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Pfizer's position as a supplier of talc to many industries, caused us to seek a reliable method to identify and detect asbestos and asbestiform minerals possibly present in talc. Ideally we wanted the technique to be simple and direct but above all, it must be positive and unambiguous. Talc is a naturally occurring hydrous magnesium silicate and you have been given the formulas from various and sundry people this morning and this afternoon; and is derived from minerals containing various amounts of tremolite, chlorite, chrysotile and a group of chrysotile-like minerals called amphiboles or asbestiform minerals. Chief among these are anthophyllite, amosite, crocidolite and actinolite. From their chemical formulas, which you can find in the reference, you can see the close chemical similarity between talc and these asbestos type minerals. It becomes particularly difficult to analyze for these when one considers that the degree of replacement of one ion for another is seldom in exact proportion. Often the best that you can do is say that this particular material is, for example, like anthophyllite or most like anthophyllite. We investigated the currently available methods and methodology for detecting asbestos and tremolite and the asbestiform minerals in the presence of talc. The three most likely candidates that we came up with were light microscopy, X-ray diffraction and transmission electron microscopy. Samples of pure talc and tremolite were obtained from various deposits owned by Pfizer. Samples of very pure and very carefully characterized asbestos minerals were obtained

from the International Union Against Cancer in the United Kingdom. The talcs and asbestiform minerals were obtained in the pure, as received state, their characteristics noted and mixtures made to determine if the detection of asbestiform minerals was possible at low levels and, if so, what might the minimum detection levels be? Experimentally we started with X-ray diffraction. We obtained patterns for all of the minerals and mixtures used in the study employing the conventional X-ray techniques of scanning at rates of from 0.5 to 1 degrees 2 theta per minute. These samples were then subjected to scrutiny by optical and electron microscopy. When we did this we found that sometimes mixtures and mineral species shown to be free of asbestos minerals by the conventional X-ray diffraction techniques mentioned above and the light microscope also exhibited very large percentages of 5% or more of tremolite and/or asbestos minerals when viewed in the transmission electron microscope. We sought to find out the reason for this paradox which then enabled us to develop reliable techniques for detecting tremolite and the asbestiforms at 0.25% to 0.5% by X-ray diffraction. Even lower levels than these could be detected by electron microscopy.

The first table, Table I, shows the d spacing for talc, chlorite, tremolite and the asbestiform minerals. These are for pure minerals and can shift by as much as 0.2 to 0.3 angstroms depending on the sample preparation. You can see the problem that is going to present itself when you consider that you are really dealing with three specific items. You are dealing with talc, you are dealing with chrysotile

asbestos and then you are dealing with tremolite with a last difficulty thrown in -- chlorite. We'll get to the chlorite in a few minutes. What we did is use an ordinary step scanning diffractometer which moved the counter .05 degrees 2 theta and the intensity of the X radiation at each step was measured for approximately 2 to 4 minutes. We then plotted the intensity of this versus 2 theta over the area of interest - roughly 13 degrees to 9 degrees 2 theta shown in figure 1. This is the type of output we obtained when we examined talc from our Montana deposit. The general upward slope as you go lower in degrees is caused by the measuring device entering the primary beam of the X-ray tube. We established calibration curves by integrating the areas under these curves and the appropriate X-ray diffraction peak, samples with as little as 0.1% and as much as 10% additions of the species under investigation were examined. We extrapolated these curves to 0% and we got the minimum background with the minimum amount detectable using the standardized formula of 3 times the square root of background level at that point. For tremolite in talc, the minimum detectable level was found to be 0.25%. For chrysotile and other asbestiform minerals the minimum detectable level by this method is 0.5% but this can only be achieved in the absence of chlorite. Referring back to the first figure, the chlorite tends to mask the presence of chrysotile asbestos because it is so close to chrysotile asbestos in its major diffraction peak. The consequance of this is that when you have chrysotile asbestos or when you have chlorite, you can't be sure exactly which of the two you have. If you have a small amount of chlorite, it will very effectly

mask the presence of the chrysotile asbestos. One of the ways out of the dilemma that presented itself to us at this time was the fact that chlorite is acid soluble so we thought what we would do in this case was to acid wash the talc containing the chlorite, get rid of the chlorite and then examine it for chrysotile asbestos and in fact, the chrysotile asbestos peak and the chlorite peak both did disappear. The only problem was that when we looked at these samples in the electron microscope, the presence of the chrysotile fibers was still found. This is explained by the fact that chrysotile asbestos is in fact partially soluble in hydrochloric acid. It is a diffusion controlled reaction and as the magnesium ions depart from the crystal structure, you are left with a shell of silicon which is amorphous to the X-ray beam - the X-ray beam does not see it. The electron microscope does pick it out.

Figure 2 demonstrates the difference in X-ray intensity one gets over the same angular region when one adds 5% tremolite or 1% tremolite. The same type of plot is available when one adds chrysotile asbestos or any of the other asbestiform minerals. Figure 3 is the calibration plot that we obtained for chrysotile. Therefore, we can put a pretty good handle on the percent asbestos or tremolite or asbestiform mineral that we are going to find. Figure 4 is the calibration curve obtained for tremolite. So you can see that it is a fairly sensitive technique and is easily performed. Table 2 shows the selected area electron diffraction maxima for talc, tremolite, chrysotile, amosite, anthophyllite. We use these for the positive identification of these minerals. Transmission electron microscopy is employed in this

identification. We need to have this type of information, because we need to be unambiguous about our analysis. We want to know for sure what we are looking at. The electron microscope has the ability to single out individual particles and by virtue of selected area electron diffraction, we can derive the same type of data as an X-ray diffraction unit except we are quite a bit more sensitive here. By virtue of its ability to examine these individual fibers and particles at very high magnification, the transmission electron microscope has been found by us to provide the technique ancillary to X-ray diffraction that is needed to complete the unambiguous detection of the identification of asbestiform minerals. The morphology of the asbestiform minerals and tremolite is generally described as acicular or fibrous. This immediately serves to isolate it from the platy talc matrix even in the presence of chlorite since chlorite, fortunately closely resembles the morphology of talc. The chlorite is platy; the talc is platy so that eliminates immediately the problem of detecting fibers since the investigator disregards all platy particles and looks only for fibers. Now if a careful examination of approximately 100 different fields of view fails to reveal any fibrous material, then we feel that talc is free of tremolite, chrysotile and the asbestiform minerals. The problem of particle curling was mentioned earlier. One of the uses for selected area electron diffraction is to diffract from curled up plates. You can tell that the diffraction pattern is no different from that of the plate from which this material starts to curl. With the transmission electron microscope, you are

looking right into the particle, you see it and then you diffract from the pseudo fiber of curled up talc and you find that its pattern is identical to that of the main talc body. Therefore, there is no difference mineralogically between the two. The talc pseudo fiber can just as likely unroll as it can roll up. Now at first we had a little problem with our talcs because when we used the Montana material, we couldn't find any fibers, we couldn't find any chrysotile, we couldn't find any tremolite, we couldn't find anything but a small bit of chlorite. This prompted us to spike some samples and we did this by adding a weighed amount of chrysotile asbestos in one case for example to a weighed amount of our talc and we were able to construct calibration curves similar to the type that we used in the X-ray diffraction work and we found for example that with 1% addition of chrysotile asbestos to the Montana talc, we would find on the average in the 100 fields of view, 6 fibers with a standard deviation of approximately 1-1/2 fibers and it also followed a Guasian distribution which made us feel a good deal better about the homogeneity of the sample. These samples were prepared by slurrying the dry powders in a Waring blender with isoprophyl or ethyl alcohol for five minutes and then allowing them to dry and then just simply crumbling them up. We also used 0.1% and 0.5% asbestos and up to 5%. Figure 6 is a typical example of Montana talc. The micron marker is 1 micron and these are all platy materials. There have been no fibers identified in this material. Figure 5 shows a typical example when you spike the sample with 1% chrysotile asbestos. Fibers are readily observed. I call your attention to the thinness

of the fibers - I would certainly call them fibers. Figure 7 shows a sample of naturally occurring talc with a naturally occurring chrysotile asbestos contaminant, the chrysotile running at an approximate 45° angle. These fibers will become individualized. They are pretty much individualized right there and they are also very thin.

We also tried light microscopy since the optical microscope has for many years been used to identify mineral species and one of the things we turned to was dispersion staining since that has been successfully applied to the identification of asbestiform fibers. There has been a great deal of work done on it and asbestiform minerals are in fact readily distinguishable from one another. The difficulty in applying this to the problem at hand is that while it works well on pure samples of fairly massive fiber length from 3 to 5 microns, our observations by transmission electron microscopy have shown that naturally occurring asbestiform minerals often lie below the working resolution capabilities of the light microscope and furthermore while massive fiber bundles can often be observed by either light or electron microscopy the observation of individual fibers smaller than 0.5 by 0.2 micrometers often will require the high resolution capability of the transmission electron microscope. In addition, the limit of detection is confounded by the presence of talc fibers which are the thin talc plates curled up at the edge. Talc plates sitting on an edge also give difficulty. The lower limit of detection was felt to be too high to be of commercial value.

In conclusion, we feel that the properly prepared samples of talc can be step scanned by X-ray diffraction to detect tremolite down to a 1/4 of a percent, and an asbestiform mineral at the 1/2% level in the absence of chlorite. In the presence of chlorite and at concentration levels lower than those stated above the transmission electron microscope was found to provide reliable detection in identification of tremolite and the asbestiform minerals. The transmission electron microscope is the most sensitive method and appears to be a more or less referee technique since when morphology observations are coupled with selected area electron diffraction studies, there are no known interferences. We found light microscopy helpful only in scrutinizing samples with very large particles at very high concentrations of objectionable fibers.

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