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CONFIDENTIAL

17th October, 1968.

Examination of Synthetic Chrysotiles from Johns-Manville

Ref. Letter from Dr. Pundsack, dated 5th July, 1968.

Two samples, No. 3566-84-6 a "pure" synthetic chrysotile and 3566-98-1 a nickel-chrysotile, containing 2.6% Nickel were received for animal experiments by Dr. Davies. Small samples were retained in Chemical Research Dept. for examination. Our results are as follows.

T.G.A./D.T.A.

Both samples had a high absorbed water content (2%) and gave thermal analysis curves typical of highly-opened chrysotile (e.g. Coalinga or finely ground Cassiar fibre). The "pure" sample showed an unusual weight loss at about 800°C. (0.2%). This might correspond to 2% talc or 0.4% $CaCO_3$. The Nickel chrysotile showed a net weight gain in this region - possibly due to oxidation of nickel. The total weight loss, including absorbed water agreed with the J-M figures given in Dr. Pundsack's letter.

Infra-Red Spectroscopy

Both samples had unusual infra-red spectra in that although the form of the three main peaks closely resembled those of natural chrysotile, two of the peaks were significantly shifted (by 0.1 to 0.3 u) to lower wavelengths. The shift cannot be associated with substitution of nickel for magnesium since it occurs in both synthetic samples and their spectra were virtually identical. It may reflect a different degree of crystalline order. It was almost certainly not due to superposition of a natural chrysotile spectrum on that of an impurity (silica, $MgCO_3$ talc etc.).

X-Ray Diffraction

The powder patterns of the two synthetics were not detectably different from one another, but were significantly less well ordered than natural chrysotile. This may be an effect of crystallinity or fine particle size, but Coalinga fibre is much better ordered.

We don't agree with the J-M comment that they have "excellent chrysotile diffraction patterns" except in the sense that J-M have obviously not synthesised talc or forsterite. From X-rays the material is obviously a synthetic serpentine and very probably chrysotile.

PLAINTIFF'S EXHIBIT
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Electron Microscopy

The E-M photos and diffraction patterns (attached) are very interesting. A rough estimate indicates only about 1% of the weight of the "pure" chrysotile is of fibrils 1 - 5 μ long and virtually none above this length. The nickel chrysotile contains even fewer "long" fibrils. In this sense the statement in the J-M letter that the maximum fibre lengths are 3 - 5 μ, though literally correct is perhaps misleading. In both samples the majority of fibrils are less than 0.2 μ long (2000A). It appears that some of them are incompletely formed cylinders, even ribbons. One fibril showed the apparent "tube in tube" effect previously seen in published micrographs of synthetic chrysotile, but this one fibril is not typical of the majority. It seems likely that these may not be "tubes in tubes", but growth steps. The long fibrils gave good chrysotile electron diffraction patterns, but the mass of short fibrils gave a poorly defined polycrystalline pattern.

Summary

Both samples were powdersand not visibly fibrous. The "pure" chrysotile was white and the nickel chrysotile green. The J-M chemical analysis, which we have not checked, shows that both contain many impurity elements, some in quite large amounts (0.35% Na_2O in both and 0.3% CaO in the "pure" sample).

Both samples have very low aspect ratios (mainly about 2 : 1 to 10 : 1) and probably have very high surface areas (B.E.T. measurements will be made to check this). Whilst no other crystalline material than "chrysotile" was detected there may be a significant amount of amorphous or poorly crystalline magnesium silicate present.

No significant structural differences were detected between the pure and the nickel containing material and in the absence of any direct evidence (except the colour difference) it can not be assumed that all the nickel is in the chrysotile lattice.

From the point of view of health studies, any difference in behaviour between the two samples may be attributable to nickel, but comparisons of either sample with a natural chrysotile should take into account the fact that the synthetics are very much shorter, less well defined crystals, probably [illegible] and probably of much higher surface area.

If cell reaction is a surface effect, as seems reasonable, they would behave differently and probably be much more effective than natural chrysotile - even when this is finely ground as in U.I.C.C. samples. For example I would expect the synthetics to be more

readily soluble and more highly absorptive than natural chrysotile, and they may contain significant amounts of free silica, MgO, $Mg(OH)_2$ etc. They also have many more "fibre ends" per unit weight than normal chrysotile.

yes!!

they may!!
If so, they should be less reactive than finely ground natural chrysotile

G. F. Heron.