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MINERALOGICAL AND PETROGRAPHICAL INVESTIGATION OF PYROPHYLLITE-BEARING ROCKS FROM MANUELS, NEWFOUNDLAND

by

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July 9, 1958.

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MINERALOGICAL AND PETROGRAPHICAL INVESTIGATION OF PYROPHYLLITE-BEARING ROCKS FROM MANUELS, NEWFOUNDLAND

Summary

Nineteen samples of pyrophyllite-bearing rocks, submitted by Mr. F. Gover, Deputy Minister, Newfoundland Department of Mines and Resources, have been examined mineralogically and petrographically. A number of investigatory techniques were tested. X-ray diffraction and chemical analysis were found to be the most informative, when used in conjunction with microscopic examination.

Intensive examinations were carried out on four samples reported to represent material from (1) Oval Quarry, (2) Mine Hill, (3) the milled product being produced at present and (4) waste rock. These results were then used as the basis for a less intensive examination of the remaining fifteen samples, many of which were similar to the first four.

The mineralogical compositions of the three important samples are shown in the following table:

	No.1 (M-81) Oval Quarry	No.2 (M-82) Mine Hill	No.3 (M-83) Milled Product
Pyrophyllite	42%	44%	66%
Muscovite	2	48	3
Quartz	50	4	26
Kaolinite	5	2	3
Zoisite	<1	<1	<1
Saussurite	<1	<1	<1
Chlorite			

It was requested that the mineralogical work be directed toward the investigation of those features which would be of assistance in determining the possibility of beneficiating the first two samples to produce materials similar in composition to No.3 (M-83). The aim of a beneficiation procedure would be, in Sample No.1 (M-81), an increase in alumina content and, in No.2 (M-82), a decrease in the alkali content. This can be accomplished, in the first case, by removal of quartz and, in the second, by removal of muscovite. It has been shown, by means of selective staining, by consideration of the crystal chemistry of muscovite and pyrophyllite and by the demonstrated absence of any other alkali-bearing minerals that all alkalies must be contained in muscovite.

Examination of sized fractions of pulverized samples has established that appreciable liberation does not occur in sizes larger than about 24 microns. Since complete removal of the contaminants is not necessary to produce products comparable to No.3 (M-83), particularly in the case of the quartz in No.1 (M-81), it may be possible that satisfactory products can be recovered without complete liberation. Blending of concentrates from the high silica and the high alkali materials would further reduce the degree of concentration of each necessary to produce a satisfactory product.

Introduction

In February 1958 nineteen samples of pyrophyllite-bearing rocks from the Manuels area of Newfoundland were submitted to this Division by Mr. Gover, Deputy Minister, Newfoundland Department of Mines and Resources. In a letter to Mr. M.F.Goudge (Ref.M.R./2/15(a), Feb.10, 1958), a mineralogical examination of them was requested. The purpose of this examination was to assist in the development of a process for the beneficiation of two types of pyrophyllite-bearing material to "....a finished product which will be not lower in alumina, nor much higher in alkali, than the chemical content of that contained in the 100 pound bag of milled product which we are sending you as sample No.3".

Two shipments of samples were received. The first consisted of four samples labelled, and described by Mr.Gover as follows: (Laboratory No.M-81) "Sample No.1, Oval, is a barrel containing about 450 pounds of crude, primary crushed pyrophyllite having a low alumina, (10 to 15%) and low alkali (less than 1%) content".

(Lab.No.M-82) "Sample No.2, Mine Hill, is a barrel containing about 450 pounds of crude, primary crushed pyrophyllite having a high alumina (over 20%) and a high alkali (over 1%) content".

(Lab.No.M-83) "Sample No.3, Milled Product, is a 100 pound bag of pyrophyllite as produced in the mill from high grade raw material (over 20% alumina and less than 1% alkali)".

(Lab. No.M-84) "Sample No.4, Country Rock, is a 50 pound bag of pyrophyllitized conglomerate which will have to be thrown out as waste by hand sorting .....".

The following partial chemical analyses by Mr. Ewart C. Burke of Newfoundland Minerals Limited were also submitted:

Lab.No.	Sample No.	% Al2O3	% L.O.I.	% Total Alkalies
M-81	1. Oval	15.47	3.02	0.42
M-83	2. Jonnies Quarry (Mine Hill)	31.74	5.16	4.00
M-83	3. Production	21.47	4.11	0.48

A second shipment of fifteen samples was received on February 25. In the accompanying letter from Mr. Gover (Ref.MB/2/15(a), Feb. 17, 1958) were included the following descriptions:

(Lab. No.M-85A) "No.1, Specimen of high grade pyrophyllite taken 1000' north of Oval Quarry".

(Lab. No.M-85B) "No.2, Specimen of silicifed? rhyolite taken at 1050' north of base line, Oval Quarry Area".

(Lab. No.M-86A) "No.3, Specimen of high grade pyrophyllite with high alkali content, taken from Jonnies Quarry in Mine Hill Area".

(Lab. No.M-86B) "No.4, Specimen of volcanic rock taken from Mine Hill, approximately 520 south and 0+15 west".

(Lab. No.M-86C) "No.5, Specimen of rhyolite breccia ?? taken from Mine Hill approximately 7+20 south, 1+20 west".

(Lab. No.M-87A) "No.6, Specimen of rhyolite, taken from Mine Hill, 9+20 south -- 1+60 west".

(Lab. No.M-87B) " No.7, Specimen of high grade pyrophyllite with high alkali content, taken from Main Lens of Zone I - Mine Hill".

(Lab. No.M-87C) "No.8, Specimen of low grade pyrophyllite, taken from Mine Hill (near DDH 10)".

(Lab. No.M-87D) "No.9, Specimen of conglomeratic rhyolite schist, taken from Oval Quarry -- waste rock".

(Lab. No.M-88A) "No.10, Specimen of high grade pyrophyllite, taken from New Cut in Oval Quarry".

(Lab. No.M-88B) "No.11, Specimen of high grade pyrophyllite, taken from northwest corner of Oval Quarry".

(Lab. No.M-88C) "No.12, Specimen of altered sediment or volcanic rock, 250'N of Oval Quarry".

(Lab. No.M-89A) "No.13, Specimen of a siliceous lens, taken from Jonnies Quarry, Mine Hill".

(Lab. No.M-89B) "No.14, Specimen of low grade pyrophyllite, taken from Jonnies Quarry, Mine Hill".

(Lab. No.M-89C) "No.15, Specimen of low grade pyrophyllite, taken from Bulk Sample No.1, Oval Quarry".

Methods of Investigation

The mineral assemblages of the samples, coupled with their extremely fine grain-size and intimate association made it very difficult to assess the samples quantitatively or even semi-quantitatively. No one procedure provided sufficient information and various combinations of the following methods were tried: microscopic examination of fragments; thin and polished-section examination; size fractionation; oil-immersion mounts of crushed material; X-ray diffraction examination of bulk samples, hand-picked material and sized fractions; differential thermal analysis (DTA); thermogravimetric analysis; acid etching and selective staining; chemical analysis.

Published work and previous Mines Branch investigations of material from the Manuels area have suggested that the principal constituents are pyrophyllite, muscovite and quartz. Preliminary X-ray diffraction analysis showed this to be true in these samples and the problem then became one of determining the distribution and proportions of the major mineral constituents, the distribution of alkalies, the identification of the minor constituents and the assessment of features which might be of value in beneficiation investigations. The work was concentrated on the four samples of the first shipment (M-81, M-82, M-83 and M-84). The results were then used as a basis for the less intensive examination of the remaining fifteen samples, which was carried out, principally, by means of thin sections and X-ray diffraction. The discussions of the two groups will therefore be presented separately.

The extremely fine grain size, intimate association and similarity in physical properties all served to make microscopic identification not practicable. X-ray diffraction, however, proved to be very informative and this technique was used with considerable success throughout the investigation.

PART A - INVESTIGATION OF SAMPLES M-81 (OVAL QUARRY), M-82 (MINE HILL), M-83 (MILLED PRODUCT) AND M-84 (WASTE ROCK)

Sample M-81. Oval Quarry-Macroscopic Features

The sample is composed of irregular, equant fragments of greyish-white material with a maximum size of about 2 to 3 inches. They are uniform in appearance and under low magnification are seen to be massive and unfoliated with small, scattered, hard, greenish-grey areas and small, vuggy zones lined with friable, yellowish aggregates of pyrophyllite and quartz. On slip surfaces in some of the fragments is a thin coating of white, fibrous pyrophyllite with quartz and kaolinite. A photograph of the fragments is shown in Fig.1 (a).

Thin Sections

Three thin sections cut from different fragments show considerable variation. They are composed of pyrophyllite and quartz with small amounts of zoisite and saussurite. Zoisite occurs, usually, as small (about 0.02 mm) grains although rare grains as large as 0.5 mm were observed (Figs. 3a, 3b). It is colourless to faintly yellow in most cases, and is easily recognized by the high relief in contrast to the quartz-pyrophyllite aggregates. The material designated as saussurite may be, in part, kaolinite and occurs as small, fine-grained, opaque, white masses (Fig.4a).

Pyrophyllite and quartz normally are found in microgranular aggregates (Figs. 4a,b; 5a,b) but the proportion of each is variable from place to place (Figs. 6a, b). Usually, the quartz-rich zones are very fine-grained and irregular in shape but rare, rounded aggregates of coarser quartz were observed (Figs. 6a, b) as well as a single large, blocky grain about 0.1 mm in size. Much of the quartz has a dusty appearance caused by a profusion of tiny zoisite (?) grains. Rare books of pyrophyllite up to 0.05 mm in width were found but most of pyrophyllite flakes are smaller than 0.02 mm. It also occurs in rare, narrow veinlets with the flakes oriented parallel to the walls.

Polished Sections

In polished sections, with vertical illumination, it can be seen that the rock is fragmental in appearance with fine-grained, hard, quartz-rich masses which take a higher polish then the fine-grained pyrophyllite-rich matrix which is too soft to polish well. This contrast, shown in Fig.2a has been accentuated by etching with hydrofluoric acid fumes.

Sample M-82, Mine Hill-Macroscopic Features

This sample is made up of irregular, elongate fragments with a splintery fracture, a yellowish-green colour, a maximum length of about 10 inches and a maximum width of about 3 inches. They are massive and homogeneous in appearance with the exception of small pits filled with a fine-grained, powdery, white mixture of pyrophyllite and muscovite. Under low magnification the fragments still appear to be fine-grained, massive and homogeneous (Fig.1.b). This sample differs from M-81 (Oval Quarry) in that it is of uniform hardness throughout and much softer. The yellowish-green colour is also distinctive.

Thin Sections

Three sections were cut from different fragments and examined microscopically. They are composed of a relatively homogeneous, microgranular aggregate of muscovite and pyrophyllite. The constituents cannot be distinguished optically. Small, opaque, white masses of saussurite and kaolinite (?) and small, equant grains of zoisite showing high relief are also present (Figs.7a,b). A small amount of quartz occurs in the form of scattered, clear grains of low relief.

Polished Sections

In polished section, with vertical illumination, the fragments still appear to be homogeneous and no trace of a fragmental nature can be seen (Fig.2b).

Sample M-81, Milled Product

This sample was received in the form of a very fine, white powder that has a distinctly soapy feel. No contaminating minerals could be observed under low magnification. The material is not amenable to study in thin or polished sections and the examination of it was restricted to X-ray, thermal, chemical and oil-immersion techniques.

Sample M-84, Waste Rock-Macroscopic Features

The sample was received in the form of lumps up to about 6 inches in size. The surfaces of most of them are discoloured by red or greenish-black stains. On fresh surfaces the rock is seen to be fragmental.

Polished Sections

On polished surfaces the fragmental nature of the lumps is much more clearly shown (Fig.8a). Most of the fragments are hard, white to light-green in colour and seem to be very fine-grained and siliceous in nature. There are also some hard, red, jaspery fragments and few of a soft, green, chloritic type. The fragments are mostly rounded to sub-angular in shape and form a higher proportion of the rock than the matrix which is also fine-grained, hard and siliceous, with some iron oxide stain. The staining is particularly noticeable along fractures, as shown in Fig. 8a.

Thin Sections

Microscopically the rock is found to be composed of fragments composed of aggregates of fine-grained, granular quartz with small flakes of pyrophyllite and/or muscovite (Figs. 8b; 9a, b). The matrix is also an aggregate of granular quartz but is usually somewhat coarser than that in the fragments and is also speckled with small flakes of pyrophyllite and/or muscovite. The soft, green fragments are composed of fine-grained chlorite. Those which appear dark in Fig. 8b are, in part, coloured by dusty, red, iron oxides. The contrast between matrix and fragments is shown in Figs. 9a and 9b, with the coarser, quartzose matrix being sharply marked off from the finer-grained, quartzose fragment.

X-ray Diffraction

The X-ray powder diffraction technique with Debye-Scherrer cameras was found to be one of the most informative methods for the investigation of the pyrophyllite-muscovite-quartz association. Although the procedure does not provide quantitative measurements it is possible to compare the strengths of the patterns of principal constituents in different samples and arrive at values which are relative estimates of the compositions. In pulverized material and in small samples of selected material the technique was much more informative than any of the other procedures used.

Sample M-81, Oval Quarry

The sample contains quartz and pyrophyllite as the principal constituents with a smaller amount of muscovite. Kaolinite was not detected in the powder photographs of the bulk sample but it was found along with fibrous pyrophyllite on certain slip surfaces in some of the fragments.

Sample M-82, Mine Hill

In this case pyrophyllite and muscovite are the main constituents with a smaller amount of quartz.

Sample M-83, Milled Product

Pyrophyllite is the chief constituent of this sample. It is accompanied by quartz and a small but detectable, a ount of muscovite.

Sample M-84, Waste Rock

It was not considered worthwhile to grind this sample and obtain a representative portion but selected material produced X-ray patterns which suggest that muscovite, rather than pyrophyllite, is the principal constituent of the tiny flakes which give a speckled appearance to the quartzose rock in thin section.

In all four of these samples the minor constituents are not present in proportions large enough to produce detectable X-ray patterns.

Size Fractionation

The samples of the three pyrophyllite-bearing rocks were split into convenient size fractions in a Roller analyzer, a type of air classifier. From a microscopic examination of the various sizes it was possible to determine the degree of liberation of the constituents and the extent of their persistence into the finer sizes. For the selection of the proper flow rates in the analyzer an average specific gravity of about 2.7 was assumed. The smallest size that could be separated was 4 microns (expressed, of course, in equivalent spherical diameters). Succeeding size limits were then chosen at 6, 12, 24 and 48 microns. Measurement of the grain sizes by means of a calibrated scale in the microscope showed that the fractions are very uniform and closely approximate actual sizes. The size limits quoted above will therefore be used in referring to the fractions.

In the +48 micron fraction there is negligible liberation of the constituents (Figs.10a, 10b). This is changed only slightly in the -48+24 micron fraction (Figs.11a,11b). Liberation is incomplete in the -24+12 micron fraction (Figs.12a, 12b) and seems to be complete in the -12+6 micron portion (Figs.13a,13b). In the -6 micron sizes no further change was noted.

The illustrations have been drawn from the results of the work on the Milled Product but the samples from Oval Quarry and Mine Hill show the same features and the conclusions may be applied equally well to all three. X-ray powder pictures were made of each of the size fractions of the Milled Product and they showed that the minimum size of most of the quartz is about 12 microns. Below 4 microns the pyrophyllite pattern became weak and diffuse, showing, perhaps, that the degree of ordering of the crystal structure was decreased in the smaller particles.

Differential Thermal Analysis (DTA)

Differential thermal analyses were carried out on pulverized portions of samples M-81, M-82, M-83 and pure samples of muscovite and pyrophyllite. The DTA records are reproduced in this report as Thermal Curves 1A, 2A, 3A, 4A and 5A, respectively, along with thermal balance curves which will be discussed in a later section.

The DTA technique utilizes the difference in temperature between an unknown sample and a thermally-inert reference material to obtain a measurement of the endothermic and exothermic reactions which take place during heating, at a constant rate, up to about 1000°C. It is useful for distinguishing many minerals but it does not seem to be diagnostic for mixtures of pyrophyllite and muscovite. Each of these gives a large endothermic reaction at about the same temperature and in mixtures the two peaks tend to merge.

Sample M-81, Oval Quarry (Thermal Curve 1A)

In this record there is a small endothermic reaction peak at 685°C and a very small peak at 865°C. These are interpreted as pyrophyllite reactions. A distinct endothermic peak caused by the inversion of quartz occurs at 575°C. Measurements of this peak area suggest that about 48% quartz is present in the sample.

Sample M-82, Pine Hill (Thermal Curve 2A)

There is a large endothermic peak with two maxima at 670°C and 690°C. Since it is known from X-ray data that the sample contains both muscovite and pyrophyllite this may be interpreted as indicating the presence of the two minerals. However, the double peak is really not so well defined that it could be used as a diagnostic criterion.

A small endothermic peak also occurs at 885°C. No quartz reaction was detected. The absence of this peak does not prove that quartz is absent but that, if present, it is below the amount necessary for detection in this mixture.

Sample M-83, Milled Product (Thermal Curve 3A)

Endothermic indications ascribed to pyrophyllite occur as a large peak at 685°C and a small one at 895°C. A small peak resulting from the quartz inversion occurs at 575°C and from measurements of its area the proportion of quartz was determined as approximately 23%.

Pyrophyllite (Curve 4A)

For comparison with the DTA curves described above, a specimen of pure pyrophyllite from Staley, North Carolina was run under comparable conditions. The curve shows only one endothermic peak, at 785°C.

Muscovite (Curve 5A)

A sample of pure muscovite from near Mattawa, Ontario was pulverized and sized in the Roller Analyzer. The -15 micron fraction produced this DTA curve. The thermal reactions in this case are very poorly defined with only one small, broad peak at about 800°C.

These curves suggest, from their variability and lack of clearly defined peaks, that the technique is not very satisfactory for resolving mixtures of muscovite and pyrophyllite. The quantitative measurements of the quartz content were obtained by heating the samples sufficiently to drive off the water contained in the pyrophyllite and muscovite, cooling down to a point below the inversion temperature of quartz and then reheating. In this way the hydrous minerals were rendered thermally inert and the area of the isolated quartz peak could be measured for quantitative determination.

Thermogravimetric Analysis

The usefulness of this technique, by which the change of weight of a sample is continuously recorded during heating at a constant rate, is similar to that of DTA. The difficulties encountered in attempting to make semi-quantitative or quantitative measurements are also similar. In this investigation the apparatus used is a standard model of a photographically-recording Chevenard thermal balance equipped with a continuous temperature-recording mechanism.

The weight-loss of hydrated minerals such as muscovite and pyrophyllite usually takes place over a fairly wide temperature range, even at slow heating rates. Particle size and the degree of crystallinity also introduce other, poorly-known complexities.

The weight-loss curves are reproduced on the same sheets as the corresponding DTA curves for convenience.

Sample M-81 (Thermal Curve 1B)

This curve shows the weight-loss to be in two stages. The first commences at about 470°C, reaches a maximum rate at 575°C and begins to slow down again at about 650°C. The second stage seems to affect the slope of the curve at 700°C and shows as a straight line portion from there to about 825°C where it again decreases. The weight-loss is completed at 870°C and totals 2.80%.

Sample M-82 (Thermal Curve 2B)

In contrast to the record of the previous sample, this one shows a much greater loss which corresponds to an increase in muscovite and a reduction in quartz content. There is a slow loss of weight noticeable from the beginning of the curve at 100°C until the major loss begins at about 400°C. It reaches a maximum rate about 575°C and the curve begins to flatten at about 725°C. The loss of weight is completed at about 860°C and totals 4.66%.

Sample M-83 (Thermal Curve 3B)

In this curve features noted in both of the previous ones are observed. There is a slow decrease in weight from 100°C to about 425°C where the rate of loss begins to increase. The maximum rate is reached about 590°C and is maintained to about 660°C. There is a change, at 730°C, to a second stage with a lower rate of loss which continues until about 890°C. The total weight loss is 3.91%.

Pyrophyllite (Curve 4B)

The sample of pure pyrophyllite from Staley North Carolina produced a curve with one clearly-defined stage of dehydration. The weight-loss begins at about 450°C and is completed at 850°C. It should be noted that the total loss of 5.13% is greater than the theoretical water content of pyrophyllite (5.00%). The fact that no water was driven off below 400°C suggests that inter-particle or absorbed water was not likely the source of this excess. A small amount of kaolinite may be the reason for it.

Muscovite (Curves 5B, 5C)

These two curves illustrate the variation in weight-loss characteristics with particle size. A specimen of pure muscovite has been pulverized and separated in the Roller Analyzer into -15 micron and +15 micron fractions. The coarser material requires a higher temperature to drive off the contained water and at the end of the run it had not reached as great a loss as had the finer. The difference in the total loss from the finer mica (4.48%) and the theoretical water content of potassic mica (4.51%) is so small that it can be assumed to be an experimental error. The loss from the coarser mica totalled 4.37%.

Selective Mineral Staining

Most mineral staining techniques are not applicable to this mineral assemblage and the only test investigated is one that is specific for potassium. The staining process is carried out by etching a polished surface of the specimen in hydrofluoric acid fumes and staining the potassium-bearing minerals in a solution of sodium cobaltinitrite, Minerals such as feldspars and feldspathoids containing potassium are stained a brilliant yellow. Ordinarily, muscovite, even though it contains potassium, is unaffected by this treatment. Pyrophyllite is also unaffected.

Polished sections and thin sections of Samples M-81 and M-82 were tested. It was found that by prolonged etching and staining, the thin sections of M-82 took a distinct yellow colour. The stained grains have a higher relief than the surrounding material and can even be distinguished in black-and-white photographs (Figs. 14a, 14b). In others, however, the contrast is not so great (Figs. 15a, 15b). Under high magnification it can be seen that the stain is restricted to certain grains in the section. This fact, along with the failure to produce a stain on the low-alkali material can be taken as proof that the stain is a definite indication of the distribution of the alkali. Since potassium occurs in only certain grains of the muscovite-pyrophyllite assemblage it must be contained in the muscovite. The crystal structure of pyrophyllite is such that it cannot accommodate univalent ions of the size of the alkalies in interlayer sites as the muscovite structure can.

It is also observed in the stained sections that the muscovite is, in general, evenly distributed through the rock and intimately associated with the pyrophyllite. There seems to be little possibility, therefore, of concentrating, efficiently, the pyrophyllite without reducing the rock to a size near that required for liberation, i.e. about 24 microns.

Chemical Analysis

Partial chemical analyses were carried out on three of the samples (M-81, M-82, M-83) so that the results of the chemical method could be compared directly with those of the mineralogical techniques. Although partial chemical analyses were submitted with the samples it seemed advisable to have information on the actual material being used. Also it was necessary to have determinations of silica, soda and potash in addition to alumina and L.O.I. to enable quantitative mineralogical calculations to be made.

CHEMICAL ANALYSES (MINES BRANCH, G.A.KENT, ANALYST)

	M-81 (Oval Quarry)	M-82 (Mine Hill)	M-83 (Milled Product)
SiO_2	80.82%	56.01%	73.02%
Al_2O_3	14.38	31.70	21.22
K_2O	0.16	5.33	0.26
Na_2O	0.06	0.25	0.10
L.O.I.	3.01	4.80	3.99
Total	97.43	98.09	98.59
Weight Loss (Thermal Balance)	2.90	4.66	3.91

The determination of the weight loss by means of the thermal balance resulted in values slightly lower than those determined by the standard L.O.I. technique. The difference is, principally, the result of the application of a correction for the small amount of water taken up by the sample during handling and weighing. It is unlikely that the error introduced in this way would be so large as to be significant in most applications. However, it is critical in the quantitative calculations of this investigation.

The mineralogical composition of each sample was calculated in the following manner, using as guides the qualitative X-ray, microscopic and thermal determinations. The alkalies were first assigned to muscovite (potassium mica) and to paragonite (the sodium analogue of muscovite). It will be understood, of course, that the alkalies are likely present, in the rock, in only one mineral, a muscovite with part of the potassium replaced by sodium. After subtracting the requisite amounts of silica, alumina and water (weight loss determination), the remaining alumina was first assigned to pyrophyllite. Since all water remaining could not be included in this amount of pyrophyllite it was necessary to divide, proportionally, the water and alumina between pyrophyllite (5.00% H_2O) and kaolinite (14.0% H_2O). The justification for this procedure is the detection of small amounts of kaolinite, by X-ray diffraction, in some of the material. The remaining silica was considered to be a measure of the quartz content.

CALCULATED MINERALOGICAL COMPOSITIONS

M-81 (Oval Quarry)

	SiO_2	Al_2O_3	H_2O	K_2O	Na_2O	Total
Muscovite	0.62%	0.52%	0.06%	0.16%	-	1.36%
Paragonite	0.30	0.35	0.03	-	0.06%	0.74
Pyrophyllite	27.70	11.74	2.08	-	-	41.52
Kaolinite	2.10	1.77	0.63	-	-	4.50
Quartz (diff.)	50.10	-	-	-	-	50.10
Total	80.82	14.38	2.80	0.16	0.06	98.22

M-82 (Mine Hill)

	SiO_2	Al_2O_3	H_2O	K_2O	Na_2O	Total
Muscovite	20.6%	17.3%	2.04%	5.33%	---	45.3%
Paragonite	1.5	1.2	0.15	---	0.25%	3.10
Pyrophyllite	29.3	12.4	2.19	---	---	43.9
Kaolinite	0.9	0.79	0.28	---	---	2.0
Quartz (diff.)	3.7	---	---	---	---	3.7
Total	56.0	31.7	4.66	5.33	0.25	98.0

CALCULATED MINERALOGICAL COMPOSITIONS (cont'd)

M-83 (Milled Product)

	SiO_2	Al_2O_3	H_2O	K_2O	Na_2O	Total
Muscovite	1.01 %	0.85 %	0.10 %	0.26 %	---	2.22 %
Paragonite	0.59	0.49	0.06	---	0.10 %	1.24
Pyrophyllite	43.8	18.58	3.28	---	---	65.66
Kaolinite	1.54	1.30	0.47	---	---	3.31
Quartz (diff.)	26.08	---	---	---	---	26.08
Total	73.02	21.22	3.91	0.26	0.10	98.51

For the purposes of these calculations the errors introduced by ignoring the small amounts of saussurite and zoisite are not great. Iron, magnesium and titanium are likely the most plentiful of the other cations present. As silicates they would claim some of the silica and, likely, some of the alumina which would result in a slight decrease in the quartz value. As oxides they would not affect the calculated values.

PART B - EXAMINATION OF SAMPLES M-85A(No.1) to M-89C(NO.15)

Procedure

These samples were received in the form of hand specimens consisting, in most cases, of a single fragment. The fragments were examined closely under low-power magnification and one thin section was cut from each, perpendicular to foliation, if any. Representative portions of the fragments and selected portions of anything that appeared to be unusual were submitted to X-ray diffraction analysis.

Sample M-85A (No.1)

This sample is composed of a very fine-grained, homogeneous, massive rock with an irregular to conchoidal fracture. It has a distinctly soapy feel and can be scratched easily with a needle.

The X-ray photograph shows only the pattern of pyrophyllite. In thin section it is found to be a homogeneous mass of flakes less than 0.03 mm in size. There are a few small, opaque, white grains of saussurite and some zoisite. Most of the grains of the latter are very small although a few are as large as 0.05 mm. The larger ones are subhedral and equant in shape with many inclusions which make them translucent. A faint zoning is visible in some of the zoisite grains.

The specimen represents a high-grade pyrophyllite that should be lower in alkalies and higher in alumina than the Milled Product.

Sample M-85B (No.2)

The single fragment in the sample is a fine-grained, light-grey, homogeneous dense rock. It is massive in appearance with an irregular fracture and appears to be felsitic or cherty in composition.

An X-ray powder picture is composed of a strong quartz pattern with very weak pyrophyllite and muscovite lines.

In thin section it is a granular mass of quartz with most grains about 0.05 mm and a few as large as 0.10 mm. Pyrophyllite and muscovite are present in very fine-grained irregular masses scattered through the microgranular quartz aggregate. There are also a few small masses of fine-grained, opaque, white saussurite and some zoisite.

Sample M-86A(No.3)

This specimen is made up of three fragments of a fine-grained, homogeneous, yellowish-green rock. The fragments are much like the Mine Hill (M-82) material in having elongate form, a splintery fracture and, externally, a sheared appearance. On a sawn surface, however, they appear to be massive and unfoliated.

A prominent pyrophyllite pattern with a weaker muscovite pattern makes up the X-ray picture.

Microscopic examination of a thin section shows that the pyrophyllite and muscovite are in the usual fine-grained, intimate mixture with a maximum grain size of about 0.02 mm. Small amounts of fine-grained quartz, saussurite and zoisite are also present.

Sample M86B(No.4)

This rock differs from the previous ones in being variable in colour. It is a fine, dense, green rock with irregular to rounded areas (fragments?) of light-green material. Both phases are hard, being barely scratched by a needle.

The X-ray picture suggests that it is composed of quartz with some muscovite and pyrophyllite.

In thin section there is an indistinct mass of irregular quartz grains with finer-grained pyrophyllite and muscovite scattered through them. The light-coloured areas are composed of quartz grains that are either larger or smaller than those in the matrix. These areas also contain a smaller proportion of muscovite and pyrophyllite. Some lenticular zones, which might be amygdules, were observed. They are lined with equant, well-formed grains of quartz and filled with fine-grained masses of chlorite. There must be some chlorite scattered throughout the rock to give it the green colour but it could not be distinguished optically.

Sample M-86C(No.5)

The rock in this sample is distinctly fragmental with fragments that are hard, fine-grained and white to green in colour. The matrix is subordinate and is composed of fine-grained, pink, sheared material. Most of the fragments are irregular to rounded in shape but some show the lenticular outline typical of sheared fragmental rocks.

In thin section the rock appears as an inequigranular aggregate of quartz, chlorite and muscovite with minor iron oxides(?). The largest quartz grains are about 0.75 mm. The sheared nature of the rock is not noticeable in this thin section.

Sample M-87A(No.6)

The sample is composed of a single fragment of a fine-grained, dense, light-grey, hard rock with ghosts of white, siliceous fragments(?) and soft, yellowish-green spots. The exterior is somewhat sheared.

A strong quartz pattern dominates the X-ray powder picture although a weak pyrophyllite pattern can be detected along with a suggestion of the basal reflection of muscovite.

A thin section is composed of fine-grained quartz (up to 0.05 mm) with irregular masses of pyrophyllite flakes (about 0.01 mm). There are also a few pyrophyllite and/or muscovite veinlets. The rock is likely a sheared, altered fragmental rock, with soft concentrations of pyrophyllite.

Sample M-87B(No.7)

The single fragment in this sample is a fine-grained, dense, soft, green rock with some grey, mottled areas. It is elongate with a splintery fracture and a massive, unfoliated structure. There are some irregular, hard, light-green areas. The rock is similar to that from Mine Hill (M-82).

X-ray pictures show that the main mass of the rock is pyrophyllite with a small proportion of muscovite. The grey areas are pure pyrophyllite and the hard green areas represent concentrations of quartz and chlorite which surely causes the green colour even though it cannot be resolved optically or in the X-ray pictures.

Microscopically, in thin section, the rock is a mass of fine-grained pyrophyllite and muscovite made up of flakes about 0.02 mm in size. There are scattered grains of saussurite and irregular masses of coarse-grained (up to 0.1 mm) aggregates of pyrophyllite.

Sample M-87C(No.8)

This rock is fine-grained, grey and dense with shadowy outlines of what were likely original fragments. It is rather hard but can be scratched by a needle. A few green spots are softer than the remainder. The fracture is uneven.

The X-ray pictures show that the rock is predominantly quartz with smaller amounts of pyrophyllite and muscovite. The soft green spots are concentrations of the latter two.

In thin section the main mass of the rock is composed of quartz grains up to 0.02 mm in size with variable concentrations of pyrophyllite and muscovite. The concentrations represented by the green spots may be pseudomorphous after a primary constituent. Veinlets of pyrophyllite and/or muscovite and quartz cut the rock. They are all much coarser than the rest of the section.

Sample M-87D(No.9)

This rock is obviously fragmental and appears to be identical to M-84 (Waste Rock). Most of the fragments are white to light-green and hard although some are soft and dark green in colour. The matrix is light-green, hard and dense. Thread-like zones of red iron oxides are visible along fractures.

X-ray pictures show that muscovite is present in addition to the dominant quartz and some chlorite.

In thin section it is apparent that most of the fragments and the matrix are composed of fine-grained quartz (less than 0.02 mm) with variable amounts of muscovite and chlorite. The fragments contain a smaller amount of muscovite than the matrix, in most cases. Some lenticular bands are visible in which the quartz grains are larger than 0.02 mm. The dark green fragments are composed of chlorite with subordinate quartz and muscovite.

Sample M-88A(No.10)

This rock is a fine-grained, dense, soft rock with a mottled, light-green colour and an irregular to conchoidal fracture.

An X-ray powder picture shows only the pattern of pyrophyllite.

Microscopically, it is a massive, unfoliated, fine-grained (less than 0.04 mm) mass of pyrophyllite with scattered opaque grains of saussurite and reddish iron oxides(?). Some grains may be either quartz or basal sections of clear grains of pyrophyllite.

Sample M-88B(No.11)

This material is almost identical in appearance to M-88A(No.10). The X-ray pattern is also that of pure pyrophyllite.

Microscopically this rock is practically identical to the previous sample but the section contains one large anhedral grain of a colourless to light-green mineral with moderate birefringence and extremely high relief (zircon?). There are also many small grains of zoisite. These two samples and M-85A(No.1) contain higher proportions of pyrophyllite than any of the other sample submitted.

Sample M-88C(No.12)

The rock fragment making up this sample is obviously of sedimentary origin. It is fine-grained, stratified and green in colour. The layers are of varying shades of green with dark green and white fragments. An apparent unconformity traverses the middle of the fragment and cross-bedding is visible. As shown in Fig.16a, the rock weathers white. X-ray pictures show that quartz, chlorite and muscovite are prominent constituents.

In thin sections the banding is also evident, with rounded grains of quartz and a fine-grained quartzose rock being the most plentiful types of fragments. Irregular masses of fine-grained chlorite represent the dark-green fragments which are visible macroscopically. The matrix is composed of a fine-grained mass of quartz and muscovite. Although it is not distinguishable, some chlorite must be disseminated through the rock to give it the green colour. Opaque white masses of saussurite(?) are scattered throughout.

Sample M-89A(No.13)

This sample is a single fragment of a medium-grained quartzose rock which has been sheared. Elongate quartz grains and some pyrophyllite are visible on the slip surfaces. Some small vuggy zones are present.

Microscopically, it is composed of a sutured aggregate of quartz grains up to 1 mm in size with a small amount of interstitial pyrophyllite. The quartz grains are clouded by a profusion of tiny inclusions less than 5 microns (0.005 mm) in size which appear, with a few exceptions, to be isotropic.

Sample M-89B(No.14)

This fine-grained, dense rock contains indistinct, white to light-green areas (fragments?) in a light-green matrix. It is sheared and cannot be scratched with a needle.

An X-ray picture is made up of a strong quartz pattern and a moderately strong pyrophyllite pattern and a faint suggestion of the muscovite basal reflection.

In thin section the rock is seen to be composed of anhedral quartz grains (about 0.1 mm) and concentrations of pyrophyllite. Pyrophyllite is also scattered through the quartz. A veinlet of pure quartz divides the section into two areas. One of these is composed of equant, anhedral quartz grains. The other contains grains that are anhedral but somewhat elongated and have a preferred orientation.

Sample M-89C(No.15)

This fine-grained, dense rock also contains indistinct, hard white areas (fragments?) in a softer, light-green matrix. It is sheared, with pyrophyllite coating the slip surfaces.

The X-ray picture is made up of moderately strong pyrophyllite and quartz patterns with, possibly, a very weak muscovite pattern.

Microscopically, it is composed of fine-grained quartz (less than 0.02 mm) with fine-grained masses and scattered flakes of pyrophyllite. There are also some small opaque white masses of saussurite.

CONCLUSIONS

A combination of microscopic examination and X-ray diffraction for identification of the constituents, with chemical analysis for a quantitative basis, was found to be the most satisfactory method of investigation for this quartz-pyrophyllite-muscovite assemblage. Selective staining and size fractionation supplied much additional information. The two thermal procedures investigated were found to be less informative, chiefly because they depend upon dehydration characteristics, which are notoriously sensitive to such variables as crystal size, degree of crystallinity and heating rate. In addition, the dehydration processes of the hydrous components in these samples occur over the same general range of temperature.

The mineralogical compositions of the three samples which were investigated intensively are shown in the following table:

	M-81 (Oval Quarry)	M-82 (Mine Hill)	M-83 (Milled Product)
Pyrophyllite	41.5%	43.9%	66.8%
Muscovite	2.1	48.4	3.5
Quartz	50.1	3.7	25.7
Kaolinite	4.5	2.0	2.5
Zoisite	<1.	<1.	<1.
Saussurite	<1.	<1.	<1.

The rocks are extremely fine-grained aggregates in which the constituents are intimately associated. The Oval Quarry Sample (M-81) contains hard, quartz-rich fragments(?) in a soft, pyrophyllite-rich matrix. The quartz and pyrophyllite are intimately mixed, however, in both phases. Small pure quartz zones are found only rarely and a few, vein-like bodies composed of pyrophyllite and/or muscovite were observed. Sample M-82 (Mine Hill) is a homogeneous, microgranular aggregate of muscovite and pyrophyllite as shown by staining tests.

Size fractionation of pulverized portions of the samples show that the Milled Product is liberated about 24 microns. The quartz-rich sample is liberated at a slightly coarser size and the muscovite-rich material is composed of monomineralic grains at a slightly finer size.

The fifteen samples in the second shipment are rather variable but are composed, essentially, of the same mineral assemblage, i.e. quartz, pyrophyllite, muscovite, zoisite and saussurite with, in some cases, chlorite and/or kaolinite. Four samples are high-grade pyrophyllite: M-85A(No.1); M-87B(No.7); M-88A(No.10); M-88B(No.11). These specimens have a pyrophyllite content that is comparable to, or higher then, that of the Milled Product (M-83). One sample, M-86A(No.3) is apparently identical to the Mine Hill sample (M-82) and composed, principally of pyrophyllite and muscovite in similar proportions. The quartz-pyrophyllite assemblage similar to M-81 (Oval Quarry) is shown by two specimens, M-89B(No.14) and M-89C(No.15). They have a similar fragmental nature with hard siliceous components in a softer pyrophyllite-rich matrix. Samples M-86C(No.5) and M-87D(No.9) are nearly identical to the sample of Waste Rock (M-84). The remaining six samples are composed, predominantly, of quartz. One of them, M-88C(No.12) has distinctive sedimentary structures. Four are likely metamorphosed quartzose fragmental rocks, either quartzites or quartz conglomerates: M-86B(No.4); M-87A(No.6); M-87C(No.8) and M-89A(No.13). The last one of this group has been strongly sheared.

One sample, M-858 is an equigranular quartz rock, likely a metamorphosed quartzite.

Although it is not feasible to discuss in detail, the origin these rocks without corresponding field evidence, many of them appear to be derived from quartzose sedimentary rocks of various kinds. Those with high proportions of pyrophyllite and muscovite are, of course, more difficult to assess. The absence of identifiable amounts of feldspars is a noteworthy characteristic of all the rocks examined.

<u>Fig. 1(a) Sample M-81 (Oval)</u> - Photograph of fragments showing equant shape and irregular fracture. Magnification 1 X, oblique illumination.

<u>Fig. 1(b) Sample M-82 (Mine Hill)</u> - Photograph of fragments showing elongate shape and splintery fracture. Magnification 1 X, oblique illumination.

<u>Fig. 2(a) Sample M-81 (Oval)</u> - Photomicrograph of polished section etched with hydrofluoric acid fumes and showing quartz-rich fragments (white) in a pyrophyllite-rich matrix (black). Magnification 5 X, vertical illumination.

<u>Fig. 2(b) Sample M-82 (Mine Hill)</u> - Photomicrograph of a polished section etched with hydrofluoric acid fumes and showing non-fragmental structure. The discontinuities represent gross fractures in the rock.

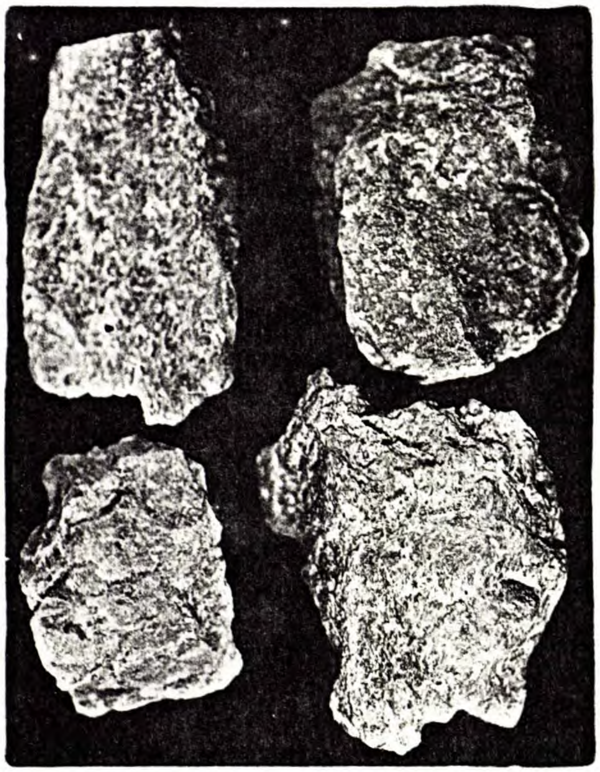


Fig.1(a)

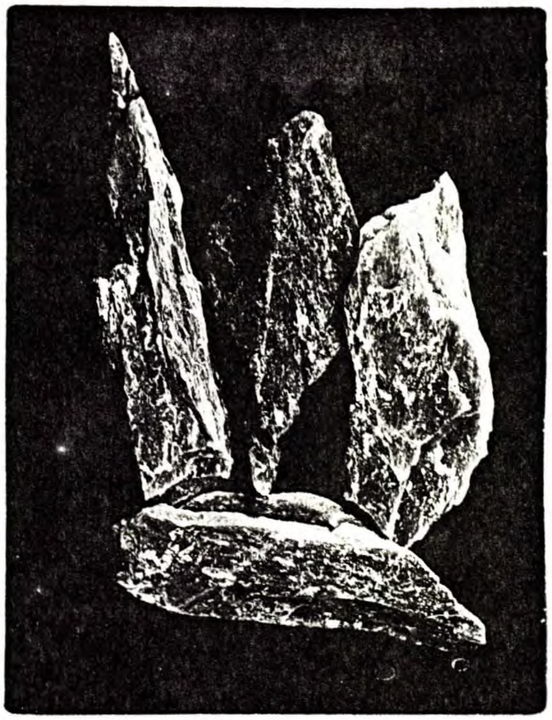


Fig. 1(b)

Fig.2(a)

Fig. 2(b)

Fig. 3(a) Sample M-81 (Oval) - Photomicrograph of a thin section showing large anhedral grains of zoisite (high relief) in a fine-grained mass of pyrophyllite and quartz. Magnification 200 X, polarized transmitted light.

Fig. 3(b) - As in 3(a) with crossed polaroids.

Fig. 4(a) Sample M-81 (Oval) - Photomicrograph of a thin section showing microgranular aggregates of pyrophyllite and quartz with a few grains of saussurite (black). The quartz shows as clear areas with low relief. Magnification 200 X, polarized transmitted light.

Fig. 4(b) - As in 4(a) with crossed polaroids.

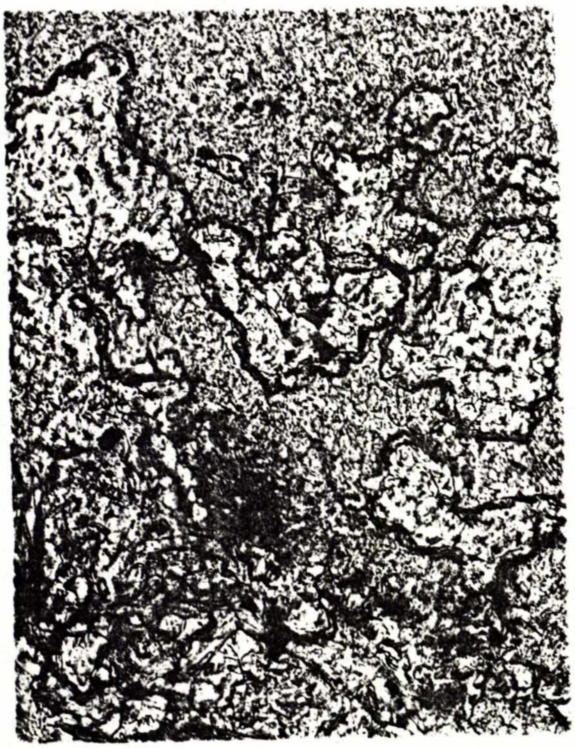


Fig. 3(a)

Fig.3(b)

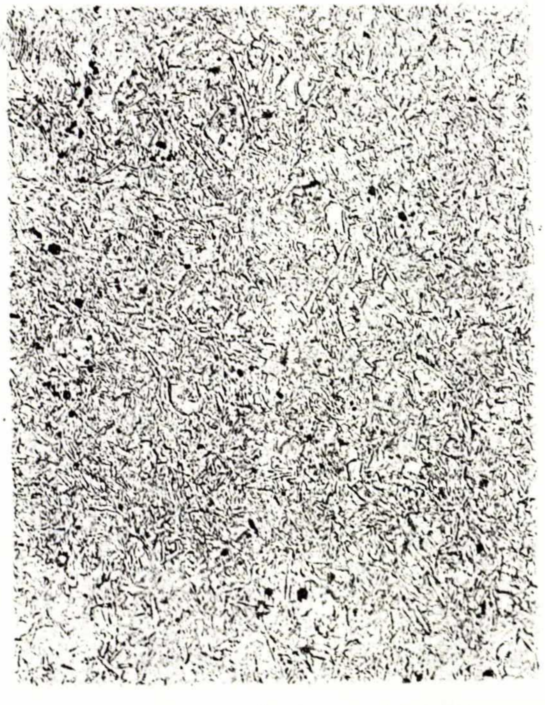


Fig. 4(a)

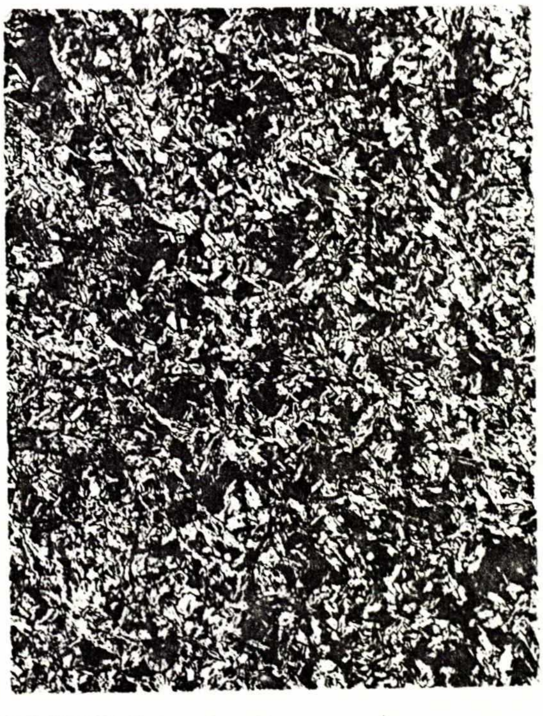


Fig. 4(b)

Fig. 5(a) Sample M-81 (Oval) - Photomicrograph of a thin section showing a microgranular aggregate of pyrophyllite and quartz with some saussurite (black) and zoisite (high relief). Note the large quartz grain in the lower left corner. Magnification 200 X, polarized transmitted light.

Fig. 5(b) - As in 5(a) with crossed polaroids.

Fig. 6(a) Sample M-81 (Oval) - Photomicrograph of a thin section showing a quartz-rich area (lower left), and a pyrophyllite-rich area (upper left) containing rounded aggregates of larger quartz grains. The remainder of the photograph is composed of a microgranular aggregate of pyrophyllite and quartz. Magnification 200 X, polarized transmitted light.

Fig. 6(b) - As in 6(a) with crossed polaroids.

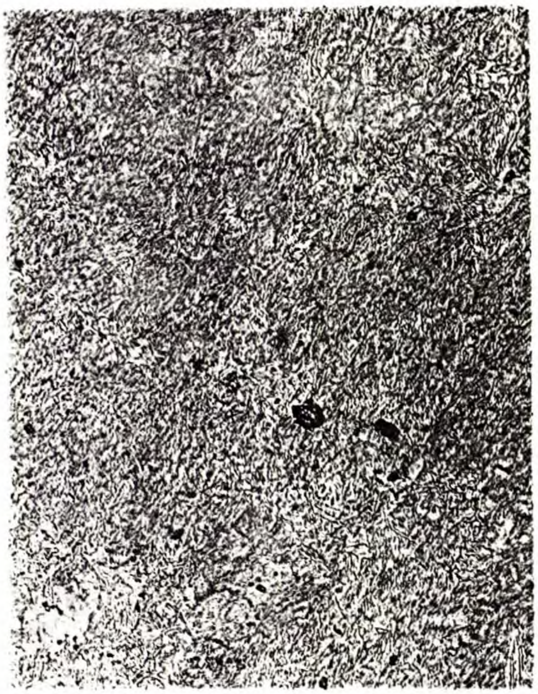


Fig. 5(a)

Fig. 5(b)

Fig. 6(a)

Fig. 6(b)

Fig. 7(a) Sample M-82 (Mine Hill) - Photomicrograph of a thin section showing a microgranular aggregate of pyrophyllite and muscovite with some saussurite (black). Magnification 200 X, polarized transmitted light.

Fig. 7(b) - As in 7(a) with crossed polaroids.

Fig. 8(a) Sample M-84 (Waste Rock) - Photograph of a polished section showing the fragmental structure of the rock. Magnification 1 X, oblique illumination.

Fig. 8(b) Sample M-84 (Waste Rock) - Photomicrograph of a thin section showing fragments of fine-grained quartz in a quartzose matrix, both of which are speckled by flakes of muscovite and dusty, red, iron oxides. Magnification 5 X, transmitted, unpolarized light.

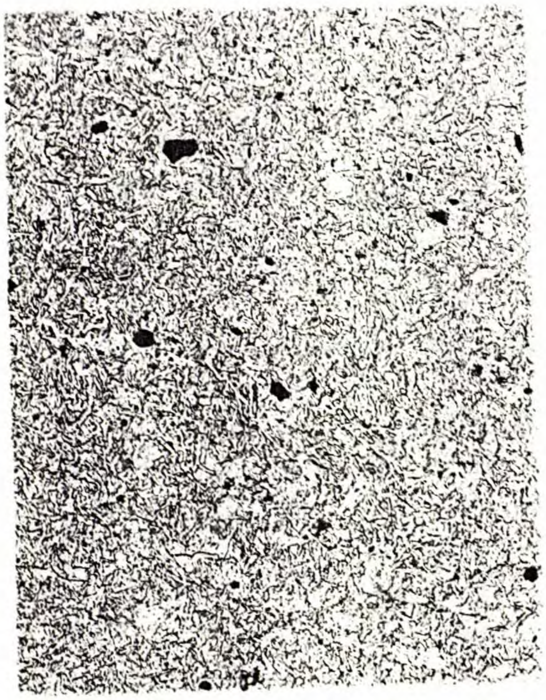


Fig. 7(a)

Fig. 7(b)

Fig. 8(a)

Fig. 8(b)

<u>Fig. 9(a) Sample M-84 (Waste Rock)</u> - Photomicrograph of a thin section showing the contact between a fine-grained quartzose fragment (top) and the coarser-grained quartzose matrix (bottom). Both are speckled by fine flakes of muscovite. A curved veinlet of muscovite or pyrophyllite is visible in the matrix. Magnification 60 X, transmitted, polarized light.

<u>Fig. 9(b)</u> - As in 9(a) with crossed polaroids.

<u>Fig. 10(a) Sample M-83 (Milled Product)</u> - Photomicrograph of +48 micron fragments in an oil immersion mount (n = 1.538) showing negligible liberation of the constituents. Magnification 200 X, polarized, transmitted. light.

<u>Fig. 10(b)</u> - As in 10(a) with crossed polaroids.

Fig. 9(a)

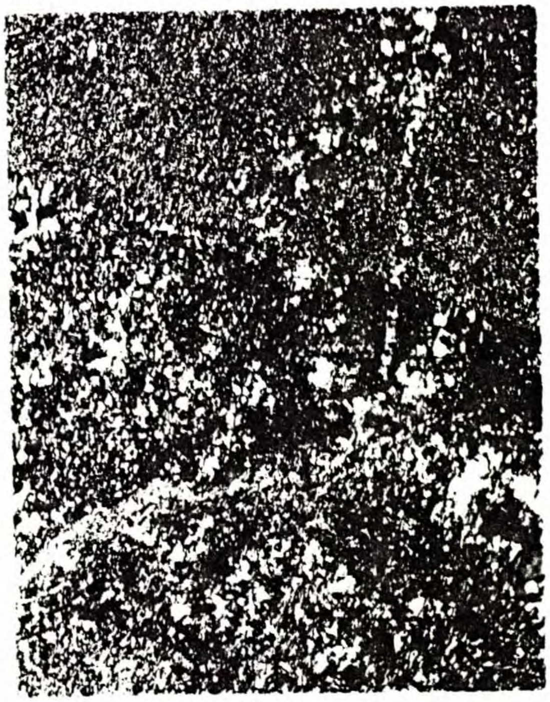


Fig. 9(b)

Fig. 10(a)

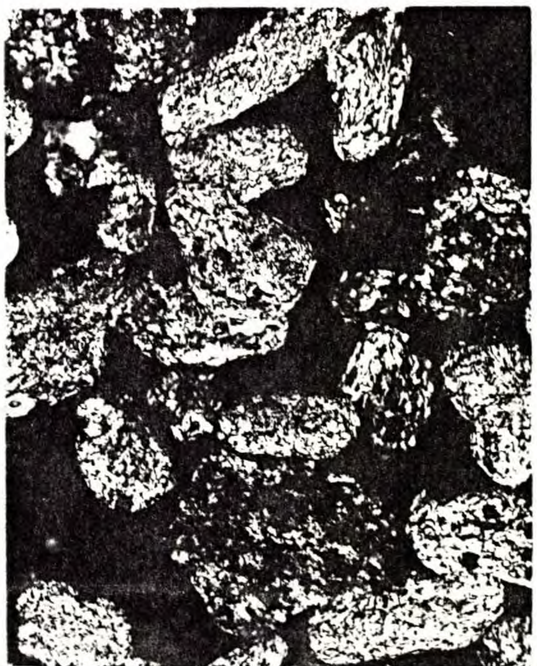


Fig. 10(b)

<u>Fig. 11(a) Sample M-83 (Milled Product)</u> - Photomicrograph of +24 - 48 micron fragments in oil immersion mount (n = 1.538) showing some liberation of constituents. Magnification 200 X, polarized transmitted light.

<u>Fig. 11(b)</u> - As in 11(a) with crossed polaroids.

<u>Fig. 12(a) Sample M-83 (Milled Product)</u> - Photomicrograph of +12 - 24 micron fragments in oil immersion mount (n = 1.538) showing considerable libration of constituents. Magnification 600 X, polarized transmitted light.

<u>Fig. 12(b)</u> - As in 12(a) with crossed polaroids.

Fig. 11(a)

Fig. 11(b)

Fig. 12(a)

Fig. 12(b)

Fig. 13(a) Sample M-83 (Milled Product) - Photomicrograph of +6 - 12 micron fragments in an oil immersion mount (n = 1.538) showing the highest degree of liberation of the constituents. Magnification 600 X; polarized, transmitted light.

Fig. 13(b) - As in 13(a), with crossed polaroids.

Fig. 14(a) Sample M-82 (Mine Hill) - Photomicrograph of a stained thin section with the coloured grains (medium relief) indicated by arrows; zoisite (high relief) and the other unstained material (low relief). Magnification 600 X; polarized, transmitted light.

Fig. 14(b) - As in 14(a), with crossed polaroids.

Fig. 13(a)

Fig. 13(b)

Fig. 14(a)

Fig. 14(b)

<u>Fig. 15(a) Sample M-92 (Mine Hill)</u> - Photomicrograph of a thin section showing stained grains (indicated by arrows) in an unstained aggregate. In this case the difference in relief is much less than in Fig.14. Magnification 600 X: polarized, transmitted light.

<u>Fig. 15(b)</u> - As in 15(a) with crossed polaroids.

<u>Fig. 16(a) Sample 88(c) 250' N57 Oval Quarry</u>
Photograph of polished section of fragment showing laminated, fragmental nature of the rock. Note the lenticular and contorted beds and the cross-bedding cut off by an unconformity. Magnification 1 X; oblique illumination.

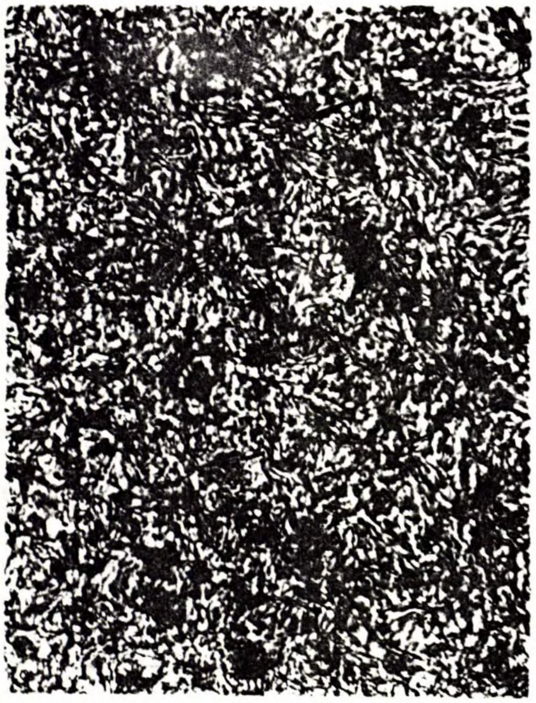


Fig. 15(a)

Fig. 15(b)

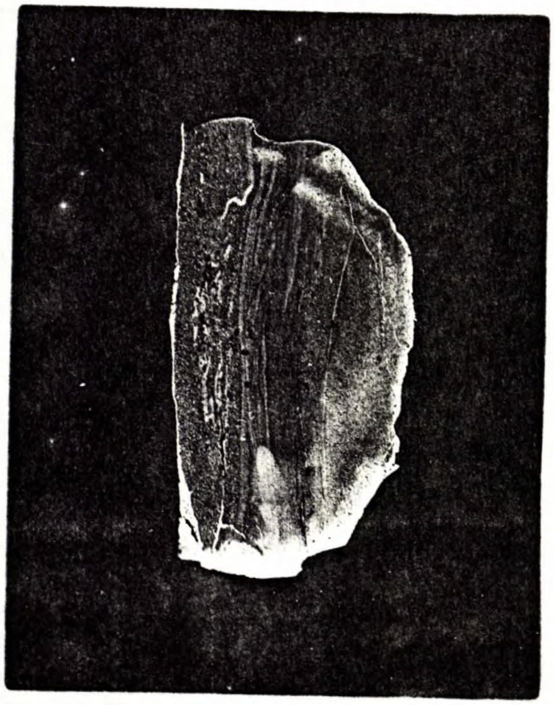
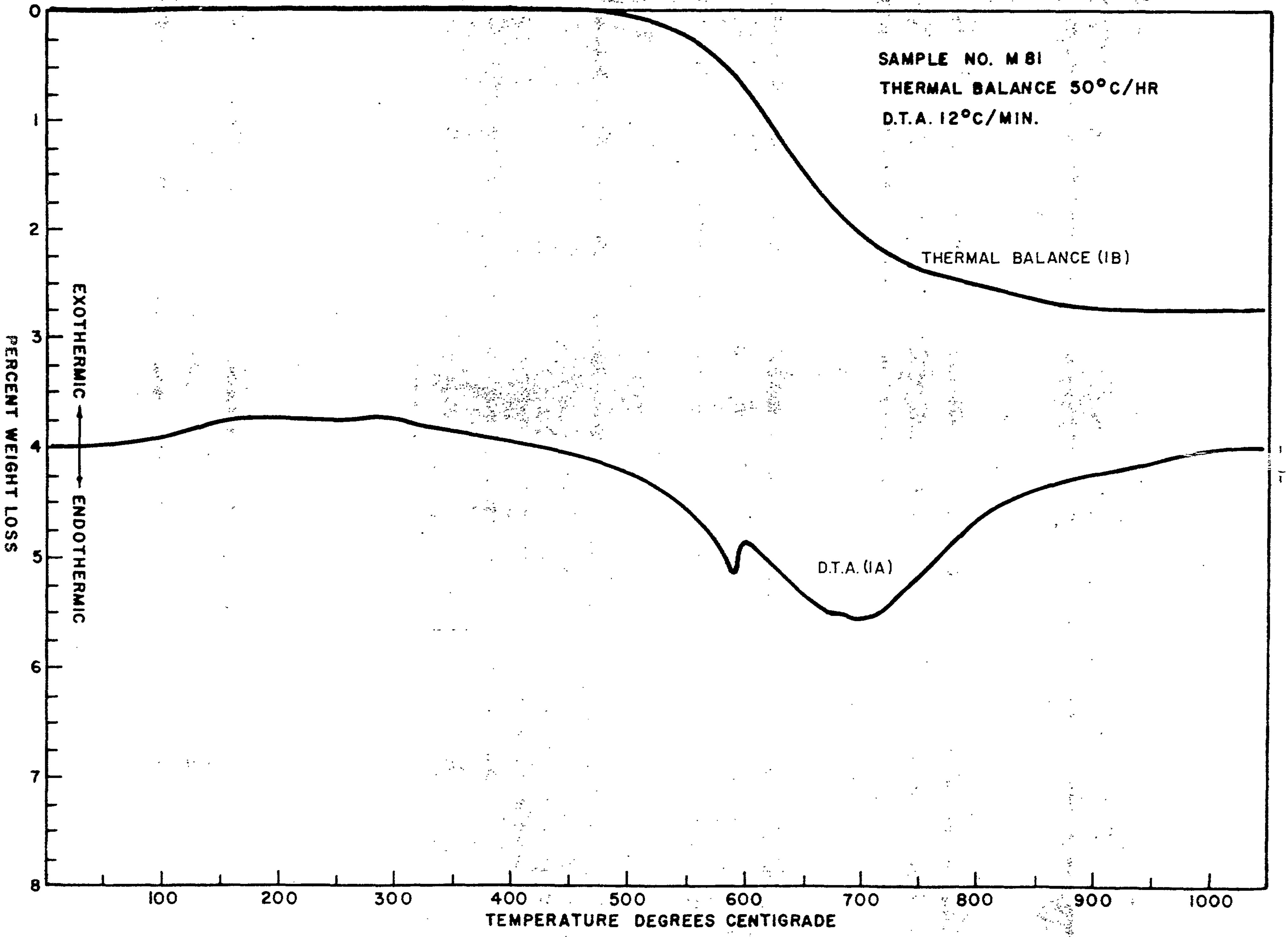
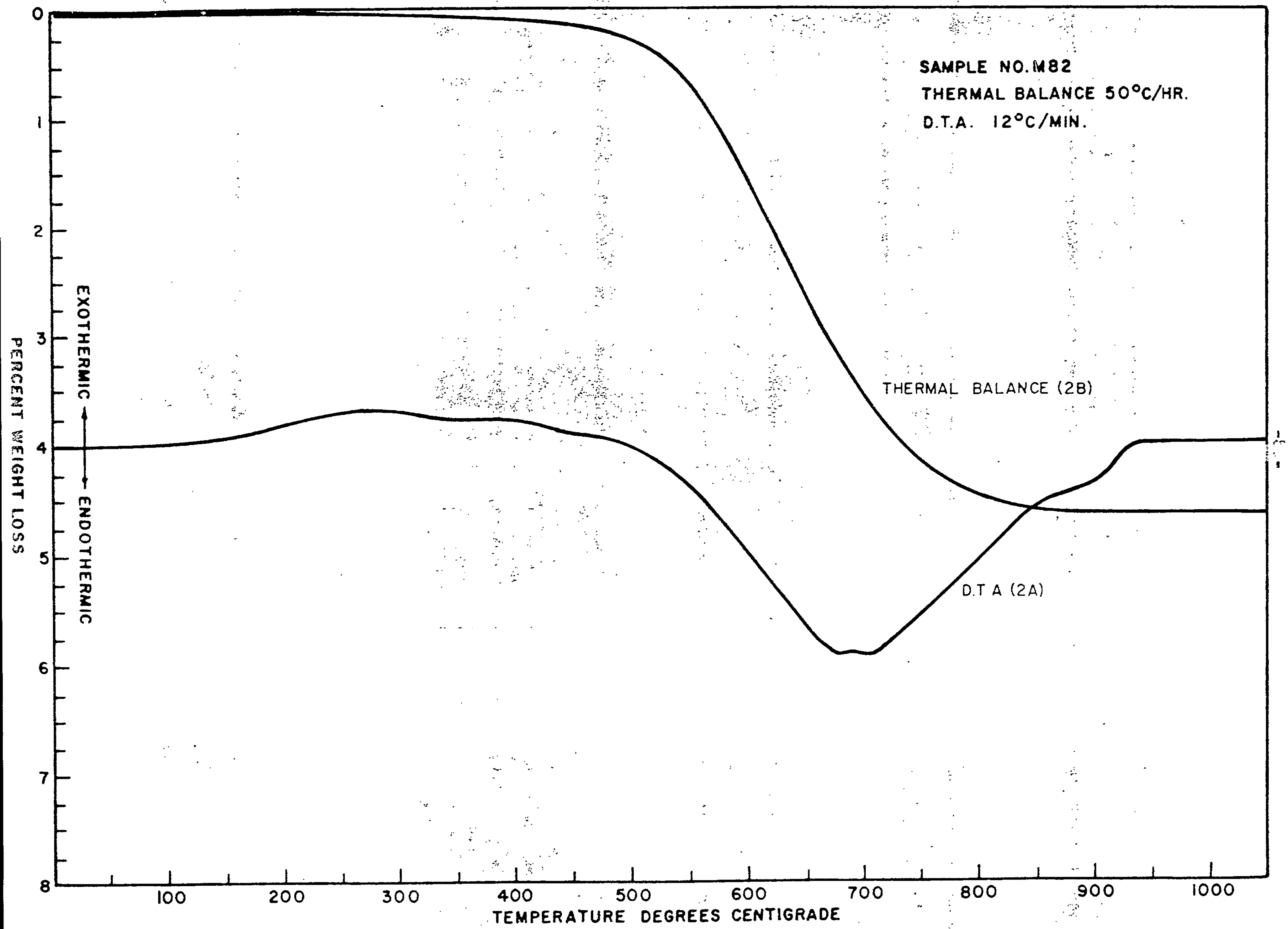


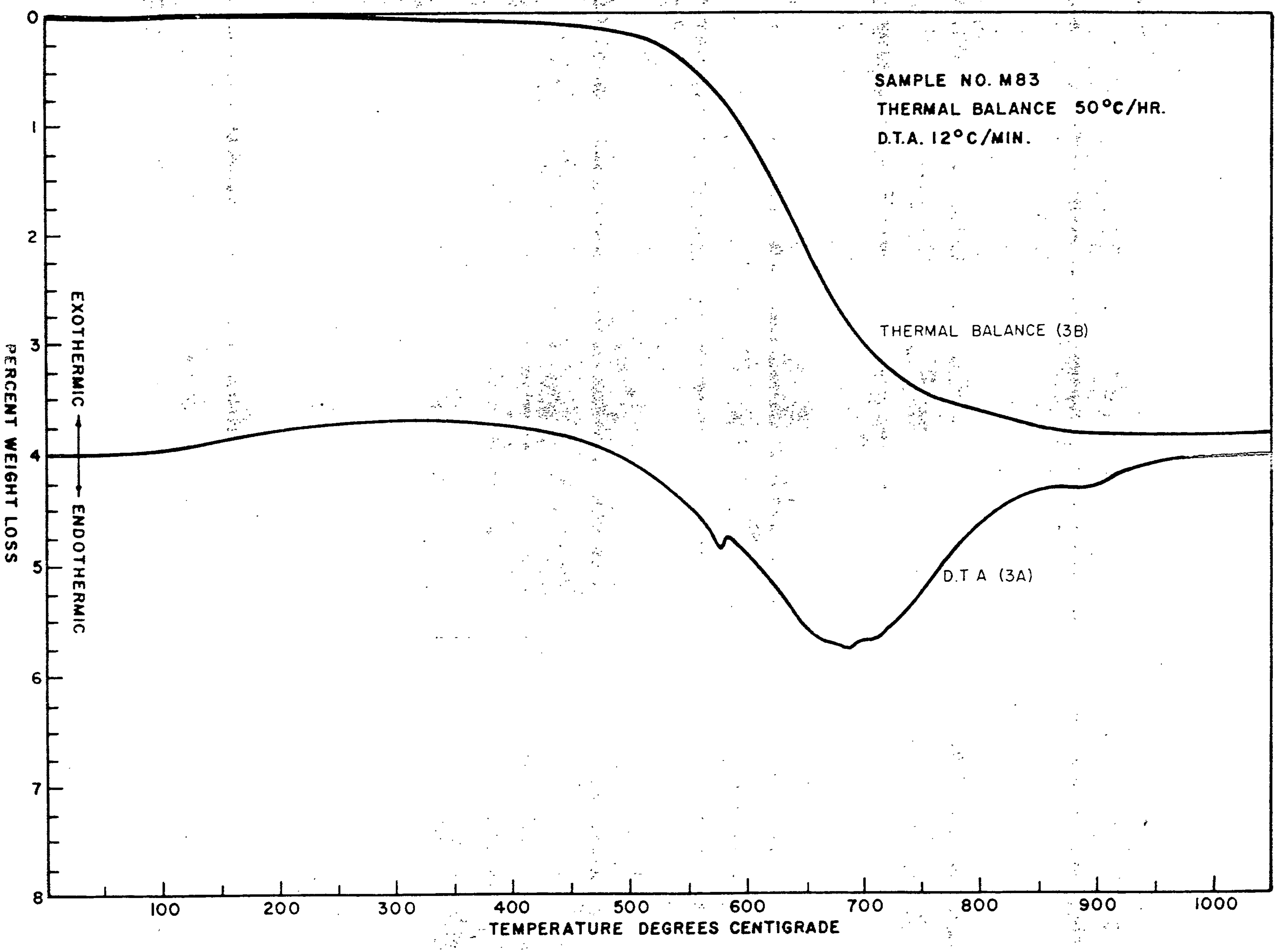
Fig. 16(a)



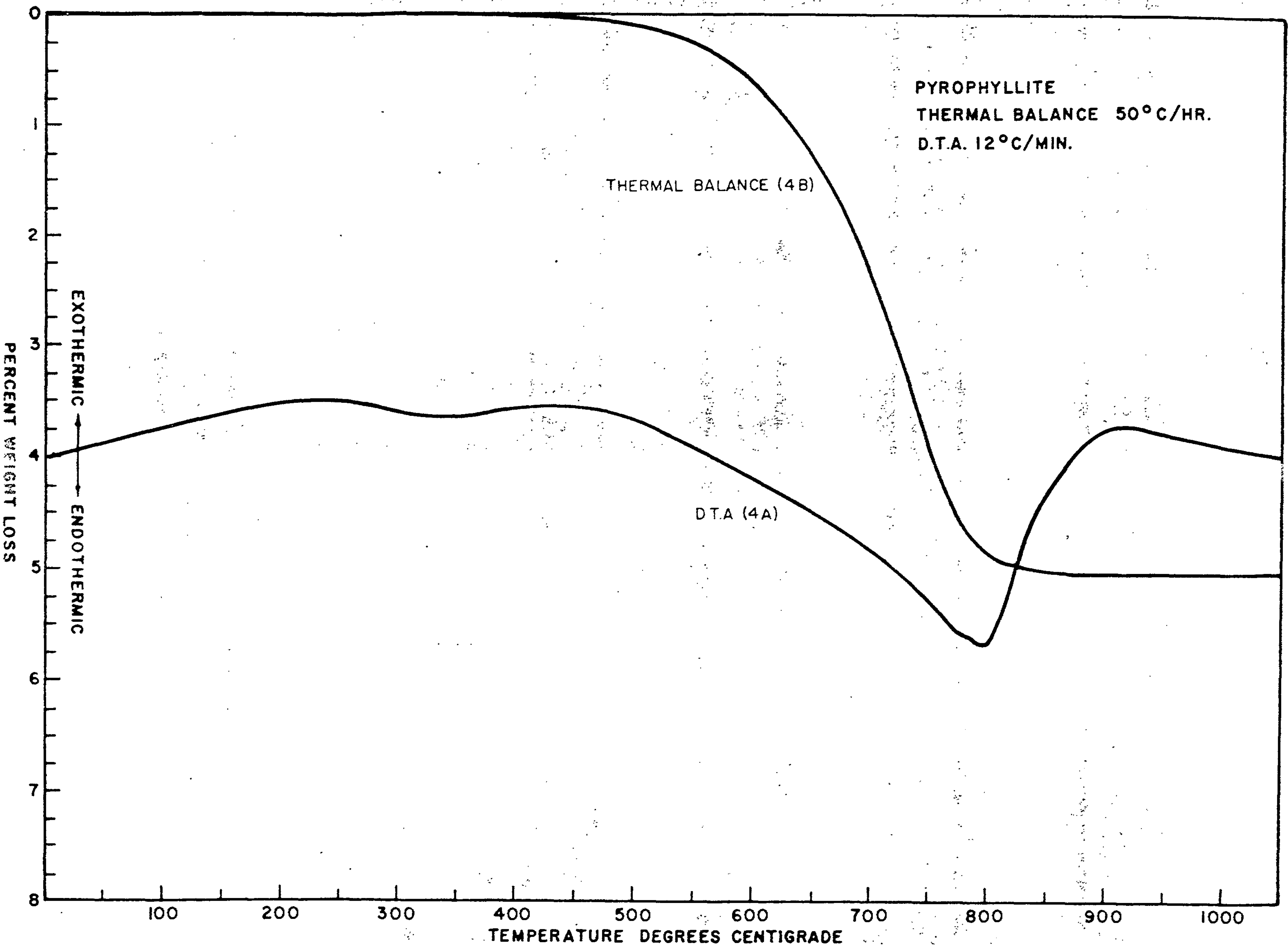
SAMPLE NO. M 81
THERMAL BALANCE 50°C/HR
D.T.A. 12°C/MIN.
THERMAL BALANCE (IB)
D.T.A. (IA)
EXOTHERMIC
ENDOTHERMIC
PERCENT WEIGHT LOSS
0
1
2
3
4
5
6
7
8
100
200
300
400
500
600
700
800
900
1000
TEMPERATURE DEGREES CENTIGRADE

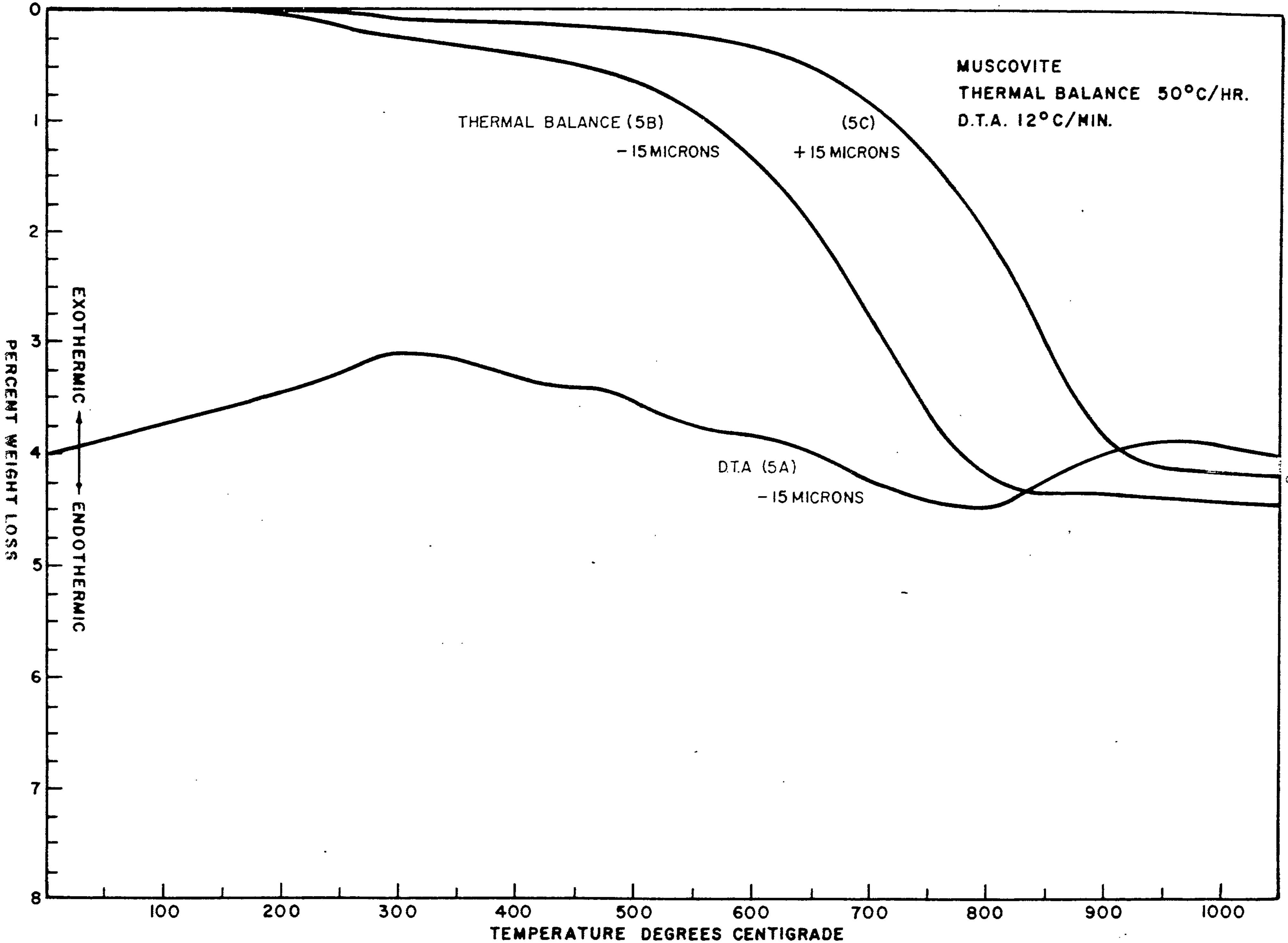


SAMPLE NO. M82
THERMAL BALANCE 50°C/HR.
D.T.A. 12°C/MIN.
THERMAL BALANCE (2B)
D.T A (2A)
EXOTHERMIC
ENDOTHERMIC
PERCENT WEIGHT LOSS
0
1
2
3
4
5
6
7
8
100
200
300
400
500
600
700
800
900
1000
TEMPERATURE DEGREES CENTIGRADE



SAMPLE NO. M83
THERMAL BALANCE 50°C/HR.
D.T.A. 12°C/MIN.
THERMAL BALANCE (3B)
D.T A (3A)
EXOTHERMIC
ENDOTHERMIC
PERCENT WEIGHT LOSS
TEMPERATURE DEGREES CENTIGRADE
0
1
2
3
4
5
6
7
8
100
200
300
400
500
600
700
800
900
1000


PYROPHYLLITE
THERMAL BALANCE 50°C/HR.
D.T.A. 12°C/MIN.
THERMAL BALANCE (4B)
D.T.A (4A)
EXOTHERMIC
ENDOTHERMIC
PERCENT WEIGHT LOSS
0
1
2
3
4
5
6
7
8
100
200
300
400
500
600
700
800
900
1000
TEMPERATURE DEGREES CENTIGRADE


MUSCOVITE
THERMAL BALANCE 50°C/HR.
D.T.A. 12°C/MIN.
THERMAL BALANCE (5B)
- 15 MICRONS
(5C)
+ 15 MICRONS
D.T.A (5A)
- 15 MICRONS
EXOTHERMIC
ENDOTHERMIC
PERCENT WEIGHT LOSS
0
1
2
3
4
5
6
7
8
100
200
300
400
500
600
700
800
900
1000
TEMPERATURE DEGREES CENTIGRADE