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CONSULTING ULTRAMICROANALYSIS • MICROSCOPY • SMALL PARTICLE PROBLEMS • SOLID-STATE CHEMISTRY

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Hearing Clerk
Food and Drug Administration
Room 6-86
5600 Fisheries Lane
Rockville, Maryland 20852

Gentlemen:

We are writing to comment on the Commissioner's proposal covering "Asbestos Particles in Food and Drugs" as published in the Federal Register, Vol. 38 No. 188, pages 27076-27081, 28 September 1973. We are interested in helping the FDA choose a practical analytical procedure for the detection and identification of asbestos.

Our laboratory has analyzed many hundreds of asbestos and asbestos-containing samples by most of the various analytical procedures: polarized light microscopy, x-ray diffraction, scanning electron microscopy with energy-dispersive x-ray analysis, transmission electron microscopy with selected area electron diffraction, electron microprobe analysis and electron microscope microprobe analysis. We would like to begin by giving you our evaluation of each of these methods.

The methods to consider are:
1. Morphology by light and electron microscopy
2. Optical properties by light microscopy
3. Diffraction by x-ray or electron diffraction
4. Elemental analysis by electron or ion microprobe

The various instruments available are capable of a variety of combinations of these methods (See Table 1 attached to this letter). The advantages and disadvantages of each area are discussed individually.

The polarizing light microscope (LM) requires great skill and background obtained only with long practice. A microscopist trained in optical crystallography and familiar with the serpentines and amphiboles still requires hours of time per sample to find and identify each of the possible asbestos types. The problem is complicated by the presence of interfering fibers (3:1 aspect ratio!), e.g., paper, talc on edge, diatoms, quartz, mineral wool, plant hairs and fibers etc. Finally, all of the asbestos types show considerable variation in all of their properties and these variations must be familiar to the microscopist.

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Dispersion staining (DS) greatly speeds up the detection and identification of asbestos fibers. It is the only practical way to apply optical crystallography to samples containing small percentages of asbestos. The technique can be learned much more rapidly than optical crystallography yet it is securely based on optical crystallographic properties.

X-ray diffraction (XRD), also based on crystal properties, is somewhat less sensitive than dispersion staining but requires no more background and skill. The sensitivity can be increased from 0.5-1.0% to 0.1-0.5% by step-scanning. One advantage over LM and DS is smaller size limit — about 50 nm compared with about 5,000 nm (5 μm).

Scanning electron microscopy with an energy dispersive x-ray analyzer is an overrated tool. Many substances besides asbestos are fibrous and it is nearly impossible to obtain a good chemical analysis of submicrometer particles in a particulate matrix with EDXRA in the SEM; there is too much interference. Fitting the SEM with a wavelength dispersive x-ray analyzer greatly improves it usefulness.

The transmission electron microscope (TEM) with selected area electron diffraction is an ideal tool when the asbestos fiber size is ≤ 0.2 μm. The shape of fibers down to 0.005 μm is immediately apparent on scanning a sample and the diffraction pattern is immediately discernible.

The electron microprobe analyzer (EMA) with its wavelength dispersive x-ray analyzer is better than the SEM fitted with EDXRA but is surpassed by an SEM fitted with wavelength dispersive x-ray analyzer. The better morphological image of the SEM tips the balance.

EMMA, the combination transmission electron microscope-electron microprobe analyzer is, by far, the most useful of the analytical tools for asbestos. It may not be needed if the fiber size is large enough (about 0.5 μm in smallest dimension), but smaller particles can be characterized by morphology, diffraction and chemical analysis in this one instrument. It is ideal for water samples containing suspended asbestos fibers.

The ion microprobe, although a highly sensitive trace analytical tool, is not usually used for asbestos fiber analysis. It does, however, detect fine fibers and identifies them by qualitative chemical analysis (quantitative when compared with standards).

Summarizing then — if the size of the asbestos fibers is very small (as it nearly always is for water samples) LM is useless and a diffraction method must be used. If the size is large enough, say 1 x 3 μm, and the percentage high enough, say $\leq 0.01\%$, then LM can do the job. If high sensitivity is not needed, say about 0.2% or greater, then XRD is the best method. Very fine particles require the TEM with selected area diffraction.

It should not be necessary to emphasize that no method is better than the analyst who makes the analysis. Few laboratories have all of the above instruments and highly skilled analysts for each. Instead, each tends to use the tools available and the methods with which their personnel are more or less familiar. In a situation as important as this, the choice should not be based on availability of tools and skills. Fortunately, the tools for detection and identification of asbestos are neither the most expensive nor the most sophisticated.