

Differential Thermal Analysis of Chrysotile Asbestos in Pure Talc and Talc Containing Other Minerals

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Synopsis—Levels of 0.5 to 1.0% CHRYSOTILE ASBESTOS in TALCS can be detected by DIFFERENTIAL THERMAL ANALYSIS (DTA). The 845°C exothermic DTA peak is used for the detection of CHRYSOTILE. Both naturally occurring serpentine† and artificially added CHRYSOTILE ASBESTOS can be detected in PURE TALC and in talcs containing other minerals.

INTRODUCTION

Cosmetic and body talc products have shown a remarkable growth in sales over the past several years. Recently, asbestos forms (namely, chrysotile and tremolite) were reported to be present in cosmetic and body talc products. Asbestos fibers have also been detected and analyzed in beverages and drinking water (1).

Cosmetic talc can be described as a mixture of naturally occurring minerals, where the major constituent is a magnesium silicate and some of the minor constituents are chlorite and carbonates. The performance of a cosmetic talc can be effected by the presence of minor mineral constituents in the talc. The effect can be either detrimental or beneficial. For example, chlorite is thought to be beneficial for perfume retention. Because of the possible inhalation hazard associated with asbestos, the development of a sensitive analytical method for its detection in cosmetic talc was necessary.

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†Serpentine is a family of asbestos consisting primarily of antigorite (nonfibrous) and chrysotile (fibrous).

The analysis of carbonates, chlorite, and other minerals in talcs is described by Pask and Warner (2). We also developed a differential thermal analysis (DTA) method for these minerals in 1972 (3).

Analytical methods reported in the literature that are applicable to mineral characterization include: X-ray diffraction; optical and electron microscopy; atomic absorption (AA); infrared (IR) spectroscopy; DTA; and thermogravimetric analysis (TGA).

X-ray diffraction has been widely used for identification of minerals, but without repetitive scanning, it is not sufficiently sensitive to yield reproducible results for constituents much below the 1% level. The various optical and electron microscopic techniques are considered tedious and time consuming, but are used as confirmation techniques in combination with X-ray diffraction. The AA method is a sensitive method for detection of many elements, but the presence of similar atoms in the minerals comprising the talc limits its effectiveness for determining asbestos. IR spectroscopy is widely used in mineral characterization, both qualitatively and quantitatively, because it is specific and reproducible, possessing a unique finger printing capability. DTA and TGA, due to their ability to provide information on the crystallographic changes occurring in heating, are also widely used for mineral characterization (2, 3).

The DTA method for both qualitative and quantitative mineral characterization is extensively described in the literature. Although the DTA method for the detection of asbestos at the 1% level was developed by Avon Products, Inc., over 2 years ago (3), a similar method has been recently reported (4). Our method offers several advantages: it is three times faster and requires no helium atmosphere. It has been found to be specific, reproducible, and requires no involved sample preparation. Therefore, it can be readily used for quality control purposes.

EXPERIMENTAL

The mineral composition of a large number of cosmetic grade talcs from the United States and other countries was determined by X-ray diffraction (5). Samples of the major talc constituents were obtained and were also analyzed by X-ray diffraction. The minerals sometimes found in cosmetic talcs are given in Table I (5). One or more of these minerals may be totally absent in cosmetic talcs.

The duPont 900 DTA equipped with the 1200°C high temperature cell was used for the analysis of chrysotile asbestos in talcs. Chrysotile asbestos from Seitz at 1.0 and 3.0% level was used in the talc blends.

The samples (38 to 40 mg) were heated in platinum cups at 30°C/min in a static air atmosphere using Al_2O_3 as a reference material. The temperature scale T was set at 0.8 MV/in., and the sensitivity of the DTA instru-

Table I

| | |
|-----------|------------------------------|
| Chlorite | $Mg_{12}Si_5O_{20}(OH)_{16}$ |
| Calcite | $CaCO_3$ |
| Dolomite | $CaMg(CO_3)_2$ |
| Magnesite | $MgCO_3$ |
| Tremolite | $Ca_2Mg_5Si_8O_{22}(OH)_2$ |

ment was set at the maximum (ΔT 0.004 MV/in.). Upon heating the samples at 30°C/min to 4.0 mVt (457°C), the recorder pen was shifted to -5 in. using the T-scale zero shift.

DISCUSSION AND RESULTS

The success or failure, with regard to determining low levels of chrysotile asbestos by DTA in complex talc products and talc itself, depends on obtaining the proper conditions, such as heating rate, atmosphere, particle and sample size, and particularly a good base line at maximum sensitivity.

In addition to these conditions, the magnitudes and positions of the endothermic and exothermic DTA peaks of other minerals present in talc could have an effect on the final success of the detection of chrysotile.

The magnitudes and positions of the DTA peaks of various minerals sometimes found in talcs, are shown in Figs. 1 and 2 and are listed in Table II. DTA data of some of the above minerals in talc are also presented in Pask and Warner (2).

As can be seen from the chrysotile thermogram, Fig. 1 (A), the large endothermic peak due to dehydroxylation at 673°C and the large exothermic peak due to dehydroxylation and crystallization at 845°C, make this mineral readily detectable in talc at low levels. The thermogram of chlorite is shown in Fig. 1 (B).

The large decarboxylation, dehydroxylation, and crystallization DTA peaks of carbonates and chlorite, if these minerals are present in quantities above 10 to 15%, interfere with the detection of low levels of chrysotile. In most cases, however, the mineral contaminants are found to be well below this figure. The thermogram of talc is shown in Fig. 2 (B). The small tremolite dehydroxylation peak at 1075°C shown in Fig. 2 (A) makes its detection difficult at these levels.

The DTA thermogram of a talc product containing 3.0% kaolin, a trace of chlorite, and 8.0% calcium carbonate is shown in Fig. 3 (A). The endotherms at 530°, 600°, 790°, and 970° are due to kaolin, chlorite, calcium carbonate, and talc, respectively.

This same talc product but with the addition of 1.0% chrysotile asbestos, is shown in Fig. 3 (B). The appearance of a small endotherm at 660°C and a medium exotherm at 845°C, clearly show the presence of chrysotile asbes-

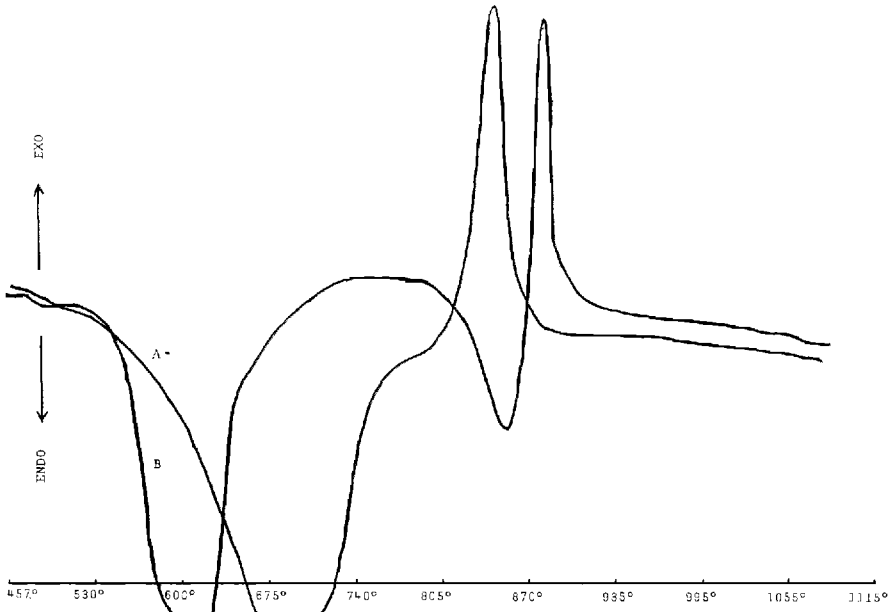


Figure 1. Sample: (A) chrysotile—12.8 mg; (B) chlorite—10.2 mg

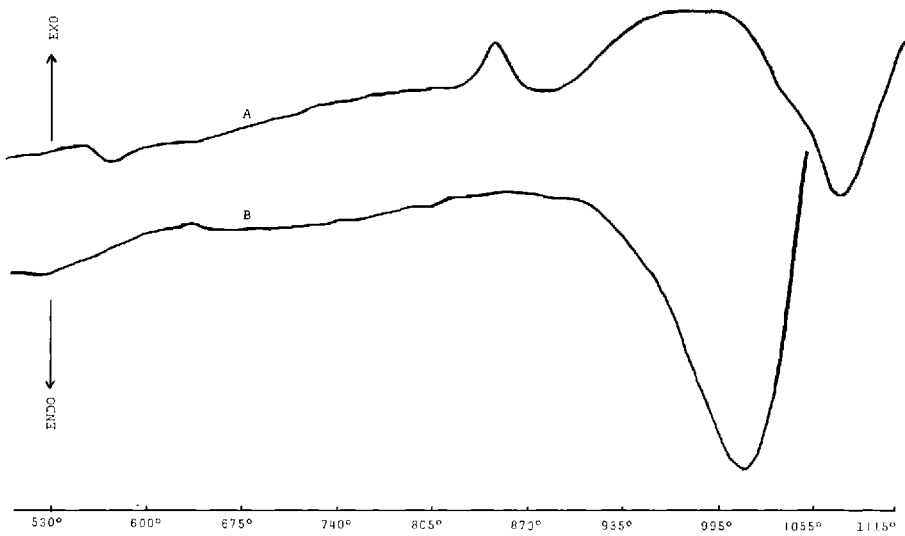


Figure 2. Sample: (A) tremolite—32.5 mg; (B) talc—30.0 mg

Table II
Differential Thermal Analysis of Talc Minerals

| Mineral | Chemical Composition | Endotherms, °C | | | Exotherms, °C | | |
|---------------|---|-------------------|--------|------|---------------|--------|------|
| | | Strong | Medium | Weak | Strong | Medium | Weak |
| Tremolite | $\text{Ca}_2\text{Mg}_5\text{Si}_8\text{O}_{22}(\text{OH})_2$ | 1075 | ... | ... | ... | ... | 845 |
| Talc—97% pure | $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$ | 1010 ^a | ... | ... | ... | ... | ... |
| Calcite | CaCO_3 | 790 | ... | ... | ... | ... | ... |
| Chrysotile | $\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4$ | 673 | ... | ... | 845 | ... | ... |
| Chlorite | $\text{Mg}_{12}\text{Si}_5\text{O}_{20}(\text{OH})_{16}$ | 600 | 860 | ... | 885 | ... | ... |
| Kaolinite | $\text{Al}_2\text{Si}_2(\text{OH})_4$ | 530 | ... | ... | 1055 | ... | ... |

^aTalcs containing other minerals, such as chlorite, carbonates, etc., will show dehydroxylation peaks at lower temperatures.

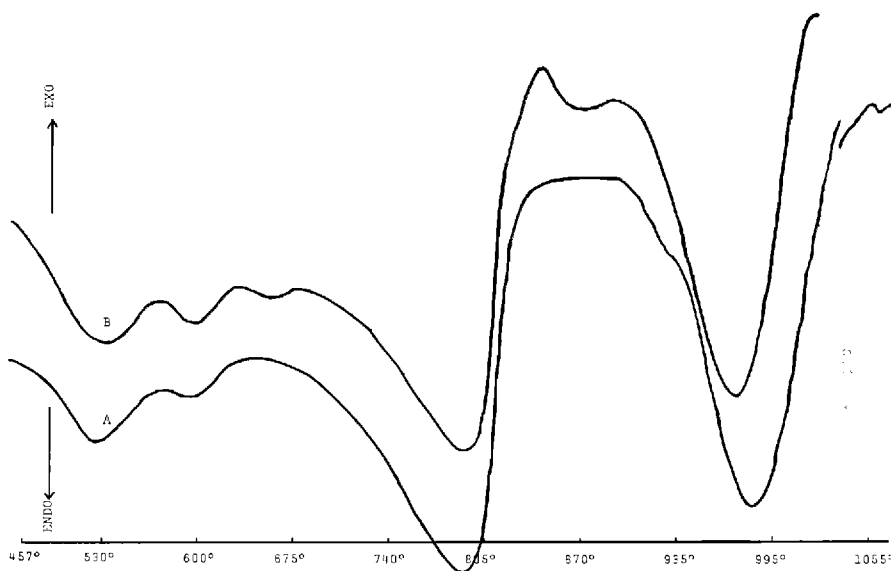


Figure 3. Sample: (A) talc product—40.0 mg; (B) talc product plus one per cent chrysotile

tos. Chrysotile is therefore not affected by the presence of kaolin, chlorite, and calcium carbonate at the above-mentioned levels.

Another talc product containing approximately 5.0% chlorite was analyzed and revealed no chrysotile asbestos. Upon addition of 1.0% chrysotile asbestos, the exothermic peak at 845°C was clearly visible.

Talc containing 10 to 15% tremolite as determined by X-ray diffraction is shown in Fig. 4 (A). Note that even at the 10 to 15% level, the endotherm

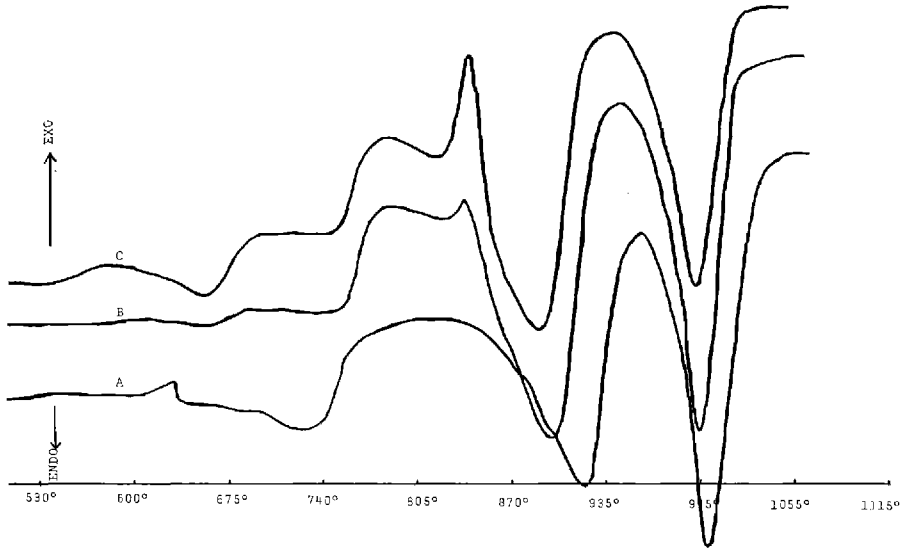


Figure 4. Sample: (A) talc plus tremolite—39.0 mg; (B) talc plus one per cent chrysotile; (C) talc plus three per cent chrysotile

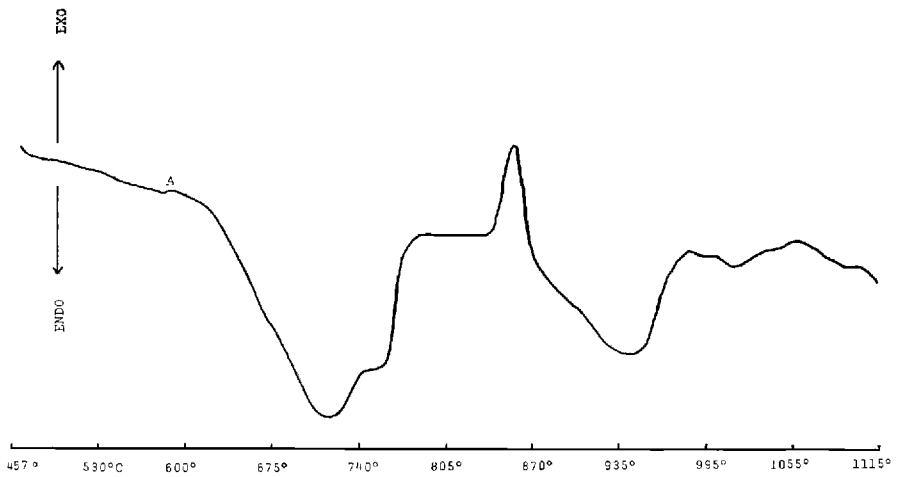


Figure 5. Sample: (A) naturally occurring serpentine in talc—10.5 mg

for tremolite will not appear; this was determined in previous DTA runs on the same talc, though this particular thermogram does not show it. The same talc containing 1.0 and 3.0% added chrysotile asbestos is shown in Fig. 4 (B) and (C). The difference between 1 and 3 per cent chrysotile asbestos in talc is clearly distinguishable.

A thermogram of naturally occurring serpentine in talc is shown in Fig. 5 (A), subsequently found by TEM to be antigorite.

CONCLUSION

From the preceding analysis, it is evident that 0.5 to 1.0% serpentine asbestos in talcs can be easily detected. Serpentine at low levels can also be detected in talcs in the presence of other minerals such as chlorite and carbonates. If an asbestos level of 1% in cosmetic talcs should be the limit, this technique can be used as a screening method. In fact, we are presently employing this technique as a screening method and have already checked approximately 200 samples. Of these 200 samples, none were found to contain chrysotile asbestos at the detectable level.

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