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A STUDY OF THE MINERALS

STURTITE and PIMELITE

by

Mithat Kayaalp

(Higher Licence in Geological Engineering,  
K.T.Ü., Turkey).

A thesis submitted for the  
Master of Science degree  
at the University of Durham

Graduate Society

March 1978

## ABSTRACT

This thesis comprises a mineralogical study of two minerals:

(a) Sturtite

(b) Pimelite

Both minerals were supplied from the collections of the Department of Geological Sciences of Durham University.

Although they have already been described by several authors there are still some doubts and major contradictions between them. The present study is an attempt to clarify those problems. These two minerals were also in the Joint Committee on Powder Diffraction Standards (JCPDS) 1977 list of minerals for which powder diffraction data was required.

The present study consists of mainly X-ray Diffraction Techniques and Thermal Techniques (Differential Thermal Analysis and Thermogravimetric Analysis). Also during the study Electron Microscope Techniques were used and Electron Microprobe Analysis obtained for both minerals.

## ACKNOWLEDGEMENTS

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A - X-RAY DIFFRACTION STUDIES

## X-RAY DIFFRACTION STUDIES

## 1. Powder Photograph Studies

The powder photographs were taken using a 114.59 mm Philips Debye-Scherrer camera. The method used was the Straumanis film mounting technique as described by Azaroff and Buerger (1958). The specimens were prepared in the following manner:

The specimen was first ground with an agate pestle and mortar and then mounted around a glass fibre (held in a brass holder) using flexible collodion as the mounting adhesive. The brass holder was mounted in a camera and then centered, by means of an alignment device, and the specimen X-rayed.

During the experiments the following instruments were used:

Philips PW 1130 3kV generator-diffractometer using iron filtered Co radiation at 50kV-30mA.

Philips PW 1010 1 kV generator-diffractometer using vanadium filtered Cr radiation at 30kV-10mA.

Philips PW 1320-1310 2kV generator-diffractometer using nickel filtered Cu radiation at 45kV-25mA.

The instrumental conditions used can be seen in Table 1.

Three different tubes Cr, Cu and Co were used in



order to minimise the problems of fluorescence in the sample. Pimelite contains large quantities of nickel and the values of mass absorption coefficient for Cr, Co and Cu radiations are 142.3, 72.5 and 48.3 (McKinley <sup>et al.</sup> 1966). respectively showing that for pimelite copper radiation is the best, but for sturtite, which contains large amounts of iron and manganese, chromium radiation was required.

X-ray diffraction line measurements were carried out using a Hilger and Watts film measuring scale fitted with a vernier device capable of reading the lines to 0.05 mm.

A computer programme (DPOW) was used to obtain the d-spacings, this was based on Cohen's least squares method which was originally written by M. George (1969), and later modified by Allan Hall, of this department (Akinici, 1971). The d-spacings obtained from this programme have been corrected for film shrinkage, however, in certain samples no back reflection data was obtained, therefore, in this case the programme was modified by introducing theoretical data into the calculations.

Two crystallographic computer programmes were also used. They were GENSTRUK and COHEN (Marples <sup>Shaw</sup> 1966), but unfortunately they proved unsuccessful.

## 2. Powder Diffractometer Studies

As well as powder photography, powder diffractometer studies were undertaken on the same instruments.

The methods followed were those described by Klug and Alexander (1962) and Taylor (1971) in which the sample was cavity-mounted and in which the powdered sample was mounted on a glass slide using acetone as the mounting medium.

The specimens were then subjected to X-ray analysis in a Philips high angle PW 1050/25 goniometer attached to all three machines and the preliminary runs taken to obtain the optimum conditions of scan speed, pulse height analysis and slit widths. These settings are shown in Table 1.

X-ray diffraction  $2\theta$  scans were made between the range  $3^\circ$  to  $100^\circ$  and the diffraction peaks obtained were converted to d-spacings by means of conversion tables. The sample Pimelite was also X-rayed under special conditions, e.g. glycolation, and an accurate measurement made of the 060 reflection, to determine the dioctahedral or trioctahedral nature of the crystal structure.

TABLE 1

Instrumental Conditions for X-ray Powder Photograph and Diffraction Studies

|               |                       |                       |   |
|---------------|-----------------------|-----------------------|---|
| Radiation     | Cr, Co, Cu K-Alpha    | Radiation             | Co K-Alpha                                    |
| Collimator    | Fine, Clay Collimator | Slits                 | 2°, 0.2°, 2°                                  |
| Camera        | 114.59 mm, Philips    | Rate of Scan          | 1° 2θ/min                                     |
| Exposure time | Varied 5-72 hrs       | Counter               | Sealed proportional and pulse height analysis |
| Film          | Ilford, Ind. G-Fast   | Mount Type            | Smear and cavity                              |
| Film Mounting | Straumanis Technique  | Operation             | Ratemeter                                     |
|               |                       | Time Constant         | 4   |
|               |                       | Multiplication Factor | 2 x 10 <sup>2</sup> cps                       |

**B - ELECTRON MICROPROBE ANALYSIS**

## ELECTRON MICROPROBE ANALYSIS

Due to the variety of chemical formulae for pimelite, it was decided to analyse both samples on the electron microprobe to determine their exact compositions.

A Cambridge Scientific Instruments Geoscan, with a take-off angle of  $75^{\circ}$  was employed during the experiment. X-ray excitation was achieved by a beam of 15KeV electrons, normal to the surface, and focused to a minimum spot size about one micron in diameter. Fixed time counting was used throughout the analysis. A beam current of about 62.5  $\mu$ A yielded an effective specimen current of approximately 0.04  $\mu$ A.

Specimens were prepared as polished samples and coated with carbon under the same conditions as the standards.

Analytical runs were begun by counting on each of seven standards. Three peak and two background readings were taken before and after counting on the unknowns. Standard readings were repeated after every eight or nine peak and background readings on the unknown, to check the extent of drift, and provide a basis for its correction.

Details of the operating conditions used are shown in Table 2.

**Corrections:**

Low counts were first corrected for a 4 micro-seconds dead time and drift, with the aid of the on-line Varian 620-100 computer, by the program "TIM-3" written by Dr. A. Peckett. This is an assembler version of the Fortran IV TIM 1 of Duncomb and Jones (1970). The program corrects for mass absorption, secondary fluorescence and the atomic number effect (stopping power and electron back-scatter) in the manner described by Sweatman and Long (1969) for K, L and M lines.

TABLE 2

Operating Conditions and Standards used for Electron Microprobe Analysis

| <u>Element</u> | <u>Line</u>  | <u>Analysing<br/>Crystal</u> | <u>Counter</u> | <u>2<math>\theta</math> (Peak)</u> | <u>2<math>\theta</math><br/>(Background)</u> | <u>Standard</u>                |
|----------------|--------------|------------------------------|----------------|------------------------------------|--|--------------------------------|
| Si             | K $\alpha_1$ | KAP                          | Flow           | 31 $^{\circ}$ 02'                  | -1 $^{\circ}$ +1 $^{\circ}$ 30'              | Wollastonite                   |
| Al             | "            | "                            | "              | 36 $^{\circ}$ 32'                  | +2   | Al <sub>2</sub> O <sub>3</sub> |
| Mg             | "            | "                            | "              | 43 $^{\circ}$ 42'                  | -2   | MgO                            |
| Mn             | "            | LiF                          | "              | 62 $^{\circ}$ 48'                  | +2   | Mn Metal                       |
| Ca             | "            | "                            | "              | 113 $^{\circ}$ 02'                 | -2   | Wollastonite                   |
| Ni             | "            | "                            | "              | 48 $^{\circ}$ 34'                  | +2   | Ni Metal                       |
| Fe             | "            | "                            | "              | 57 $^{\circ}$ 20'                  | +2   | Fe Metal                       |

C - DIFFERENTIAL THERMAL ANALYSIS (DTA)



## DIFFERENTIAL THERMAL ANALYSIS (DTA)

DTA has proved especially useful in the determination of the clay minerals, because clay minerals are not easily identified by classical methods due to their fine grain size and similarity in chemical composition.

The method used was that described by Mackenzie (1957).

The specimen under examination was heated up at a uniform rate of  $10^{\circ}\text{C}/\text{min.}$  side-by-side with a sample of pure dry alumina powder (which is thermally inert) in matching platinum crucibles. Any differences in temperature between the two, caused by reactions in the specimen, are recorded on a chart recorder as either an endothermic peak or an exothermic peak depending on whether heat is either absorbed or evolved.

During the experiments a Stanton Equipment Standata 5-50" model in conjunction with a West Controller were employed.

D - THERMOGRAVIMETRIC ANALYSIS

## THERMOGRAVIMETRIC ANALYSIS (TGA)

Powdered samples of both sturtite and pimelite were submitted to TGA studies using a Stanton-Automatic Direct Reading - Thermobalance.

In this technique the loss of weight of the sample is measured as the sample is gradually heated up at a constant rate.

Due to the disagreements between analysis and formulae given in the literature, and also the low analysis totals obtained from Electron Microprobe studies, an attempt was made, using the TGA, to obtain water loss and dehydration curves for both minerals.

To obtain the water loss the following formula was used:

$$\frac{\text{Scale loss (in milligrams)}}{\text{Weight of sample}} \times 100 = \% \text{ Water}$$

I. STURTITE



Plate 1. A photomicrograph of Sturtite (reddish-brown). Carbonate minerals in light-coloured areas. X nicols, transmitted light, 100x.

STURTITE  
Sample No: 6624 Broken Hill, NSW,  
Australia

This name was given in honour of the explorer Captain Charles Sturt who was the first white man to have visited Broken Hill on the 22nd October 1844.

The mineral was first described by Hodge-Smith (1930) who concluded that it was an amorphous mineral; unfortunately this is the only information available.

a. Physical Characters

Sturtite is a very brittle and friable mineral, easily broken up by the fingers. The hardness is slightly over 3; the colour is jet black and the streak is yellowish-brown.

b. Microscopic Observation

It appears under the microscope to be colourless to pale brown and has the appearance of an isotropic (Plate 1) substance. / Very slight anisotropy and bireflectance, together with reddish internal reflections may be attributed to impurities or alteration (iron hydroxides). Under both reflected and transmitted light microscopic examination reveals discrete grains of sturtite of pseudo-hexagonal outline.

The reflectivity of the mineral is very low and may be approximately 7%.

The examined sections show that galena, sphalerite and some chalcopyrite droplets together with major amounts

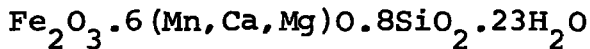
of carbonate (calcite) minerals are associated with sturtite. Some quartz, probably replaces the minerals along the cracks.

Both sturtite and associated minerals are deformed in the same manner, they are partially cracked and stressed. This possibly indicates that deformation took place after the formation of the associated minerals and the sturtite. Subsequently ironhydroxides (goethite) replaced sturtite starting from the cracks or pseudograin boundaries which are thought to be prior to alteration or deformation (metamorphism?).

According to Hodge-Smith (1930) quartz, spessartite and rhodochrosite are also associated minerals. But the specimen examined showed no trace of these minerals.

### c. Chemistry

Hodge-Smith (1930) gives the formula:



His chemical analysis of sturtite is shown below, together with the analysis obtained in the present study.

|                                | A<br><u>wt.%</u> | B<br><u>wt.%</u> |
|--------------------------------|------------------|------------------|
| SiO <sub>2</sub> (Free)        | 0.79             |                  |
| SiO <sub>2</sub> (Combined)    | 32.35            | 36.41            |
| Fe <sub>2</sub> O <sub>3</sub> | 10.22            | 13.53            |
| Al <sub>2</sub> O <sub>3</sub> | 0.44             | -                |
| MnO                            | 25.18            | 20.52            |
| CaO                            | 2.19             | 2.81             |
| MgO                            | 0.65             | 0.98             |
| H <sub>2</sub> O               | 28.16            | 13.00            |
|                                | <hr/>            | <hr/>            |
| Total                          | 99.98            | 87.25            |

A - Hodge-Smith; B - present study.

The water loss obtained from TGA gives 13 H<sub>2</sub>O % (see fig.1) which is insufficient to take Electron Microprobe Analysis to 100 percent. The total weight percent of sturtite obtained from the microprobe was 74.25% and the addition of 13 percent H<sub>2</sub>O only increased the total weight percent to 87.25%.

d. X-ray Study of Sturtite

During the study several X-ray powder photographs and diffraction charts were taken on carefully picked specimens using varying exposure times and conditions.

As a result, it was found that Hodge-Smith's (1930) conclusion was correct, since some specimens gave no diffraction lines and all the lines found in other specimens could be accounted for as impurities.

The amorphous nature was later confirmed by using the Electron Microscope (JEM 120 Transmission El.Mic) which gave no diffraction pattern. However, it was observed during the electron microscope experiment that diffraction patterns were produced after some time, presumably due to the heating effect of the electron beam and water loss under vacuum conditions causing crystallisation. A number of different patterns were obtained from the ~~pimelite~~ <sup>sturtite</sup> at these elevated temperatures, but unfortunately none of them corresponded with any minerals from the JCPDS index (1977).



d-spacings obtained from hand specimen material  
containing sturtite:

| <u>2 <math>\theta</math> (Degrees)</u> | <u>d (<math>\text{\AA}</math>)</u> | <u>Rel.Int.</u> | <u>Corresponding<br/>Impurities</u> |
|--|------------------------------------|-----------------|-------------------------------------|
| 39.226                                 | 3.41                               | 40              | Galena                              |
| 42.997                                 | 3.12                               | 20              | Sphalerite                          |
| 44.994                                 | 2.99                               | 100             | Tetrahedrite                        |
| 45.618                                 | 2.96                               | 30              | Galena?                             |
| 55.306                                 | 2.47                               | 5               | Calcite                             |
| 60.949                                 | 2.26                               | 10              | Calcite                             |
| 66.393                                 | 2.09                               | 60              | Galena                              |
| 67.067                                 | 2.07                               | 10              | Tetrahedrite                        |
| 73.284                                 | 1.92                               | 10              | Calcite                             |
| 74.832                                 | 1.88                               | 10              | Calcite                             |
| 76.330                                 | 1.85                               | 20              | Tetrahedrite                        |
| 79.776                                 | 1.78                               | 20              | Galena                              |
| 84.046                                 | 1.71                               | 5               | Galena                              |
| 119.525                                | 1.32                               | 5               | Galena                              |

Radiation = CrK $\alpha$  28/10  
Exp. Time = 72 hrs  
Coll. = Fine

After the above X-ray data of compound sturtite was taken, an attempt was made to obtain X-ray data from individual materials. The following results were obtained from the individual specimens which were carefully picked under the microscope.

X-ray data from area of high metallic lustre;

| <u>2<math>\theta</math></u> | <u>d (Å)</u> | <u>Rel.Int</u> |
|-----------------------------|--------------|----------------|
| 26.025                      | 3.42         | 80             |
| 30.025                      | 2.97         | 100            |
| 43.125                      | 2.09         | 50             |
| 51.125                      | 1.78         | 40             |
| 53.425                      | 1.71         | 20             |
| 62.55                       | 1.48         | 10             |
| 69.85                       | 1.34         | 5              |
| 71.00                       | 1.32         | 5              |
| 78.95                       | 1.21         | 5              |
| 84.875                      | 1.14         | 5              |

All these lines correspond with Galena.

Conditions

Rad = CuK $\alpha$   
 Exp. Time = 10 hrs  
 Coll = Fine  
 Rating = 40/20

The sample from an area of dull or resinous lustre gives a pattern which fits calcite and sphalerite.

| <u>2<math>\theta</math></u> | <u>d (Å)</u> |                |
|-----------------------------|--------------|----------------|
| 32.20                       | 3.13         | 100 Sphalerite |
| 34.35                       | 3.03         | 5 Calcite      |
| 42.00                       | 2.49         | 5 "            |
| 46.00                       | 2.29         | 5 "            |
| 50.60                       | 2.09         | 5 "            |
| 55.45                       | 1.92         | 25 "           |
| 56.90                       | 1.88         | 5 "            |
| 65.975                      | 1.64         | 5 "            |

Radiation = Co K 45/25  
 Exp. Time = 16 hrs  
 Coll. = Fine

Specimen from the brownish area (under the microscope)  
gives the following d-spacings.

| <u>2<math>\theta</math></u> | <u>d (Å)</u> | <u>Rel.Int</u> |              |
|-----------------------------|--------------|----------------|--------------|
| 29.775                      | 3.00         | 100            | Tetrahedrite |
| 36.325                      | 2.47         | 5              | Calcite      |
| 39.80                       | 2.26         | 10             | "            |
| 43.65                       | 2.07         | 10             | Tetrahedrite |
| 48.10                       | 1.89         | 20             | Calcite      |
| 49.10                       | 1.85         | 20             | Tetrahedrite |
| 57.95                       | 1.59         | 30             | "            |
| 61.327                      | 1.51         | 20             | "            |

e. T.G.A. Analysis of Sturtite

The total water loss obtained from thermogravimetric analysis gave 13% water (Fig.1). The first water loss occurs between the temperatures of 50<sup>o</sup>-250<sup>o</sup>C (10.5%) and second loss occurs between the temperatures of 250-450<sup>o</sup>C.

f. Conclusions

Carefully hand picked sturtite gave no X-ray or electron diffraction pattern and is therefore amorphous. The chemical composition of the material studied compares closely with that for the original sturtite except in the water content. The amount required to bring the total on the present microprobe analysis to 100% would give a water figure closely similar to the original analysis.

The maximum temperature attained in the T.G.A. analysis was 500<sup>o</sup>C and it is known that some minerals can retain water to much higher temperatures. Unfortunately the significance of this fact was realised when no further time was available for experimental work.

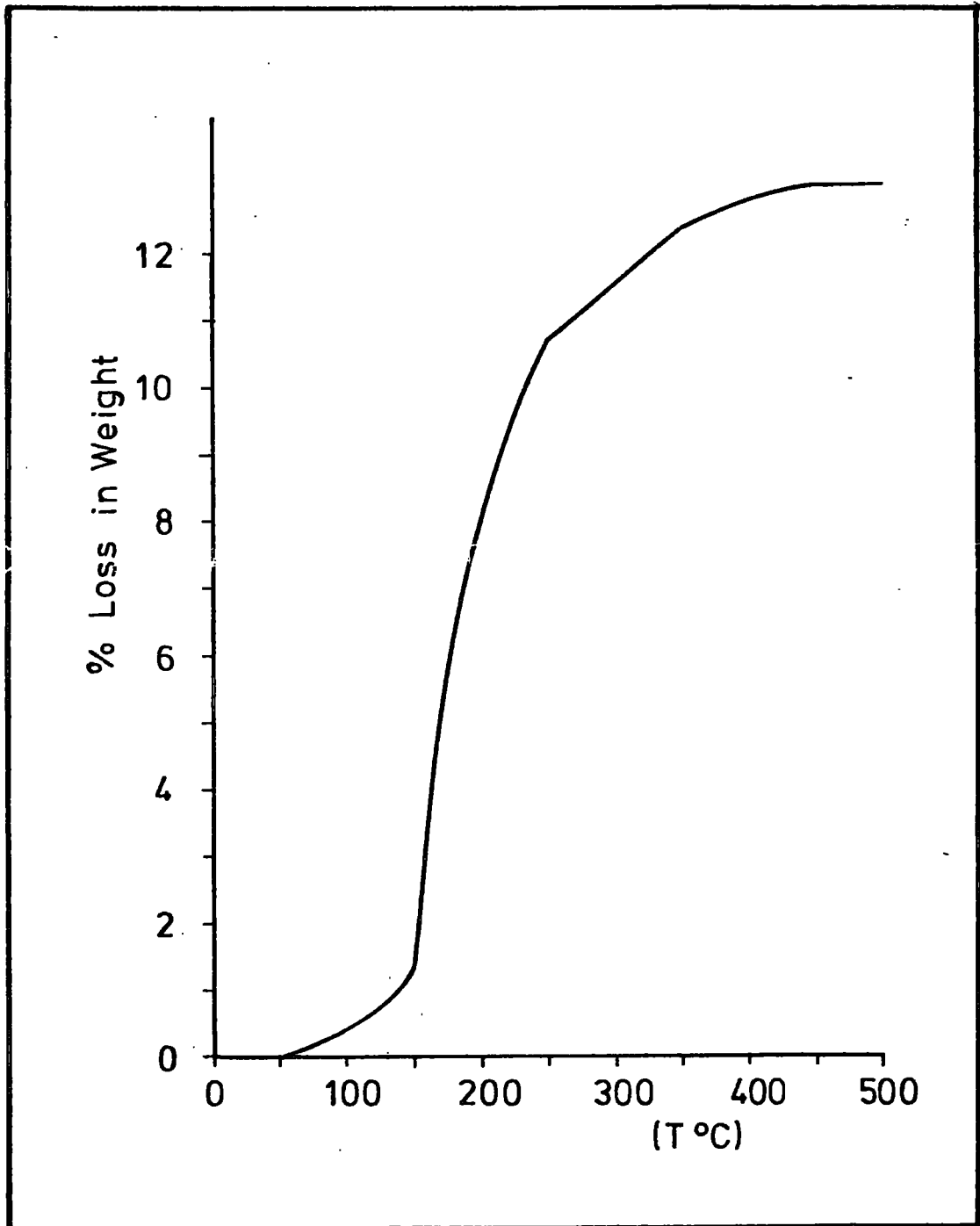


Figure 1. Dehydration Curve for Sturtite

## II. PIMELITE

## PIMELITE

## a. Literature Survey

This mineral was first described by Professor Martin H. Klaproth in 1788 as "Gruner Chrysopraserde" (Faust, 1966), one of the members of the "hydrous nickel-magnesium silicates".

According to Dana (1920, 6th Edn.) the name pimelite comes from the Greek, fatness. Dana states that pimelite minerals consist of two members; hard pimelite (presumably the pimelite itself in the present study) and pimelite Schmidt (Alipite) which are very similar to each other in many respects. The only distinction between the two appears to be the fact that alipite adheres to the tongue and hard pimelite does not. Specimen 9032, in the present study strongly adheres to the tongue which suggests that this may well be alipite rather than hard pimelite. However, no other reference to alipite in literature has been found.

Dana also states that pimelite is a massive mineral, apple green in colour and has a greenish-white streak. The hardness is 2.5 and it has a greasy feel.

Dana gives the following analysis for both pimelites:

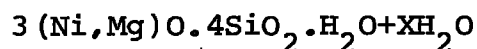
|                                | <u>Hard Pimelite</u> | <u>Pimelite<br/>Schmidt (Alipite)</u> |
|--------------------------------|----------------------|---------------------------------------|
| SiO <sub>2</sub>               | 35.80                | 54.63                                 |
| Al <sub>2</sub> O <sub>3</sub> | 23.04                | 0.30                                  |
| Fe <sub>2</sub> O <sub>3</sub> | 2.69                 | (FeO) 1.13                            |
| NiO                            | 2.78                 | 32.66                                 |
| MgO                            | 14.66                | 5.89                                  |
| CaO                            | -                    | 0.16                                  |
| H <sub>2</sub> O               | 21.03                | 5.23                                  |
|                                | <hr/>                | <hr/>                                 |
| Total                          | 100.00               | 100.00                                |

Cailliere (1935) indicates that pimelite appears to be a very altered serpentine mineral. Thermal analysis gave both loss of free water and also a peak about  $550^{\circ}$ - $750^{\circ}$ C. X-ray analysis gives the following d-spacings:

8.16  
6.92  
4.37  
3.58  
2.48  
2.00  
1.67  
1.55  
1.30

These values are entirely different from the ones obtained for pimelite in the present study and previous researches. Therefore, it seems that this material was not pimelite.

Spangenberg (1938) gives the formula for pimelite as follows:



and says no essential alumina or ferric iron is present in pimelite (Faust, 1966). It contains much zeolitic water and shows lattice shrinkage like montmorillonite; the layer spacing falls on dehydration from  $12.8\text{\AA}$  to  $9.6\text{\AA}$ , when  $X = 0.3$  and the X-ray diffraction pattern is then exactly similar to that of talc.

Strunz (1949) suggests that pimelite shows the characteristics of a nickel-saponite.

Voorthuijsen and Franzen (1951) concluded that they had synthesized pimelite and gave to their preparation the formula (per unit-cell):  $\text{Ni}_6\text{Si}_8\text{O}_{20}(\text{OH})_4$

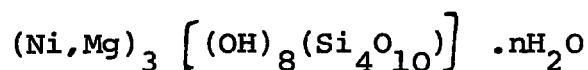


This formula is an idealized one, showing no layer charge. It seems likely, as in the case of Strese and Hofmann's Mg-montmorillonite, that there are some gaps in the octahedral layers giving rise to a negative charge.

The d-spacings obtained from their work are shown below

| <u>d(Å)</u> |       |
|-------------|-------|
| 4.21}       | 8 Br  |
| 4.13}       |       |
| 2.425       | 10 Br |
| 1.691       | 3 Br  |
| 1.513       | 8 Sh  |
| 1.307       | 5 Br  |
| 1.288       | 2 Br  |
| 1.136       | 2 Br  |
| 1.069       | 2 Br  |
| 1.008       | 2 Br  |
| 0.893       | 2 Br  |
| 0.846       | 1 Br  |
| 0.823       | 2 Br  |

Slansky (1955) suggests the following formula for pimelite (from Kremze):



He states that pimelite from Kremze shows three colour varieties:

- A. Almost white, very light-green
- B. Light-green, a little more intensively coloured than A
- C. Darker-green

All the varieties form a compact aggregate, non-transparent, but are somewhat translucent at the edges.

Hardness 2-3.

The pimelite occurs most frequently in veins (often in a network of veins) running irregularly in the weathered serpentine, less abundantly as filling between a network of veins of  $\text{SiO}_2$  and as crusts on weathered material. Some samples indicate the origin of the pimelite from colloidal solutions - the cavities in the veins are reniformly delimited, the veins are zoned by the different varieties (Slansky, 1955).

According to Slansky, under the microscope pimelite appears mostly as an anisotropic aggregate without any marked structure, more rarely as veinlets with a fibrous structure, or as more or less perfect spherulites. He also gives the following table for refractive indices of pimelite.

| <u>Pimelite</u> | <u>n<sub>Na</sub></u>    | <u>α'Na</u>              | <u>γ'Na</u>    |
|-----------------|--------------------------|--------------------------|----------------|
| Variety A       | 1,570 <sup>+</sup> 0.002 |                          |                |
| Variety B       | 1,589 <sup>+</sup> 1.598 |                          |                |
| Variety C       |                          | 1,563 <sup>+</sup> 1.368 | 1,566 to 1,571 |

The following qualitative Spectral Analysis of pimelite for the essential elements, are in accord with the present study (Table 3).

TABLE 3

Results of qualitative spectral analysis of pimelite (Data for pimelite  
A, B, C, after Slansky, 1955)

| <u>Pimelite</u> | Mg | Ni | Si | Mn | Fe | Ca | Al | Ba | Co | Cu | Ti | Cr | Zn | Na | Ag | Sr | K |
|-----------------|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|---|
| Variety A       | X  | X  | X  | +  | -  | +  | -  | -  | -  | -  | -  | -  | -  | -  | -  | -  | ? |
| Variety B       | X  | X  | X  | +  | -  | +  | -  |    | ?  | -  | -  | -  |    |    |    |    |   |
| Variety C       | X  | X  | X  | +  | -  | +  | -  | -  | -  | -  | -  | -  | -  |    | -  | -  | ? |
| Present Study   | X  | X  | X  | -  | -  | +  |    |    |    |    |    | -  |    |    |    |    | - |

X = essential

+ = subordinate to small

- = trace amount

## X-ray data of pimelite (after Slansky, 1955)

compared with pimelite from Zabkowice.  $\text{CuK}\alpha$  30/20.

| <u>Pimelite<br/>Kremze<br/>Variety A</u> |     | <u>Pimelite<br/>Křemže<br/>Variety B</u> |     | <u>Pimelite<br/>Křemže<br/>Variety C</u> |     | <u>Pimelite<br/>Zabkowice<br/>(Polsko)</u> |     |
|--|-----|--|-----|--|-----|--|-----|
| 10.0                                     | ss  | 9.9                                      | ss  | 10.0                                     | ss  | 9.9  | ss  |
| 4.49                                     | ss  | 4.67                                     | s   | 4.50                                     | ss  | 4.54                                       | ss  |
|  |     | 4.30                                     | ss  |  |     |  |     |
| 3.22                                     | s   | 3.31                                     | s   | 3.25                                     | s   | 3.21                                       | s   |
|  |     | 3.14                                     | s   |  |     |  |     |
| 2.56                                     | ssB | 2.53                                     | ssB | 2.56                                     | ssB | 2.56                                       | ssB |
| 2.29                                     | ww  | 2.28                                     | ww  | 2.27                                     | ww  | 2.27                                       | ww  |
| 1.73                                     | w   | 1.73                                     | w   | 1.73                                     | w   | 1.73                                       | w   |
| 1.52                                     | ss  | 1.52                                     | ss  | 1.53                                     | ss  | 1.54                                       | ss  |
| 1.37                                     | ww  |  |     |  |     |  |     |
| 1.31                                     | m   | 1.32                                     | m   | 1.32                                     | m   | 1.32                                       | m   |
| 1.26                                     | ww  | 1.27                                     | ww  |  |     |  |     |
| 0.99                                     | w   | 1.00                                     | w   | 1.00                                     | w   | 1.00                                       | w   |
| 0.89                                     | w   | 0.88                                     | w   | 0.89                                     | w   | 0.87                                       | w   |

Although there are minor differences, it can be seen from the above data that the varieties are identical.

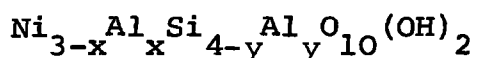
Slansky (1955) also states that pimelite does not belong to the smectite group. His evidence for this is the fact that smectites have their first basal reflection at about  $15\text{\AA}$ , whereas the first basal reflection of pimelite is about  $10\text{\AA}$ . However this does not take into account that there is the possibility of either 1, 2 or 3 layers of water in the interstitial position (layers).

According to Slansky (1955) the course of the differential thermal curves of the pimelite is

characterized by two endothermic peaks, the first between 50-200°C and the second between 750-900°C. It is probable that the first reaction represents the escape of the free water and the second one represents the escape of the constitutional water (see fig.2).

Mempton and Roy (1955) give the stability limit of pimelite as 335°C. At this point pimelite gave Ni-Talc as a decomposition product.

Warshaw and Roy (1961) indicate that pimelite is a trioctahedral smectite due to the 2:1 layering and show an idealised formula of:



Ostrowicki (1965) has pointed out that pimelite represents the richest crystalline phase of the weathering zone of serpentinites from Szklary. It forms veins which vary in thickness from tenths of a millimetre to several centimetres. Vein forms often assume a nest-like shape. Pimelite occurs in large amounts in the green and more rarely in the red weathering products of the deposit.

He concludes that pimelite is related to the talc group because of its physical and optical properties and structure. He also suggests that the position of pimelite within specific mineral groups needs to be revised.

Ostrowicki indicates that the nickel content of pimelite ranges from 17.34 to 31.81 weight percent.

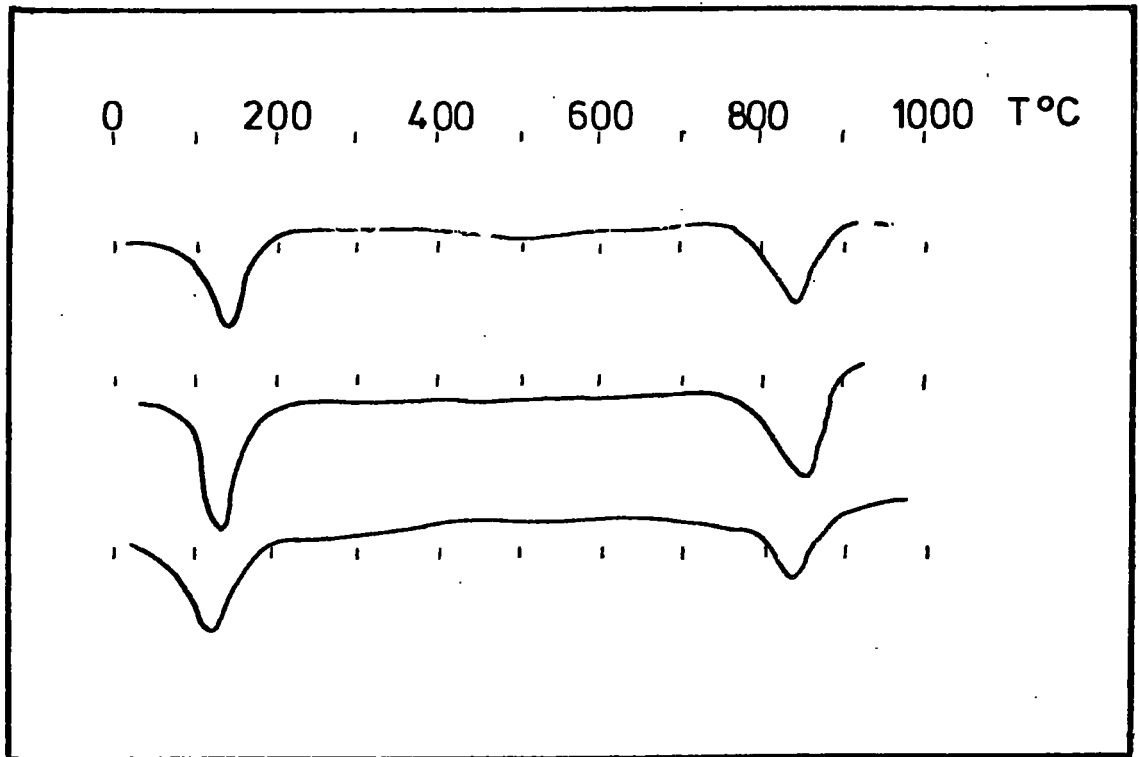


Figure 2. DTA Curves for Pimelite (From Slansky, 1955).

His analysis is shown below:

|                                |              |
|--------------------------------|--------------|
| SiO <sub>2</sub>               | 50.74        |
| Al <sub>2</sub> O <sub>3</sub> | 0.13         |
| Fe <sub>2</sub> O <sub>3</sub> | 0.04         |
| NiO                            | 31.81        |
| MgO                            | 7.36         |
| CaO                            | 0.07         |
| MnO                            | 0.02         |
| Na <sub>2</sub> O              | 0.06         |
| K <sub>2</sub> O               | 0.07         |
| H <sub>2</sub> O+              | 6.60         |
| H <sub>2</sub> O-              | 2.98         |
| Total                          | <u>99.88</u> |

Ostrowicki also gives differential thermal analysis results almost the same as Slansky (1955) (fig.3).

The following analysis was given by Faust (1966) during a study of the hydrous nickel-magnesium silicates:

|                                |              |
|--------------------------------|--------------|
| SiO <sub>2</sub>               | 47.20        |
| Al <sub>2</sub> O <sub>3</sub> | 0.22         |
| Fe <sub>2</sub> O <sub>3</sub> | 0.20         |
| NiO                            | 27.66        |
| MgO                            | 11.42        |
| CaO                            | 0.16         |
| H <sub>2</sub> O+              | 9.38         |
| H <sub>2</sub> O               | 3.64         |
| Total                          | <u>99.88</u> |

He suggests from his X-ray powder diffraction studies that pimelite is a member of the smectite group of minerals.

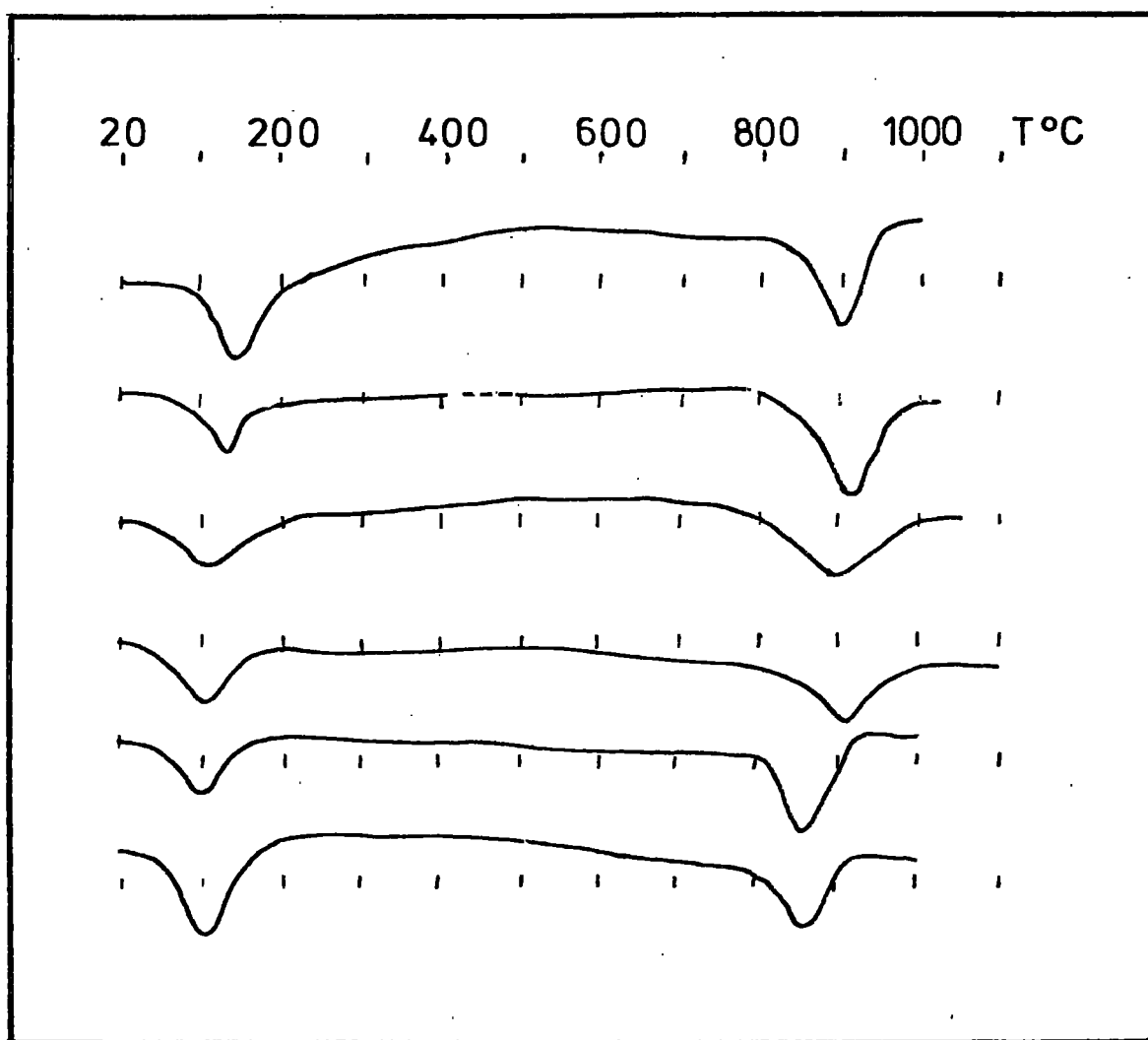


Figure 3. DTA Curves for Pimelite (From Ostrowicki, 1965).



Thorez (1975) gives the following d-spacings for  
pimelite

|       |   |     |
|-------|---|-----|
| 4.21  | } |     |
| 4.13  | } | 8B  |
| 2.425 |   | 10B |
| 1.691 |   | 3B  |
| 1.513 |   | 8B  |

and states that pimelite belongs to the Ni-saponite  
group, but does not discuss the problem.

PIMELITE IN THE PRESENT STUDY

b. Microscopic Observations: (Specimen No.9032 Silesia)

Microscopic determination of the mineral shows that it is very fine to cryptocrystalline in grain size.

The colour is always very light green. Some quartz grains(?) may be present in fine grained aggregates.

Under plane polarized light, it appears to be faintly pleochroic, while crossed-polars examination shows its medium to high birefringence (may be upper second, third order).

Beyond the presence of tiny quartz grains the mineral is fairly pure. Many irregular polishing scratches indicate that pimeelite is quite a soft mineral which is in agreement with Dana's numerical data (hardness 2.5).

Result of Electron Microprobe Analysis:

During the probe analysis the following results were obtained:

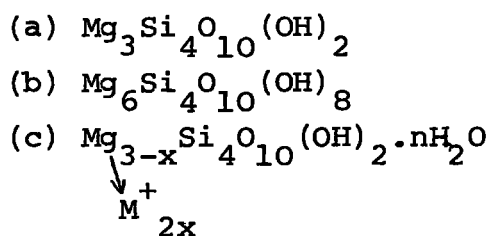
|                                |                           |
|--------------------------------|---------------------------|
| SiO <sub>2</sub>               | 41.50                     |
| Al <sub>2</sub> O <sub>3</sub> | -                         |
| FeO                            | 0.02                      |
| NiO                            | 30.78                     |
| MgO                            | 8.09                      |
| CaO                            | 0.10                      |
| H <sub>2</sub> O               | 11.00 (Obtained from TGA) |
|                                | <hr/>                     |
| Total                          | 91.49                     |

According to the above result pimelite should contain 19.51% H<sub>2</sub>O but the water loss obtained from the thermogravimetric analysis (fig.6) makes the total weight 91.49% which still indicates the presence of other oxides. Unfortunately it has not been possible to check these results within the available time.

The variation between the analysis given by Dana (1970), Ostrowicki (1965), Faust (1966) and the present study suggests that materials named pimelite by various authors are members of a solid solution series from a nickel rich end member to a magnesium end member.

#### c. Atomic Formula of Pimelite

According to the suggestions of various authors, pimelite may have the same structure as either (a) talc, (b) serpentine or (c) the smectite group. The corresponding atomic formulae would then be close to



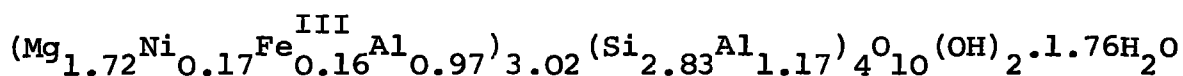
Some of the available analyses were therefore recalculated on a water free basis to 11 oxygens for (a) and (c) and 14 oxygens for (b).

The Dana analysis for alipite, which corresponds closely in nickel content with that found in the present study, in each case gave Si values appreciably higher than 4, suggesting that the material may have been

contaminated with  $\text{SiO}_2$  (Table 4).

His analysis for hard pimelite (Table 5) contains much more  $\text{Al}_2\text{O}_3$  and less  $\text{NiO}$  than in all other analyses. Compared with the normal range of compositions for talc, silica is low, alumina and water too high. For (b) the water content is low and also the number of octahedrally coordinated cations (Table 5).

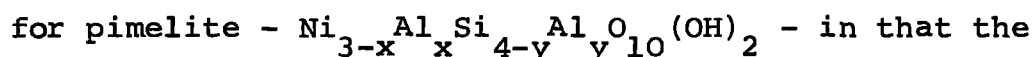
For (c) however, the formula is reasonable:



and corresponds with the formula for saponite given by



It does not correspond with the formula which they give



nickel content is so low.

The analyses of Faust (1966) (Table 6) and the present study (Table 7) also give poor agreement for (a) and (b) but can be fitted reasonably to (c) if allowance is made for vacancies in the tetrahedral positions as indicated in the formula for stevensite given by Warshaw and Roy (loc.cit.) -  $\text{Mg}_{3-x} \square_x \text{Si}_{4-y} \square_y \text{O}_{10} (\text{OH})_2$ . The absence of alumina in this formula distinguishes stevensite from all other trioctahedral smectites with the exception of hectorite, which is lithium bearing.

The formula can be written:

TABLE 4

Recalculated chemical analysis of Alipite (Dana 6th Edn., p.678)

|                                | <u>Wt.%</u>   | <u>Mol.Wt.</u> | <u>Mol.Prop</u> | <u>O's</u>    | <u>Cats.</u> | <u>Cats<br/>110's</u> | <u>Cats<br/>140's</u> |
|--------------------------------|---------------|----------------|-----------------|---------------|--------------|-----------------------|-----------------------|
| SiO <sub>2</sub>               | 54.63         | 60             | 9105            | 18210         | 9105         | 4.38*                 | 5.67*                 |
| Al <sub>2</sub> O <sub>3</sub> | 0.30          | 102            | 29              | 87            | 58           | .03                   | .04                   |
| NiO                            | 32.66         | 74.7           | 4372            | 4372          | 4372         | 2.10                  | 2.67                  |
| FeO                            | 1.13          | 72             | 156             | 156           | 156          | .08                   | .10                   |
| MgO                            | 5.89          | 40.3           | 1461            | 1461          | 1461         | 0.70                  | .89                   |
| CaO                            | 0.16          | 56             | 29              | 29            | 29           | .01                   | .02                   |
| H <sub>2</sub> O               | 5.23          | 18             | 2905            |               | 5810         | 2.79                  | 3.56                  |
|                                | <u>100.00</u> |                |                 | <u>2.2854</u> |              |                       |                       |

F1 11/2.2854 = 4.8131

F2 14/2.2854 = 6.1258

\*Both Si too high, but 11 oxygens nearest

?Probably contamination with SiO<sub>2</sub>

TABLE 5

Recalculated chemical analysis of Hard Pimelite (Dana 6th Edn., p.677)

|                                | <u>Wt.%</u>  | <u>Mol.Wt</u> | <u>Mol.Prop</u> | <u>O's</u>        | <u>Cats.</u> | <u>Cats.</u><br><u>110's</u> | <u>Cats.</u><br><u>140's</u> |
|--------------------------------|--------------|---------------|-----------------|-------------------|--------------|------------------------------|------------------------------|
| SiO <sub>2</sub>               | 35.80        | 60            | 5967            | 11934             | 5967         | 2.83                         | 3.59                         |
| Al <sub>2</sub> O <sub>3</sub> | 23.04        | 102           | 2258            | 6774              | 4516         | 2.14                         | 2.72                         |
| Fe <sub>2</sub> O <sub>3</sub> | 2.69         | 160           | 168             | 504               | 336          | .16                          | .20                          |
| NiO                            | 2.78         | 74.7          | 372             | 372               | 372          | .17                          | .22                          |
| MgO                            | 14.66        | 40.3          | 3638            | 3638              | 3638         | 1.72                         | 2.19                         |
| CaO                            | -            |               |                 |                   |              |                              |                              |
| H <sub>2</sub> O               | <u>21.03</u> | 18            | 11683           | <u>          </u> | 11683        | 5.53                         | 7.04                         |
| Total                          | 100.00       |               |                 | 2.3222            |              |                              |                              |

F1  $11/2.3222 = 4.7368$

F2  $14/2.3222 = 6.0287$

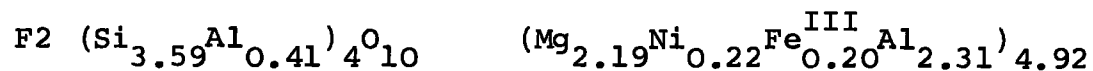
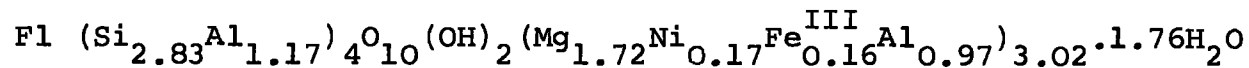


TABLE 6

Recalculated Chemical Analysis of Pimelite  
(based on Faust (1966) Chemical Analysis)

|                                | <u>Wt.%</u>  | <u>Mol.Wt.</u> | <u>Mol.Prop</u> | <u>O's</u>   | <u>Cats.</u> | <u>Cats.</u><br><u>110's</u> | <u>Cats.</u><br><u>140's</u> |
|--------------------------------|--------------|----------------|-----------------|--------------|--------------|------------------------------|------------------------------|
| SiO <sub>2</sub>               | 47.20        | 60             | 7867            | 15734        | 7867         | 3.86                         | 4.91                         |
| Al <sub>2</sub> O <sub>3</sub> | 0.22         | 102            | 22              | 66           | 44           | 0.02                         | 0.02                         |
| Fe <sub>2</sub> O <sub>3</sub> | 0.20         | 160            | 13              | 39           | 26           | 0.01                         | 0.02                         |
| NiO                            | 27.66        | 75             | 3688            | 3688         | 3688         | 1.81                         | 2.31                         |
| MgO                            | 11.42        | 40.3           | 2834            | 2834         | 2834         | 1.39                         | 1.77                         |
| CaO                            | 0.16         | 56             | 29              | 29           | 29           | 0.01                         | 0.02                         |
| H <sub>2</sub> O+              | 9.38         | 18             | 5211            | -            | 10422        | 5.12                         | 6.52                         |
| H <sub>2</sub> O-              | 3.64         |                |                 |              |              |                              |                              |
| Total                          | <u>99.88</u> |                |                 | <u>22390</u> |              |                              |                              |

Fi 11/2.239 = 4.9129

F2 14/2.239 = 6.2528

F1 (11 oxygens) (SiAlFe<sup>3+</sup>)<sub>3.89</sub> (Ni<sub>1.81</sub>Mg<sub>1.39</sub>)<sub>3.2</sub> (OH)<sub>2</sub><sup>1.56</sup>H<sub>2</sub>O

(a) Si low, Mg, Ni high, too much H<sub>2</sub>O

(b) Si too high for Si<sub>4</sub>O<sub>10</sub>, total Ni and Mg too low

F2 (14 oxygens) Ni<sub>1.81</sub>Mg<sub>1.19</sub>)<sub>3</sub> (Si<sub>3.86</sub>Al<sub>0.02</sub>□<sub>0.12</sub>)<sub>4</sub>O<sub>10</sub> (OH)<sub>2</sub><sup>1.56</sup>H<sub>2</sub>O ≈ Stevensite

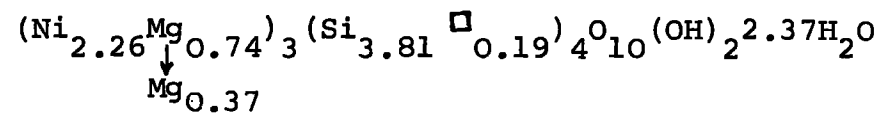


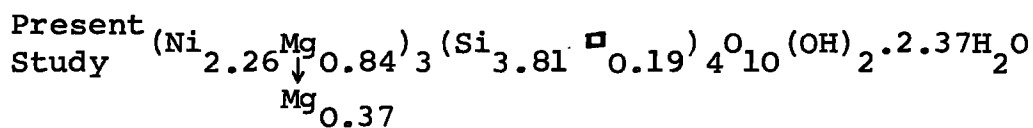
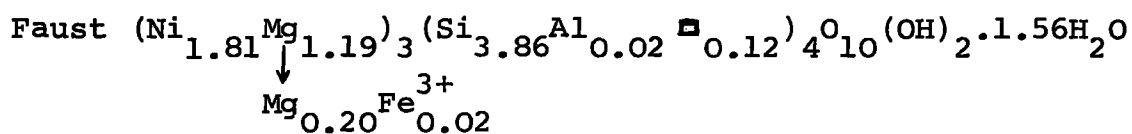
TABLE 7

Recalculated Chemical Analysis of Pimelite

|                                | <u>Wt.%</u>  | <u>Mol.Wt</u> | <u>Mol.Prop.</u> | <u>Oxygens</u> | <u>Cations</u> | <u>Cations<br/>11 oxygens</u> |
|--------------------------------|--------------|---------------|------------------|----------------|----------------|-------------------------------|
| SiO <sub>2</sub>               | 41.50        | 60            | 6916             | 13832          | 6916           | 3.81                          |
| Al <sub>2</sub> O <sub>3</sub> | -            |               |                  |                |                |                               |
| FeO                            | 0.02         | 72            | 2                | 2              | 2              | -                             |
| NiO                            | 30.78        | 75            | 4104             | 4104           | 4104           | 2.26                          |
| MgO                            | 8.09         | 40.3          | 2007             | 2007           | 2007           | 1.11                          |
| CaO                            | 0.10         | 56            | 1                | 1              | 1              |                               |
| H <sub>2</sub> O               | <u>11.00</u> | 18            | 6111             | -              | 12222          | 6.74                          |
| Total                          | 91.49        |               |                  | <u>19946</u>   |                |                               |

$$11/1.9946 = 5.5149$$





Both analyses show a similar deficiency in silicon which will give a layer charge that is apparently compensated by interlayer cations as indicated. The presence of vacancies in the octahedral layer indicated by the Warshaw and Roy formula is not supported by these analyses. The pimelite of the present study has a somewhat higher nickel content than that of Faust, but both are apparently nickel-rich varieties of the trioctahedral smectite stevensite.

Confirmation of this possibility required the examination of X-ray diffraction data.

d. X-ray study of pimelite in present work

The following results were obtained from the X-ray powder photographs;

| <u>2 <math>\theta</math></u> Degrees | <u>d</u> (Å) | <u>Rel.Int</u> (Visual Estimate)  |
|--------------------------------------|--------------|-----------------------------------|
| 8.825                                | 10.00        | 80 (Broad)                        |
| 19.50                                | 4.55         | 50 (line at head of diffuse band) |
| 27.55                                | 3.23         | 20 (Broad)                        |
| 33.90                                | 2.64         | 40 (Head of diffuse band)         |
| 39.60                                | 2.27         | 20                                |
| 53.175                               | 1.72         | 10                                |
| 60.775                               | 1.52         | 100                               |
| 71.825                               | 1.31         | 5                                 |

The diffraction lines were difficult to measure accurately due to their broad nature and the values shown above are the average values obtained from the measurements of a number of films. Also due to the crystallization of the specimen, no back reflections were obtained. The only distinct line produced was the  $1.52\text{\AA}$  (060) reflection indicating the trioctahedral nature of pimelite (Warshaw and Roy, 1961).

The d-spacings obtained from the X-ray diffractometer shown below;

| <u>d(<math>\text{\AA}</math>)</u> | <u>Rel.Int</u> |
|-----------------------------------|----------------|
| 9.97-10.27                        | 100            |
| 4.54- 4.56                        | 40             |
| 3.23- 3.24                        | 20             |
| 2.62- 2.64                        | 40             |
| 1.52-                             | 80             |

After soaking overnight in ethylene glycol the following results were obtained:

| <u>d(<math>\text{\AA}</math>)</u> | <u>Rel.Int</u> |
|-----------------------------------|----------------|
| 15.09                             | 100            |
| 4.56                              | 40             |
| 3.34                              | 20             |
| 2.64-2.68                         | 40             |
| 1.52                              | 80             |

The first recorded reflection expands up to  $15.09\text{\AA}$  but changes in the other reflections were negligible. The  $1.52\text{\AA}$  line remained constant (Plate 2). The fact that the first recorded peak expands from  $10\text{\AA}$  up to  $15\text{\AA}$  suggests a smectite type structure with interlayer water molecules being replaced by ethylene glycol molecules.

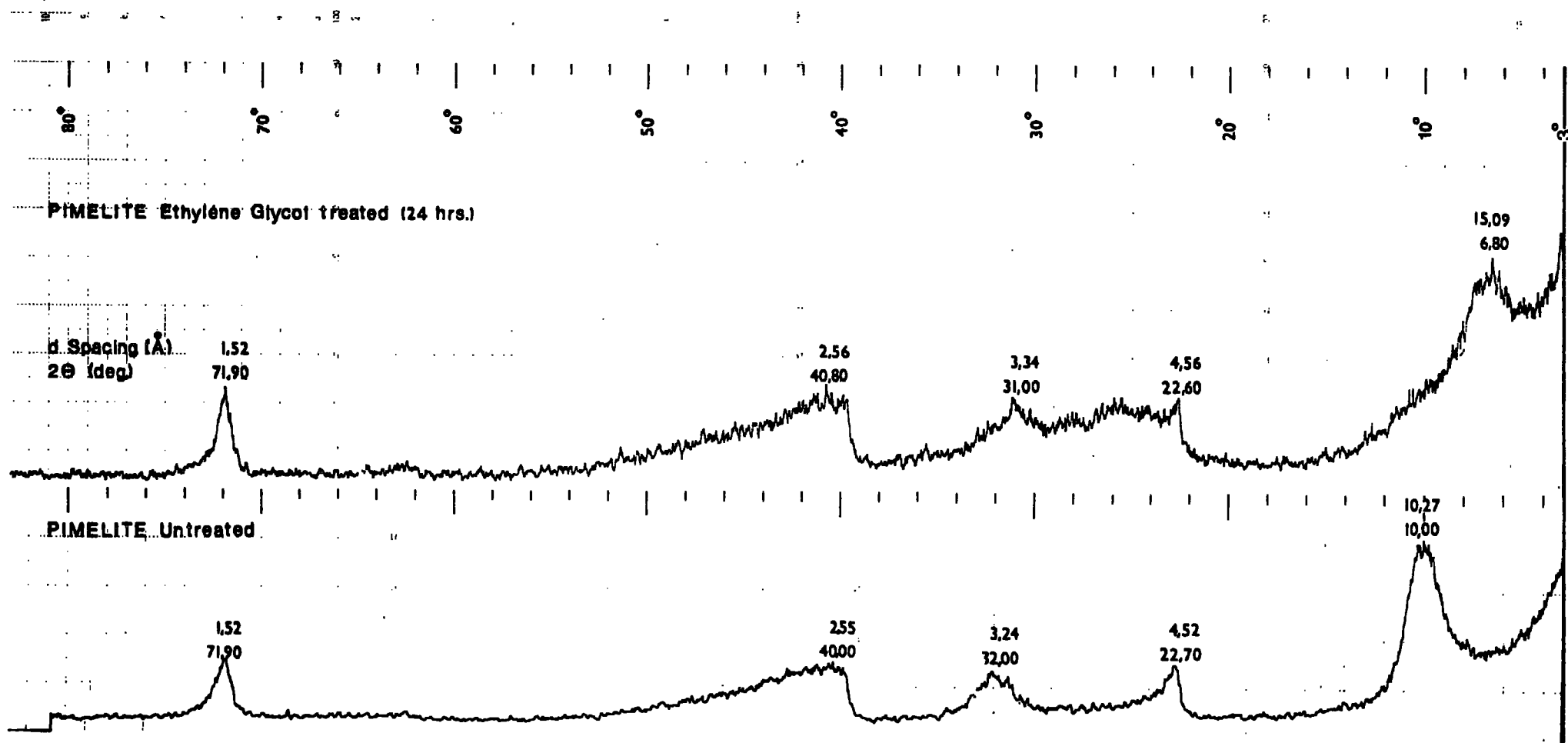


Plate 2. Untreated and Ethylene glycol treated (24 hrs) X.R.D. charts for pimelate.

As well as smear mounts, cavity mounts of the pimelite sample were run and the results obtained are shown below.

| <u>d(Å)</u> | <u>Rel.Int.</u> |
|-------------|-----------------|
| 10.27       | 100             |
| 4.56        | 50              |
| 3.22        | 20              |
| 2.59        | 50              |
| 1.52        | 80              |
| 1.31        | 5 (negligible)  |

There are indications from the films obtained using copper radiation of a diffraction maximum at approximately  $15\text{Å}$  although this peak was rather uncertain and difficult to measure as it was barely clear of the hole in the film (Plate 3).

#### e. DTA Analysis of Pimelite

During the differential thermal analysis the pimelite was first heated in air flow which gave two endothermic and one exothermic peak (fig.4). The first endothermic peak occurs when the temperature is between  $50^{\circ}$  and  $200^{\circ}\text{C}$  indicating the escape of free water as Slansky (1955) stated. The second endothermic peak occurs between the temperature of  $800^{\circ}$ - $1000^{\circ}$  (if the sample firmly packed, fig.4/c). This peak also confirms the conclusion of Slansky (1955) in fig.2 and Ostrowicki (1965) in fig.3 which indicates the escape of the combined water at the same temperature.

Apart from these two peaks, between the temperature of  $300^{\circ}$ - $500^{\circ}$  and sometimes  $200^{\circ}$ - $500^{\circ}$  (this difference is

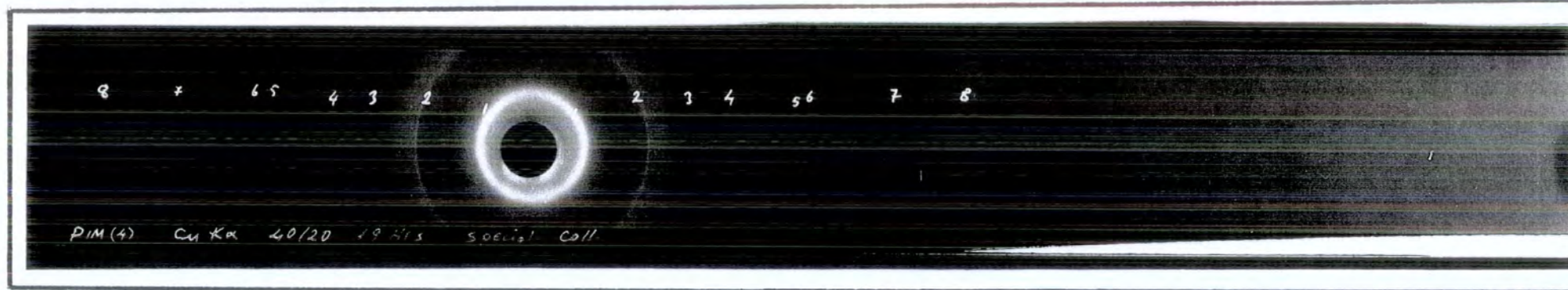


Plate 3. X-ray film of Pimelite

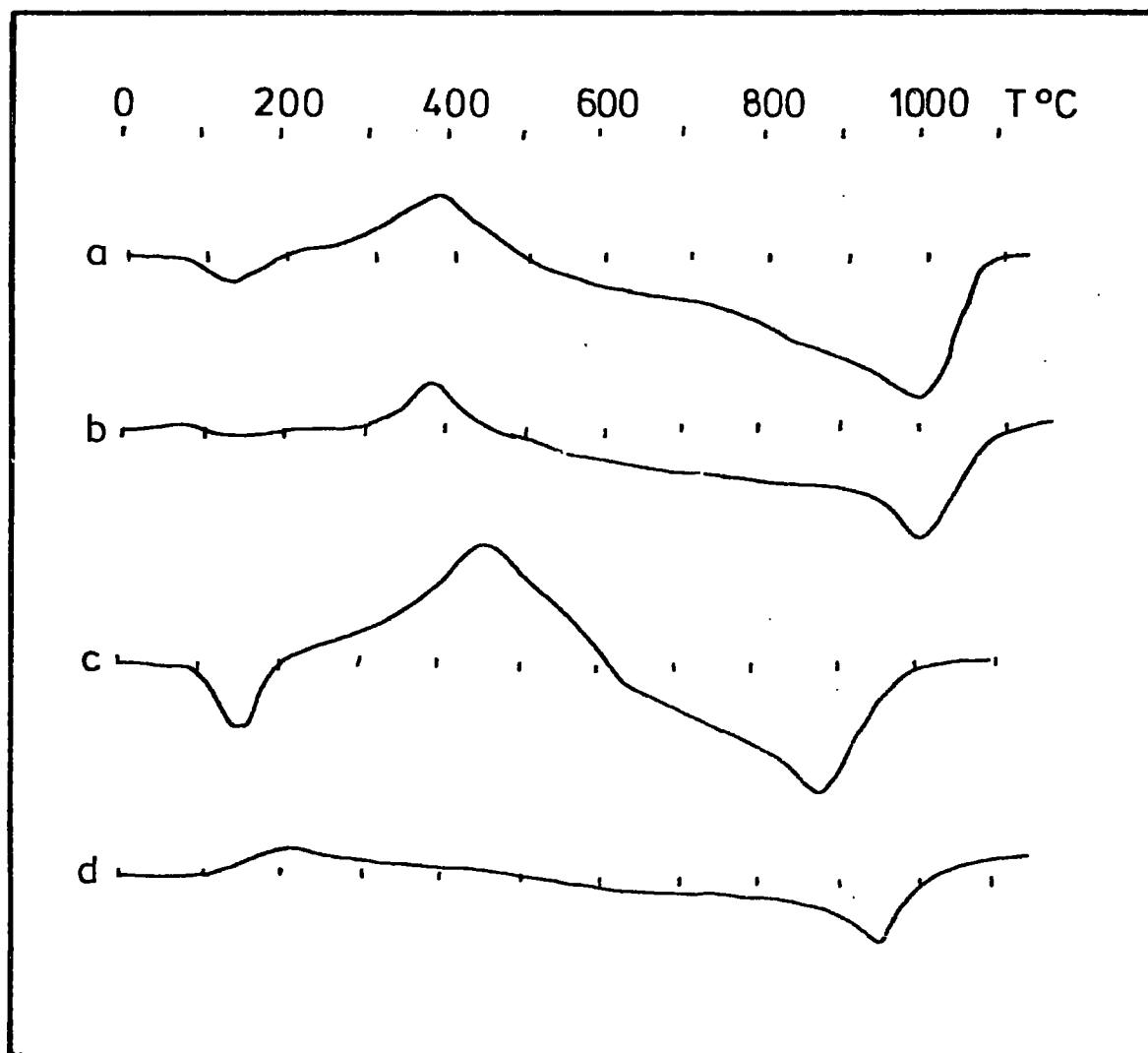


Figure 4. DTA Curves for Pimelite

a,b running in air flow

c " " " " (specimen firmly packed)

d running in nitrogen flow

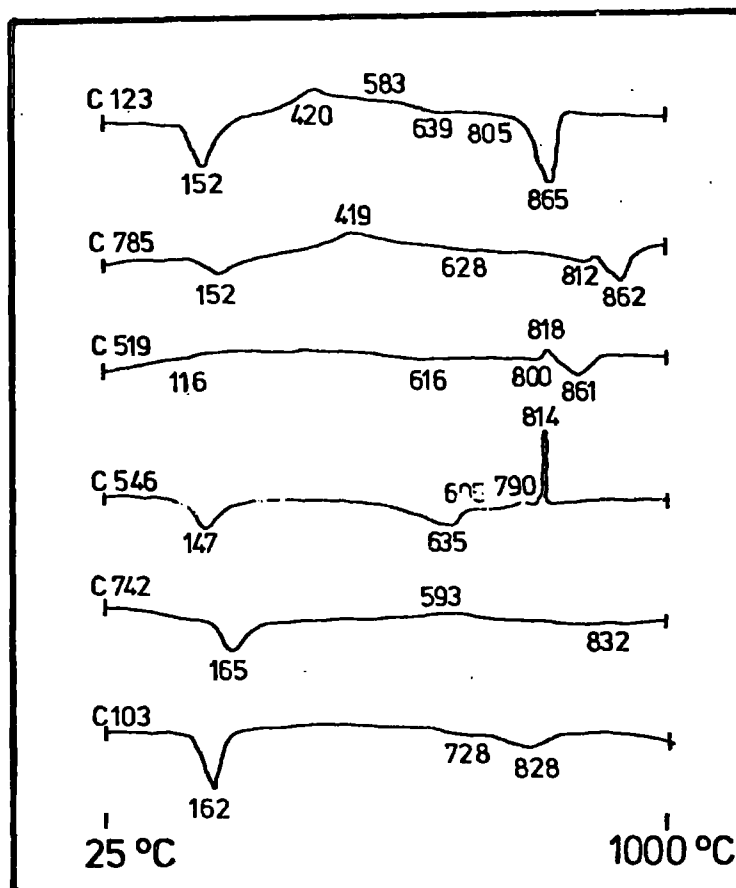


Figure 5. DTA Curves for Stevensite  
(From Faust et al., 1959).

C-123, Stevensite No.4, Paterson, New Jersey;

C-785, Synthetic stevensite, Bowen & Tuttle preparation T-2-37.

C-519, Stevensite No.5 with serpentine, Mine Creek, North Carolina, USA.

C-546, Ghassoulite, Morocco, C-742; and Hectorite, Hector, California,

C-103.



most probably related to the firmness of packing and the amount of material) an exothermic peak occurs. This peak has not been recorded in the literature.

An attempt was made to investigate the presence of any organic material, in case its presence might be having an effect on the results in air flow. Consequently during the DTA in a nitrogen flow the exothermic peak decreased substantially and the first endothermic peak almost vanished, whereas the second endothermic peak remained constant (fig.4/d).

After this section had been written, the curves for stevensite shown in figure 5 were discovered in the paper by Faust, Hathaway and Millot 1959. The first of these curves (C-123) is closely similar to figure 4a obtained in this study.

f. TGA analysis of pimelite

The average of three thermogravimetric analyses of pimelite gave about 11% water loss (see fig.7) (Dehydration curve of pimelite done by Slansky 1955 also can be seen in fig.6).

The total analysis of pimelite from the electron microprobe together with the  $H_2O$  obtained from TGA still shows a low value suggesting perhaps the presence of other oxides. No  $CO_2$  was observed by wet chemistry using the method suggested by Groves (1951).

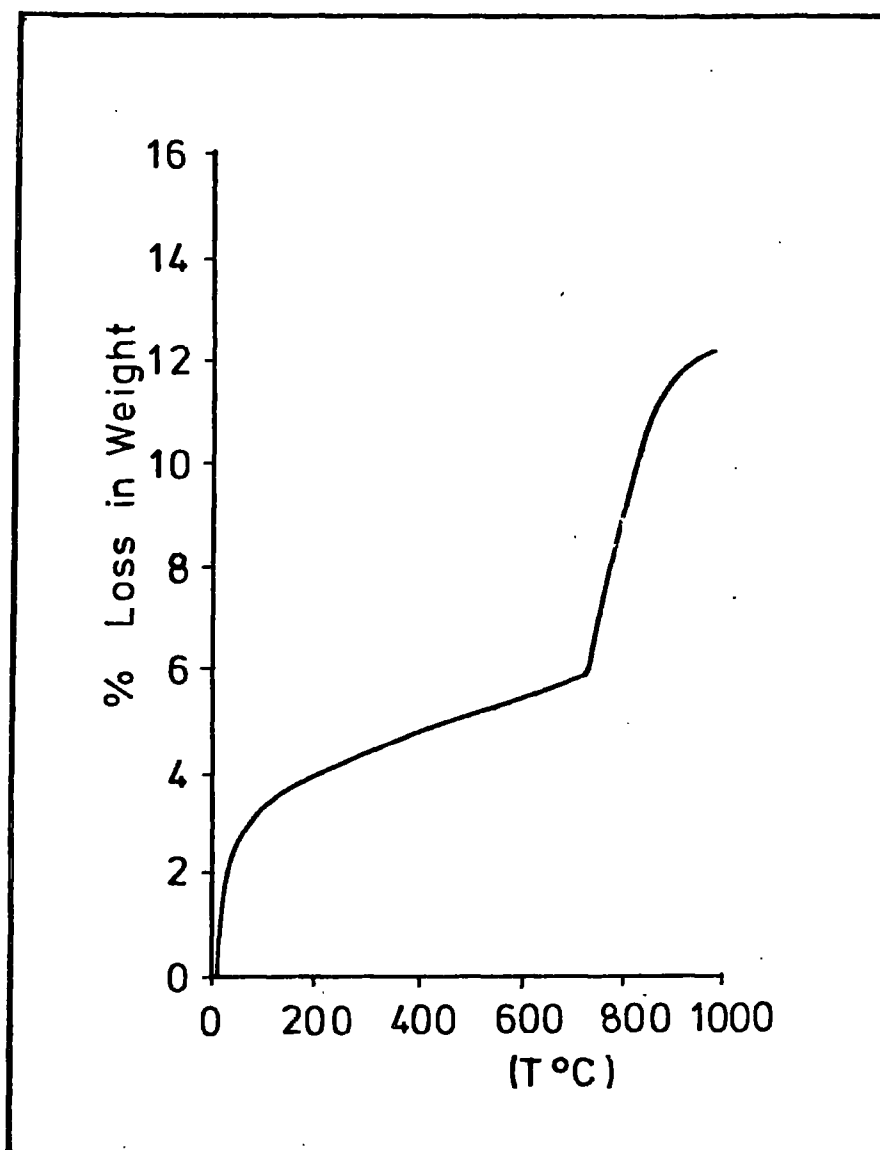


Figure 6. Dehydration Curve for Pimelite  
(From Slansky, 1955).

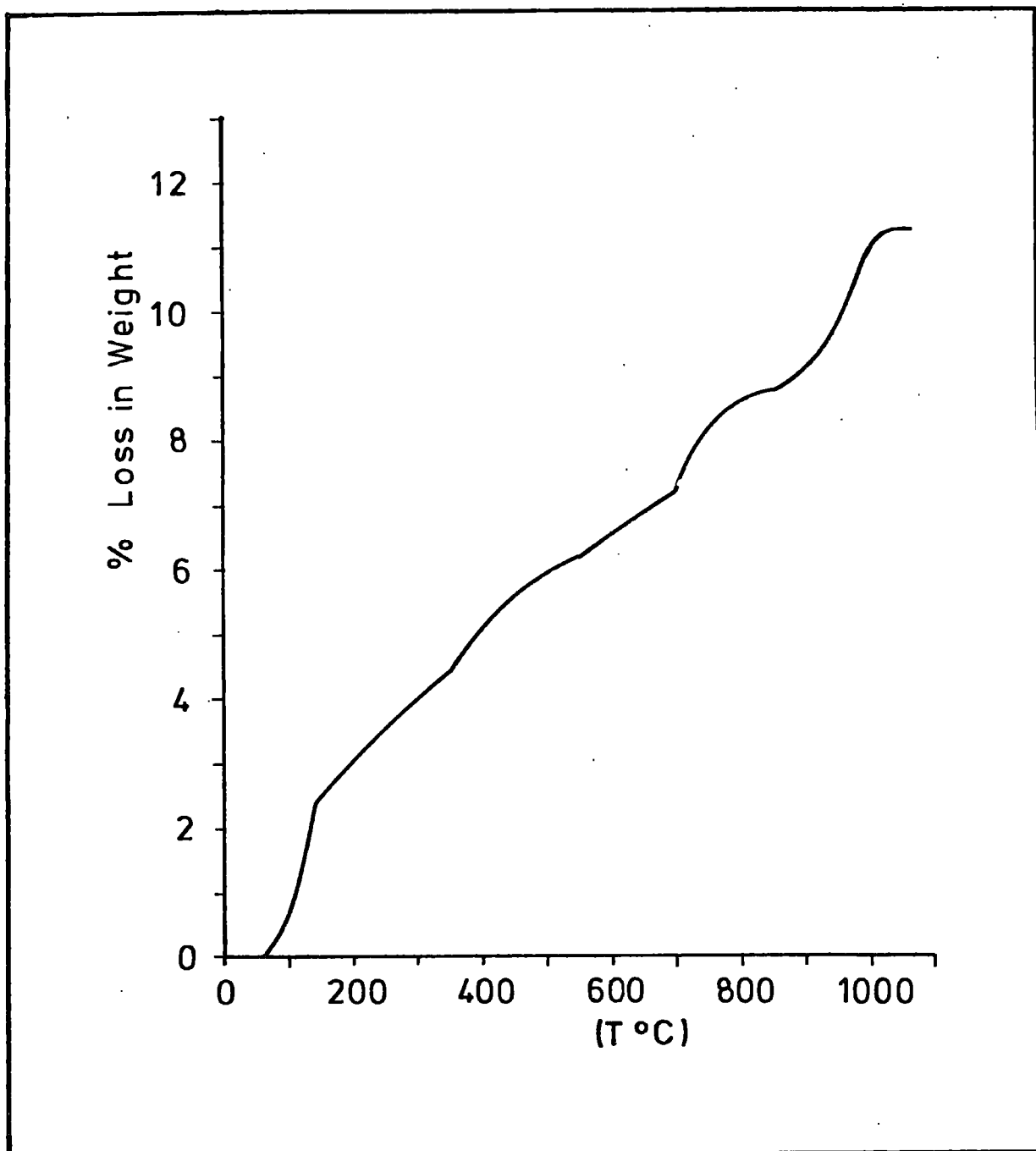


Figure 7. Dehydration Curve for Pimelite

The total analysis obtained, together with several analyses from the literature are shown for pimelite on pages 33-36.

g. Conclusions

From the swelling properties of the pimelite examined and recalculation of the chemical analysis, it is concluded that it is a smectite mineral related to stevensite. D.T.A. results correspond with those for stevensite given by Faust (fig.5) and pimelite is apparently a nickel stevensite. The mineral of this study is equivalent to 75% of the pure nickel end member with 25% of the pure magnesium end member and interlayer magnesium. The Faust (1961) analysis is less rich in nickel.

Having reached these conclusions, a paper by Brindley and Maksimovic(1974) was brought to the author's attention. Their conclusions differ from those given above, suggesting that pimelite is talc-like and can only be caused to swell partially and with difficulty by soaking in ethylene glycol for about 1 month (note added in proof p.227). This was not found in the present investigation - overnight soaking was sufficient.

Thus it is not thought necessary to modify the above conclusions, except to agree with Brindley and Maksimovic (1974) that further work on a wider range of pimelite specimens would be justified.

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