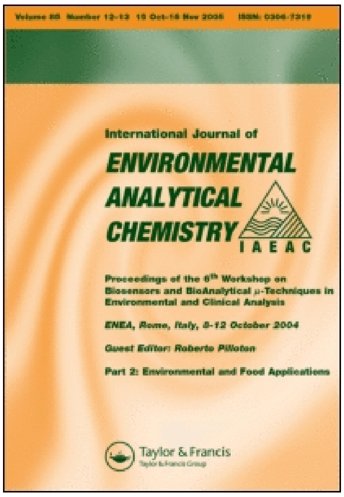

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About the Quantitative Determination of Chrysotile Asbestos and Alpha Quartz in Airborne Dusts by X-ray Diffraction†

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In the framework of the JRC indoor pollution program an X-ray diffraction method has been developed for the determination of chrysotile asbestos, alpha quartz and christobalite in breathed air. The limit of detection for chrysotile was about $10\,\mu g$ and for α quartz $5\,\mu g$. Calibration curves and precision of the technique are reported. Several outdoor and indoor air samples have been collected on membrane filters and were analysed. The results are discussed.

KEY WORDS: X-ray diffraction, quantitative determination, alpha quartz, chrysotile asbestos, airborne dusts.

INTRODUCTION

Asbestos or asbestiform minerals include several types or groups of fibrous crystalline substances with special thermal and electrical properties that have long encouraged their industrial use in the manufacture of such products as roofing, insulation, brake linings, fireproof curtains, etc.

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Their occurrence as pollutants in the ambient air and in supplies of food and drinking water has caused considerable concern because occupational exposures to asbestos have been found to induce mesothelioma of the pleura and peritoneum as well as cancer of the lung, esophagus and stomach, after latent periods of about 20 to 40 years.[1–3]

Table I lists the asbestos minerals including the asbestiform varieties of anthophyllite, tremolite and actinolite.

The first three minerals chrysotile, crocidolite and amosite, designate specifically the fibrous or asbestiform varieties of their vastly more abundant nonfibrous counterparts. In contrast, the latter three amphiboles-anthophyllite, tremolite and actinolite, include all forms of these very common rock forming minerals which rarely occur in fibrous or asbestiform habit.

TABLE I

Asbestiform variety	Chemical composition	Non-asbestiform variety
Chrysotile	$Mg_3(Si_2O_5)(OH)_4$	Antigorite Lizardite
Crocidolite	$Na_2Fe_3Fe_2(Si_8O_{22})(OHF)_2$	Riebeckite
Amosite	$(MgFe)_7(Si_8O_{22})(OH)_2$	Cummingtonite
		Grunerite
Anthophyllite asbestos	$(MgFe)_7(Si_8O_{22})(OHF)_2$	Anthophyllite
Tremolite asbestos	$Ca_2Mg_5(Si_8O_{22})(OHF)_2$	Tremolite
Actinolite asbestos	$Ca_2(MgFe)_5(Si_8O_{22})(OHF)_2$	Actinolite

The inhalation of, one or more of the polymorphic forms of silicon dioxide-quartz, cristobalite or tridymite—leads to silicosis, known as miners black lung disease. The silica particles in the lungs form modules which block the normal gas exchange in the lungs. This leads to difficult breathing and a shortened lifespan. Since the free silica in industrial dusts is primarily alpha-quartz, and chrysotile comprises nearly 90% of all asbestos used worldwide, we have studied, in the framework of the Indoor Pollution Program, the possibility to determine these 2 compounds using the X-ray diffraction technique.

EXPERIMENTAL

A Philips PW1140 X-ray generator was used in combination with a vertical goniometer and a proportional counter.

All measurements were obtained using a copper X-ray tube run at a power level of 1400 W. The instrumental configuration employed in this study included a 1° receiving slit and a 2° compensating divergence slit. A pulse height selector and a focusing graphite monochromator were used to reduce background.

Silver membrane filters, pore size 0.45 μm, have been utilised as a support for the standard samples. Integrated peak areas were measured through the use of a step scanning mode.

Chrysotile and alpha quartz were ground and sieved. A weighed amount of the sieved fraction was brought into suspension and deposited by filtration on the silver membrane filters.

RESULTS AND CONCLUSIONS

1. Chrysotile asbestos

A series of chrysotile calibration samples was prepared by the diluted suspension technique. By filtrations, amounts of 20 to 1000 μg of chrysotile were collected on silver membrane filters. The (002) diffraction peak at 7.36 Å has been used as analytical line. Figure 1 shows the calibration curve for known amounts of chrysotile. The detection limit for chrysotile, defined as the amount of material required to give a diffracted X-ray intensity equal to three times the standard deviation of the background was found to be about 10 μg. Precision of the method is $\pm 10\%$ in the 10–50 μg concentration range.

If for the collected dust thin film assumptions can be considered, then the measured X-ray intensity, above background, is directly proportional to the mass per unit area of the chrysotile asbestos. For samples of less than infinite thickness the active dilution technique must be used or corrections for X-ray absorption by the analyte and

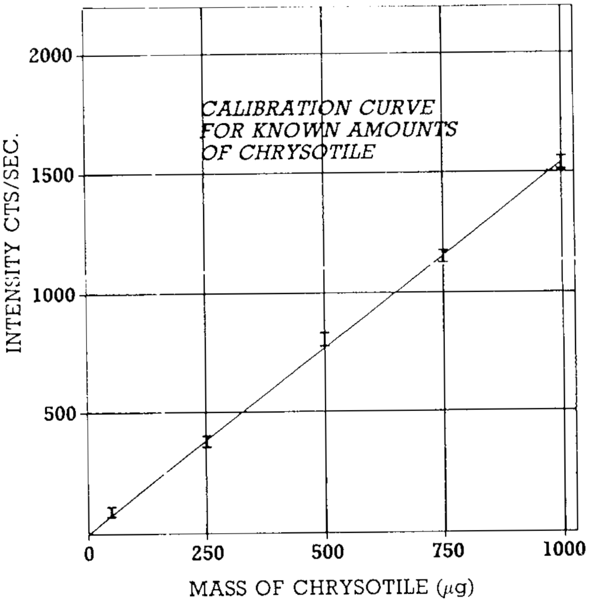

FIGURE 1 Calibration curve for known concentrations of chrysotile asbestos.

any co-existend matrix must be made[4] using the equation

$$I_c = I_m \frac{F \ln R}{1 - R^F}$$

where I_c is the corrected intensity, I_m the measured intensity, $F = \sin\theta_{\mathrm{Ag}}/\sin\theta_{\mathrm{Chr}}$ and $R = I_{\mathrm{Ag}}/I_{\mathrm{Ag}^\circ}$ where I_{Ag} and I_{Ag° represent the intensity of the attenuated and unattenuated silver peaks respectively. A drawback of the method is the problem of interferences.

Minerals such as antigorite, lazardite, members of the kaolinite group and possibly chlorite are potentially serious interferences with chrysotile. However adequate sample treatment and X-ray line profile analysis can correct for these interferences.

Considering the limit of detection found with our equipment, it is evident that for airborne dusts collected on filter membranes this method can only be employed successfully at working places in the asbestos industries or in the neighbourhood of large asbestos factories, owing to the large volumes of air which have to be collected. Although detection limits in the submicrogram range can be achieved, a minimum detectable amount of 0,15 μg has been reported,[5] using electrostatically aligned asbestos fibers and a double-detector X-ray diffraction system, the more frequently used methods for trace chrysotile analysis are phase contrast microscopy, scanning and transmission electron microscopy with limits of about 0.01 μg.

Nevertheless, the X-ray diffraction technique could be of some interest in indoor pollution studies, in analysing bulk samples, as collected for instance by a cleaner, to identify and eventually determine the presence of crystalline compounds.

2. Alpha quartz

Membranes with known amounts of quartz for establishing an analytical curve were prepared by dispersing a known weight in a volume of water, followed by deposing the suspension on silver filters. The strongest quartz diffraction line is the (100) reflexion at 3.34 Å, which has been used. Other quartz diffraction peaks are, in general, too weak for accurate quantitative measurement, since sensitivities to quantities less than 100 μg quartz are usually required. The X-ray pattern showing peak heights for 25–75 and 175 μg amounts of quartz are given in Figure 2.

A limit of sensitivity of approximately 5 μg of alpha quartz can be achieved. A precision study gave a relative standard deviation of 7% in the 50–100 μg concentration range.

To compensate for self-absorption effects due to the sample matrix, the addition of an internal standard is advisable. Fluorite has been used extensively because it has a strong diffraction line which is close to the 3.34 Å line of quartz. Therefore samples collected on organic membranes are ashed, the internal standard fluorine added and the mixture transferred to a silver membrane.

It should be pointed out that any crystalline material having a diffraction line at 3.34 Å will be determinated as alpha quartz. Some materials that will cause interferences with this quartz line, are muscovite, biotite, sillimanite, graphite and aragonite. The interference due to graphite can be eliminated by ashing the sample in a low-temperature asher prior to deposition on the silver filter.

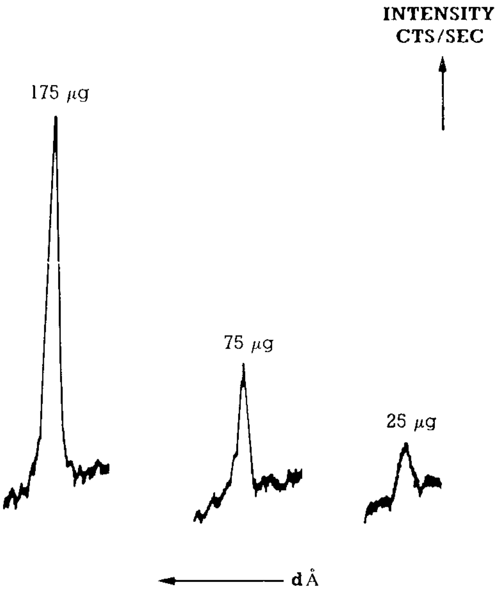

FIGURE 2 X-ray diffraction peaks for different amounts of α quartz.

Aragonite and iron oxides can be removed by treatment with dilute acid, whereas a chemical separation using pyrophosphoric acid, removes the silicate minerals, but leaves the silica minerals.

The application of X-ray diffractometry to the identification of particulate air contaminants is demonstrated by the Figures 3 and 4, showing the X-ray diffraction patterns of airborne dusts, collected by a low volume total suspended particulate sampler. The sampled air volume was about $30\,m^3$ for both samples, collected on millipore membrane filters, with pore width of $0.45\,\mu$.

The sample in Figure 3 was collected in Turin (urban area) whereas the X-ray pattern in Figure 4 represent air particulate from the Ispra surroundings (semi-rural zone).

It can be observed that there are no great differences in the cristalline compounds content between the 2 samples.

The clay minerals illite, chlorite and kaolinite, α Fe_2O_3 and α quartz were detected in both samples. These latter was determinated and resulted to be about $1.8\,\mu g/m^3$ air for both samples. Remarkable

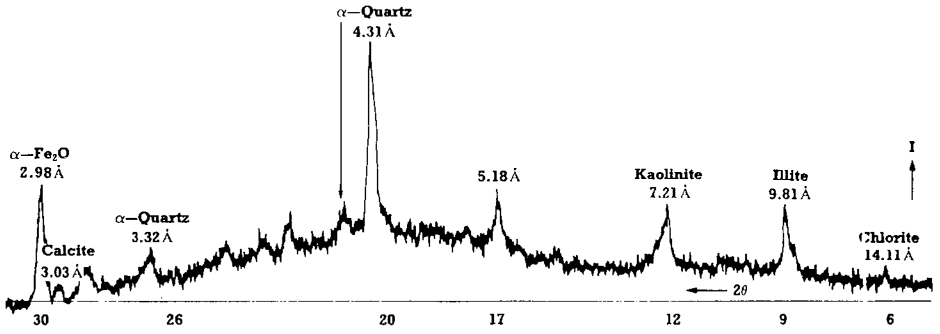

FIGURE 3 X-ray diffraction pattern of airborne particulate matter collected at Turin, Italy.

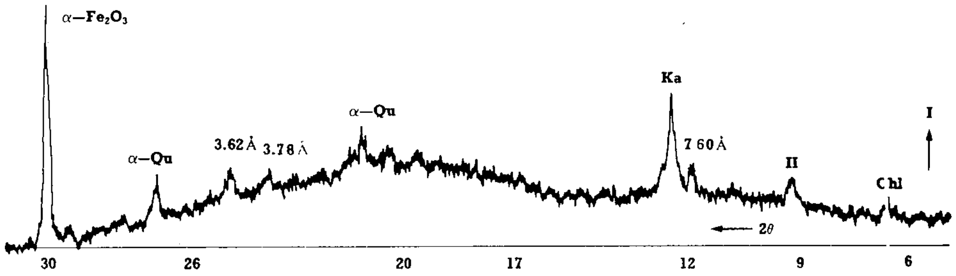

FIGURE 4 X-ray diffraction pattern of airborne dust collected at Ispra, Lago Maggiore, Italy.

is the possible presence of ammoniumsulfamate (4.31 Å and 5.18 Å peaks) in the Turin sample.

Figure 5 finally gives the X-ray diffraction scan of a dust sample collected by a cleaner in a residential building near Ispra. The presence of much more cristalline substances, yet unidentified, and a much higher α quartz concentration, in comparison with the air-borne dusts samples are remarkable.

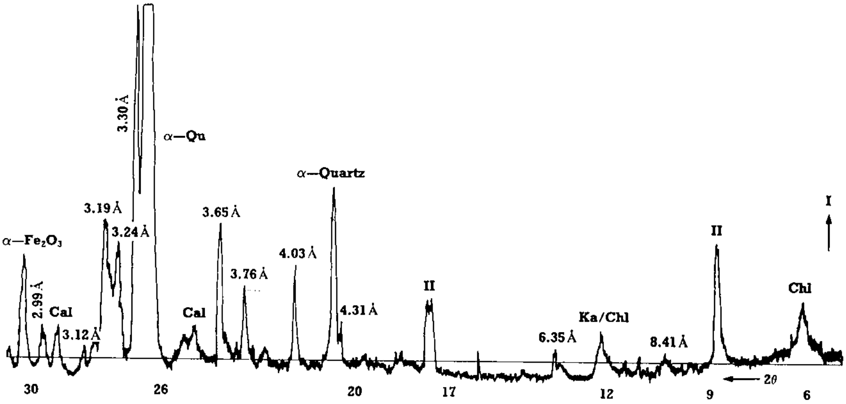

FIGURE 5 X-ray diffraction scan of an untreated sample, collected by a cleaner in a residential building near Ispra, Italy. (cal = calcite).

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