbestos-processing industry, the dust samples will contain but few asbestos fibres among much material of a different nature. In that case, high demands are made on the methods of investigation as regards selectivity and sensitivity. This problem is treated in the next paragraph. If the concentration is higher, e.g. during manufacturing of asbestos containing materials, light microscopy is an attractive analytical tool.
The dust trapped on a membrane filter is examined under the microscope. For this purpose, the filter is made transparent by means of glycerol triacetate. Since in normal light-microscopy the refractive indices of the transparent filter and of the asbestos fibres are virtually the same (so that the asbestos fibres cannot be properly distinguished), use is made of phase contrast. By means of this technique, fibres having a diameter of ca. 0.5 μm can still be detected. By definition, one speaks of fibres when the length to diameter ratio is at least 3 : 1 [10].
The presence of fibres other than asbestos fibres is possible, but in a low concentration they do not interfere. In phase contrast microscopy, synthetic fibres are lighter and straighter, while cotton fibres can never be brought into sharp focus over their entire length. Moreover, when the focusing adjustment is shifted, the latter fibres seem to curl up.
The relative accuracy of the count is $\pm$ 20%.
If the concentration of asbestos in the dust is higher, it can be determined by means of physico-chemical methods, i.e. with the aid of x-ray diffraction or infra-red spectrophotometry.

X-ray diffraction is based on the phenomenon that the atoms that are vibrating around fixed points in the mineral act as centres of scatter. The diffraction pattern (Fig. 3) is characteristic, while the intensity is a measure of the quantity of the asbestos present [11]. The sensitivity of the determination is ca. 0.5 mg asbestos. The asbestos content of the dust sample should be at least 3%.

Infrared spectroscopy is based on the phenomenon that the vibrating atoms in the mineral can absorb energy. However, only certain wavelengths can be used to magnify the amplitude of the vibrations of the atoms (resonance). At these wavelengths increased absorption of electromagnetic radiation occurs (Fig. 4). The absorption spectrum is characteristic, and the extent of the absorption is a measure of the quantity of asbestos. The sen-

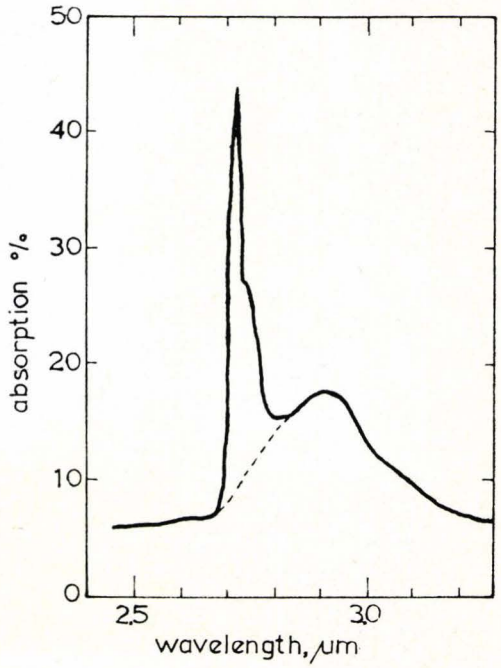

Fig. 4. Infrared spectrum of chrysotile asbestos

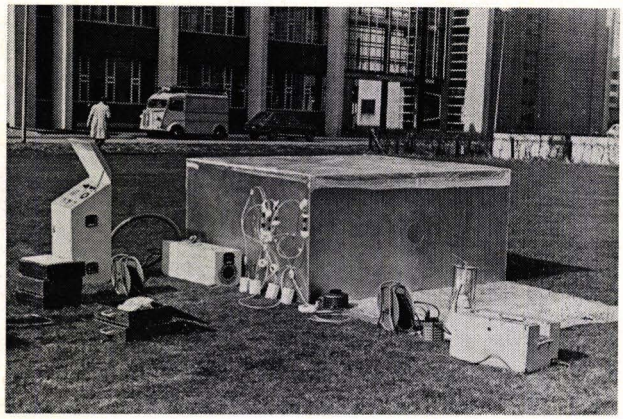

Fig. 5. Evaluation of several methods used for the determination of asbestos concentrations by creating a homogeneous dust concentration in the box shown on the picture

sitivity of determination is ca. 10 μg, while the asbestos content of the sample should be ca. 1% [12, 13].

Finally, it can be mentioned that when the airborne dust contains much asbestos, methods may be used that are not specific. Examples are atomic absorption and emission spectroscopy [9]. Within their limitations these are sensitive methods.
As asbestos is built up of elements which are very common such as magnesium, aluminium, and iron, these elements can only be used as indicators for the asbestos concentration if the concentration level of the asbestos is such that it stands out clearly against the always present background of these elements.

Detection of sub-microscopic asbestos fibers

The classic method of determining the concentration of sub-microscopic particles is by precipitation of the particles from a known volume of air on a substrate. For this purpose, among others, the following techniques can be used: filtration, electrostatic of thermal precipitation, and centrifugation [14, 15, 16, 17]. The particles are subsequently counted by use of electron microscopy. The problems which arise center around reproducebility and whether the numbers of particles counted are representative. The last two conditions are especially difficult to meet when filtration methods employing high polymer filters are used for the separation of solid particles from a known volume of air. The reason is that relatively elaborate manipulations have to be performed in order to prepare a suitable specimen for electron microscopy. Yet this method offers great advantages if long term averages of the concentration have to be determined. The instruments which are at our disposal are based on the alternative techniques mentioned earlier. They are all short time sampling instruments (sampling between 5-20 minutes). Using these instruments for the determina-

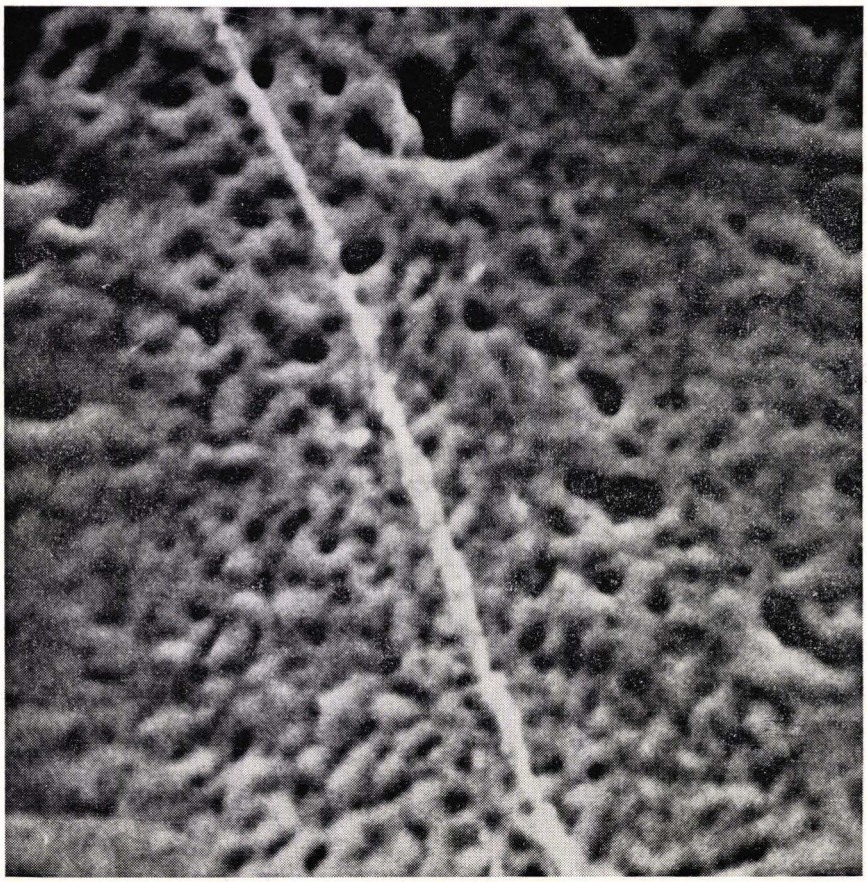

Fig. 6. Scanning electronmicrograph of a dust sample, as used for qualitative and quantitative evaluation of asbestos in air (11.550 × C.E.M. Central Lab. TNO).

tion of „long time averages" would be extremely costly. Finally it should be mentioned that asbestos, especially chrysotile, disintegrates easily into many fine fibers. The techniques used must prevent this as far as possible.
The latter property makes long time sampling e.g. with electroprecipitators, where the dust is continuously washed from the electrodes by a liquid, less attractive.
For evaluation purposes, the „filter method" was compared with direct short time sampling methods. In the latter case, the dust is collected on a specimen holder which can be directly used for electron microscopy.
The comparison was made by installing a number of filter samplers together with the short time samplers (thermal precipitator or electrostatic precipitator) in a box. Under the experimental condition the asbestos concentration, though fluctuating in time, is at a certain moment the same in the whole box (figure 5).
Using this method, it has not been possible to obtain comparable results when preparing specimens from filters for transmission electron microscopy. On the other hand, by taking samples from a filter, ashing them at 400° C in a golden specimen holder, and viewing the remaining deposit by a scanning electron microscope (stereoscan) [18] reproducible results are obtained (figure 6).
A disadvantage of using the scanning microscope (Cambridge Stereoscan) is the lower resolution (0.05 μm) as compared with the transmission electron microscope ($\sim$ 0.001 μm). Another drawback is that identification under the scanning microscope is only possible by X-ray fluorescence analysis of the elements which constitute the particle under observation; no information on crystal structure is given.
The latter information is provided by electron diffraction analysis which is accomplished by electron microscopy (figure 7a and b) [19, 20].
As it is believed that there is a connection between crystal structure and biological activity, information on crystal structure is important when structural transformations come into play under the influence of high temperatures. An example is braking. Use of asbestos containing brake linings results in the spreading of asbestos fibers into the surrounding air. According to some authorities, the asbestos decomposes at the temperatures which prevail during braking [21]. Therefore, work is still in progress which attempts to find a suitable sampling method for this situation.
Finally, it should be mentioned that the lowest practical level of detection with the stereoscan is 10^{-14} gr; with the electron microscope $\sim$ 10^{-15} gr. In principle, there is no limit to the amount of other material which may accompany the asbestos. The percentage of asbestos which can still be detected in the sample, is a function of the time

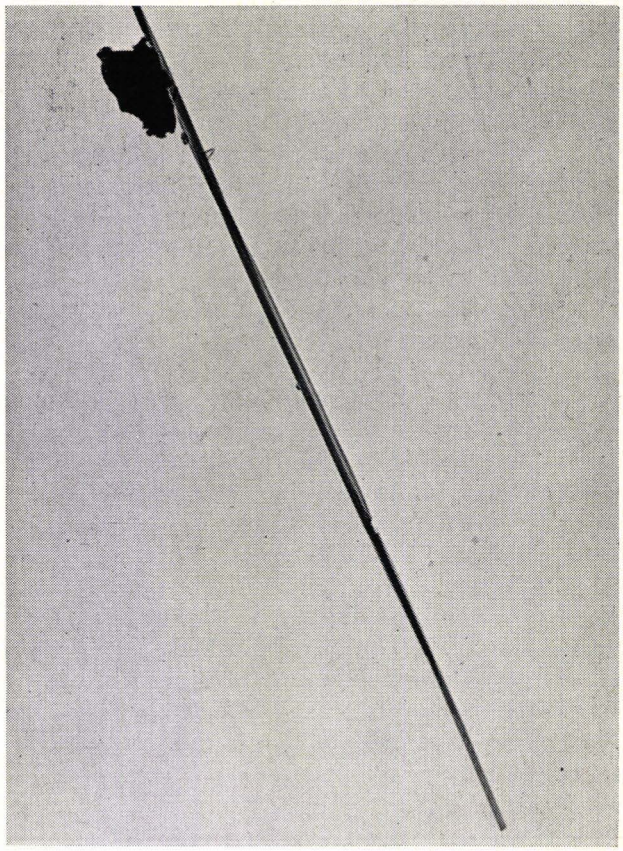

Fig. 7a. Electronmicrograph of chrysotile asbestos (14.500× Metal Institute TNO)

Fig. 7b. Electron diffraction pattern of the same fiber.

used to screen the samples. It is not an inherent limitation of the instrument as is the case with infrared and X-ray diffraction analysis which was mentioned in the preceding paragraph.
Though electron microscopy is a time consuming, and therefore expensive analysing tool, it is at present, by many orders of magnitude, the most sensitive detector available for the problem at hand.

Acknowledgement

Modern research sometimes requires complicated and expensive instruments and skills in order to obtain the desired results. These are not always found in one institute. In our case we should like to thank the Central Laboratory TNO, Metal Research Institute TNO, Technical Physics Department TNO-TH, Drs. J. F. van de Vate, Reactor Centrum Nederland and Ir. A. R. Kolff van Oosterwijk, Hertel & Co. N.V., member of the Project Group on Asbestos Exposure TNO, established by the Organization for Health Research TNO, for their interest and co-operation in the work described.

References

[1] Biological aspects of asbestos, pp. 255-335, Annals of the New York Academy of Science, December 31, 1965.

[2] Ullmanns Encyklopädie der technischen Chemie IV, p. 40, Urban & Schwarzenberg, München — Berlin, 1953.

[3] Stumphius, J., De Veiligheid *46* (1970) 2, p. 52 and information provided by the „Economische Voorlichtingsdienst", Ministry of Economic Affairs, The Hague

[4] Private communications.

[5] Asbestos Health Precautions in Industry, Department of Employment and Productivity HMSO.

[6] Werken met asbest, P. no. 116, Arbeidsinspectie.

[7] Kolff van Oosterwijk, A. R.: Toepassing van asbest De Ingenieur *82* (1970) 14, p. G 43.

[8] Mellenbergh, J. H. C.: Vervangingsmiddelen voor asbest.
De Veiligheid *46* (1970) 9, p. 293.

[9] Keenan, R. G. and Lynch, J. R.: Techniques for the detection, identification and analysis of fibers.
Am. Ind. Hyg. Ass. J. *31* (1970) 5, p. 587.

[10] Technical Note 1. The measurement of airborne asbestos dust by the membrane filter method.
Technical note 2, Dust sampling procedures for use with the asbestos regulations 1969, The Asbestosis Research Council, Secretary Dr. S. Holmes, P.O. Box 40, Rochdale, Lancashire, England.

[11] Goodhead, K. and Martindale, R. W., The Analyst *94* (1969) 1124, p. 985.

[12] Flick, K.: Beitrag zur Untersuchung von silikogenen und asbesthaltigen Stäuben mit Hilfe der Infrarot-Spektralphotometrie.
Arbeitsschutz (1969) 7.

[13] Gadsden, J. A., Parker, J. and Smith, W. L.: Determination of chrysotile in airborne asbestos by an infrared spectrometric technique.
Atm. Environ. *4* (1970) 6, p. 667.

[14] Walkenhorst, W.: Feinste Siebe für die quantitative Probenahme von Schwebestoffen zur elektronenmikroskopischen Auswertung.
Staub Reinh. Luft. *30* (1970) 6, p. 246.

[15] Van de Vate, J. F.: Luchtstofbemonstering.
Innovatie 2 (1972) 5, p. 7.

[16] Walkenhorst, W.: Staubprobenahme mit dem Thermalpräzipitator unter besonderer Berücksichtigung elektronenmikroskopischer Auswertung.
Staub *15* (1955) 40, p. 241.

[17] Stöber, W. und Zessack, U.: Zur Messung von Aerosol Teilchen-grössenspektren mit Hilfe von Zentrifugalabscheidern.
Zbl. biol. Aerosolforschung *13* (1966), p. 263.

[18] Cosslett, V. E.: Modern Microscopy.
Bell & Sons, London, 1966.

[19] Skikne, M. I. a.o.: Electron diffraction patterns of U.I.C.C. Asbestos samples.
Environmental Research *4* (1971) 2, p. 141.

[20] Gross, P. a.o.: Asbestos versus non asbestos fibers.
Arch. Environ. Health *20* (1970) 5, p. 571.

[21] Lynch, J.: Brake lining decomposition products.
J. A.P.C.A. *18* (1968) 12, p. 824.