M & R
LS/6/58
4010

STATE OF CALIFORNIA
DEPARTMENT OF PUBLIC WORKS
DIVISION OF HIGHWAYS

REPORT
on
DIFFERENTIAL THERMAL ANALYSIS
AS APPLIED TO
HIGHWAY ENGINEERING MATERIALS

58-23

June 1958

58-23
LIBRARY COPY
Materials & Research Dept.

ClibPDF - www.fastio.com

State of California
Department of Public Works
Division of Highways
Materials and Research Department

June 30, 1958

Mr. F. N. Hveem
Materials & Research Engineer
Division of Highways
Sacramento, California

Dear Sir:

Submitted for your consideration is:

REPORT

on

DIFFERENTIAL THERMAL ANALYSIS

AS APPLIED

to

HIGHWAY ENGINEERING MATERIALS

Study made by.....Foundation Section
Under general direction of.....A. W. Root
Work supervised by.....E. D. Drew
Report prepared by.....Carl Setzer, Jr.

Very truly yours,

A. W. Root
Supv. Materials & Research Engr.

Attach.

ClibPDF - www.fastio.com

TABLE OF CONTENTS

	Page
The Clay Mineral Concept	1
Methods of Clay Mineral Determination	1
Theory & Method of Differential Thermal Analysis	2
Clay Minerals	3
Groups	3
Molecular Structural Units	5
Kaolinites	5
Montmorillonites	5
Illites	6
Mixed Layer Clays	6
Differential Thermal Characteristics of Clay Minerals	6
Kaolinites	6
Montmorillonites	7
Illites	7
Thermal Characteristics of Some Non Clay Minerals	7
Quartz	7
Carbonates	8
Limonite	8
Gypsum	8
Organic Matter	8
Clay Minerals Found in Soils	8

ClibPDF - www.fastio.com

	Page
Differential Thermal Analysis Equipment	10
Furnace	10
Sample Holder	10
Recording Equipment	11
Program Control Unit	11
Power Supply Unit	12
Thermograms of Selected California Soil Samples	13
District I	13
District II	13
District III	14
District IV	15
District V	16
District VI	18
District VII	19
District VIII	19
District X	19
District XI	20
Thermograms of Suites of Soil Samples	21
Westside Freeway	21
X-SJ-4-C	22
Contra Costa County	22
Quantitative Determinations of Clay Minerals	25
Conclusions	25
Bibliography	27

ClibPDF - www.fastio.com

LIST OF ILLUSTRATIONS

Figure No.	Title	Follows Page
1	Basic Clay Mineral Structural Units	5
2	Structure of Silica Tetrahedrons	5
3	Kaolinite Structure	5
4	Montmorillonite Structure	5
5	Illite Structure	6
6	Diagrammatic Representation of Temperature - Differential Temperature Recording System	12
7	Thermograms of Material from Districts I & II	13
8, 9 & 10	Thermograms of Material from District III	14
11 & 12	Thermograms of Material from District IV	15
13 & 14	Thermograms of Material from District V	16
15	Thermograms of Material from District VI	18
16	Thermograms of Material from District VII & VIII	19
17	Thermograms of Material from District X & XI	20
18 to 26	Thermograms of Soil Samples from Westside Freeway VI-Fre-238-A,B	21
27	Magnesium Waste Material X-SJ-4-E	22
28 & 29	Piezometer Borings Contra Costa County	24
30 & 31	Reference Chart of Clay Minerals	24

ClibPDF - www.fastio.com

Synopsis

Determinations of the clay mineral content of soils used in highway construction projects by means of differential thermal analysis have been made by the Materials and Research Department since 1955. This report presents data on the equipment used in the differential thermal analysis process, and a summary of the theoretical background, data on some of the soil samples tested, and sample preparation techniques.

Introduction

The suitability of a soil for engineering purposes is dependent on its ability to remain in place, and to support loads. In most situations, clays are detrimental to stability, and it is necessary for the engineer involved in earthwork construction to know the nature of the clay minerals present and their influence on the engineering properties of the soil.

While the study of clay mineralogy is not a new field, it has been only in the past 20 years since the acceptance of the clay mineral concept, and the recognition that clay minerals are composed of extremely small particles of a limited number of crystalline minerals that progress has been made in the characterization of clay minerals.

The Clay-mineral Concept

The clay-mineral concept holds that clays are essentially composed of extremely small crystalline particles of one or more members of a small group of minerals known as clay minerals. Clay minerals are essentially hydrous aluminum silicates, with magnesium or iron proxying wholly or in part for the aluminum in some minerals, and with alkalies or alkaline earths present as an essential constituent in some of them. Some clays are composed of a single clay mineral, but in many of them, there is a mixture of several clay minerals. These crystalline clay minerals are the essential components of nearly all clays, and therefore are most important in determining the properties of soils. In addition some clay materials contain varying amounts of non-clay materials such as quartz, calcite, gypsum, and pyrite as well as organic matter and water soluable salts.

Methods of Clay Mineral Determination

There are several methods for determination of the clay mineral type such as X-ray diffraction, petrographic microscopy, electron microscopy, and chemical analysis, but the most rapid and practical method for soils engineering is that of differential thermal analysis. The differential thermal analysis method is based on the fact that the application of

ClibPDF - www.fastio.com

heat to many minerals causes certain physical and chemical changes that are reflected in endothermic and exothermic reactions. By comparing the change of a thermally inert substance such as gamma aluminum oxide heated under the same conditions, a curve or pattern is obtained which is characteristic of the particular mineral under examination.

The use of a double hot-junction thermocouple to the study of changes which take place in minerals when they are heated was first made by Le Chatelier in 1887. Not much interest or use of this method occurred until the 1930's when American and French workers in the field of clay mineralogy started to experiment with, and use thermal analysis. However, much difficulty in obtaining reproducible curves was encountered, and the great progress in the field of differential thermal analysis did not occur until the post war developments in electrical instrumentation.

Theory and Method of Differential Thermal Analysis

Substances undergo exothermic and endothermic changes upon the application of heat. No two minerals have chemical bonds of identical strength, and they decompose, oxidize, and change phase at different temperatures. Differential thermal analysis determines, through use of suitable instrumentation, the temperature at which reactions take place in a substance when heated continuously to an elevated temperature, and the intensity and character of the reaction. The results are plotted as a continuous curve in which the thermal reactions are plotted against furnace temperatures.

The sample to be analyzed is placed in one hole of a specimen holder and an inert material, usually calcined gamma aluminum oxide, is placed in the other hole of the specimen holder. One junction of the differential thermocouple is placed in the center of the sample and the other junction in the center of the inert material. The holder and thermocouples are placed in a furnace so controlled as to produce a uniform rate of temperature increase. The temperature of the inert material increases regularly as the temperature of the furnace increases. When a thermal reaction occurs in the sample, the temperature of the sample is greater or less than that of the inert material for an interval of time until the reaction is completed, and the temperature of the sample again comes to that of the furnace. During this time interval, the temperature of one junction of the thermocouple is different from that of the other, and an electromotive force is set up in the differential thermocouple circuit which is recorded automatically by the X-Y recorder. When no thermal reaction is occurring in the sample, the temperatures at both junctions of the difference couple are the same, and no electromotive force is set up. The direction of the electromotive force in the circuit is dependent on whether the temperature of the sample is above or below that of the inert material. The thermal curve is

ClibPDF - www.fastio.com

a differential function, and it is dependent on those effects which do not occur simultaneously and equally in the specimen and inert material. This is accomplished by maintaining, as nearly as possible, identical geometrical spacing of the inert and sample holes to the furnace axis.

Clay Minerals

Clay is any material finer than two microns in size. It may be of any mineralogic composition. Usual engineering usage of the term "clay" implies a natural, earthy fine-grained material developing plasticity when mixed with a limited amount of water. This physical characteristic is due to the presence of clay mineral or minerals. A clay mineral is a substance composed of essentially small crystalline particles. Chemically, they are hydrous aluminum silicates, with magnesium or iron proxying wholly or in part for the aluminum in some minerals and with alkalies or alkaline earths present as essential constituents in some of them. Some clays are composed of a single mineral, but many are a mixture of several. Quartz, calcite, feldspar, and pyrite, non-clay minerals, are frequently present in the clay sized fraction of soils. Organic matter and water soluble salts are also present.

Clay Mineral Groups

The usual method of clay mineral classification is based on the structural attributes. The following table serves as a basis for the discussion of clay minerals.

Clay minerals may be divided into three main categories: (1) micaceous or platy clay minerals, (2) fibrous clay minerals, and (3) amorphous clay minerals. The latter two groups have little interest to the soils engineer and are mentioned only in passing.

The micaceous or platy clay minerals are divided into four classes: (1) the kaolinite group, (2) the montmorillonite group (3) the illite group and (4) the mixed layer group. Each of these groups, except the fourth, is characterized by distinct molecular structure and chemical bonding.

ClibPDF - www.fastio.com

(From Clay Mineralogy, R. E. Grim, McGraw-Hill, 1953)

I Amorphous

Allophane group

II Crystalline

A. Two layer type (sheet structures composed of units of one layer of silica tetrahedrons and one layer of alumina octahedrons

1. Equidimensional

Kaolinite group

a. Kaolin, Nacrite, etc.

2. Elongate

a. Halloysite group

B. Three-layer types (sheet structures composed of two layers of silica tetrahedrons and one central dioctahedral or trioctahedral layer)

1. Expanding lattice

a. Equidimensional

Montmorillonite group

Montmorillonite, Sauconite, etc.

Vermiculate

b. Elongate

Montmorillonite group

Nontronite, Saponite, Hectorite

2. Nonexpanding lattice

Illite group

C. Regular mixed-layer types (ordered stacking of alternate layers of different types

Chlorite group

D. Chain-structure types (hornblende-like chains of silica tetrahedrons linked together by octahedral groups of oxygens and hydroxyls

ClibPDF - www.fastio.com

containing Al and Mg atoms)

Attapulgite
Sepiolite
Palygorskite

Molecular Structural Units of Clay Minerals

The two structural units involved in the atomic lattices of most clay minerals are the octahedron and the silica tetrahedron. The octahedron consists of two sheets of closely packed oxygens or hydroxyls in which aluminum, iron or magnesium atoms are embedded in octahedral coordination so they are equidistant from six oxygens or hydroxyls. (See Figure 1). The second unit is built of silica tetrahedrons. In each tetrahedron, a silicon atom is equidistant from four oxygens or hydroxyls arranged in the form of a tetrahedron with a silicon atom at the center. The silica tetrahedral groups are arranged to form a hexagonal network. The tetrahedrons are arranged so that the tips of all point in the same direction, and the bases are in the same plane. (See Fig. 2).

The Kaolinite Group

The kaolinite minerals have a structure composed of a single silica tetrahedral sheet and a single alumina octahedral sheet combined in a unit so that the tips of the silica tetrahedrons and one of the layers of the octahedral sheet form a common layer. (See Fig. 3). All the tips of the silica tetrahedrons point in the same direction and toward the center of the unit made of the silica and octahedral sheets. The minerals of the kaolinite group consist of sheet units of the type described continuous in the a and b directions and stacked one above the other in the c direction. The variation between members of this group consists in the way in which unit layers are stacked above each other, and possibly in the position of the aluminum atoms in the possible positions open to them in the octahedral layer.

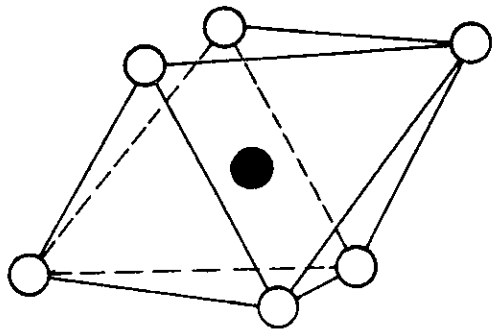
The chemical formula for the group is $Al_2O_3 \cdot 2SiO_2 \cdot nH_2O$. The usual grouping is kaolin, nacrite and dickite which have a chemical formula of $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$ and halloysite which has a chemical formula of $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$ and $Al_2O_3 \cdot 2SiO_2 \cdot 4H_2O$.

The Montmorillonite Group

The montmorillonite minerals occur in extremely small particles. Montmorillonite is made up of two silica tetrahedral sheets with a central alumina octahedral sheet. All the tips of the tetrahedrons point in the same direction and toward the center of the unit. The tetrahedral and octahedral sheets are combined so that the tips of the

ClibPDF - www.fastio.com

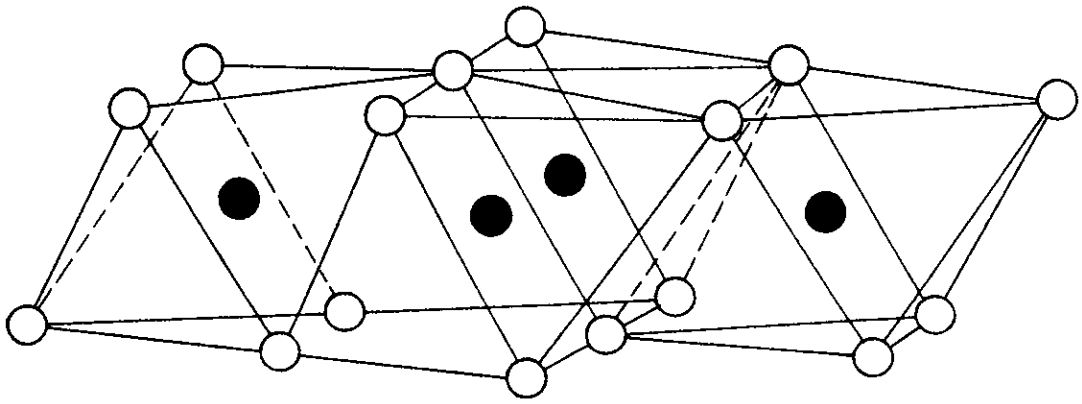
BASIC CLAY MINERAL STRUCTURAL UNITS



SINGLE OCTAHEDRAL UNIT

○ Hydroxyls

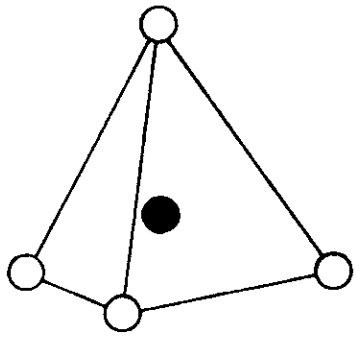
● Aluminums, Magnesiums, Etc.



SHEET STRUCTURE OF OCTAHEDRAL UNITS
AFTER GRIM, CLAY MINERALOGY

Figure 1

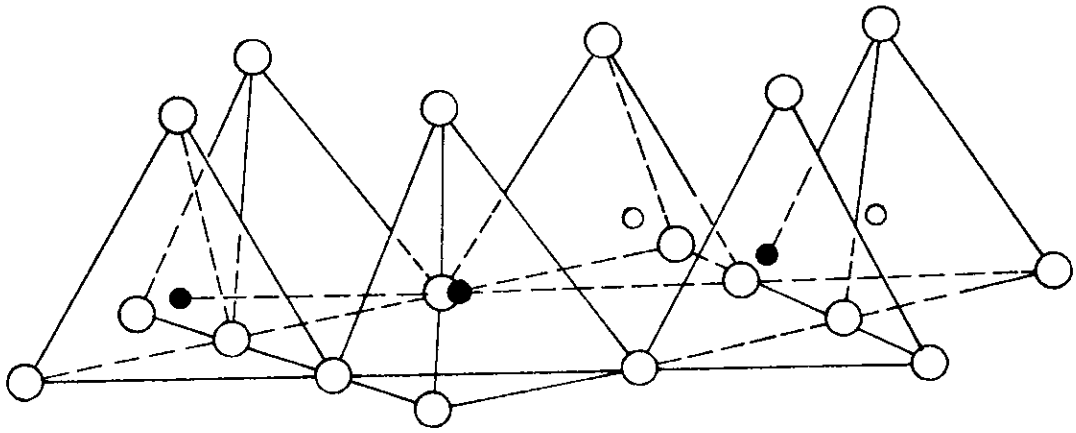
ClibPDF - www.fastio.com



SINGLE SILICA TETRAHEDRON

○ Oxygens

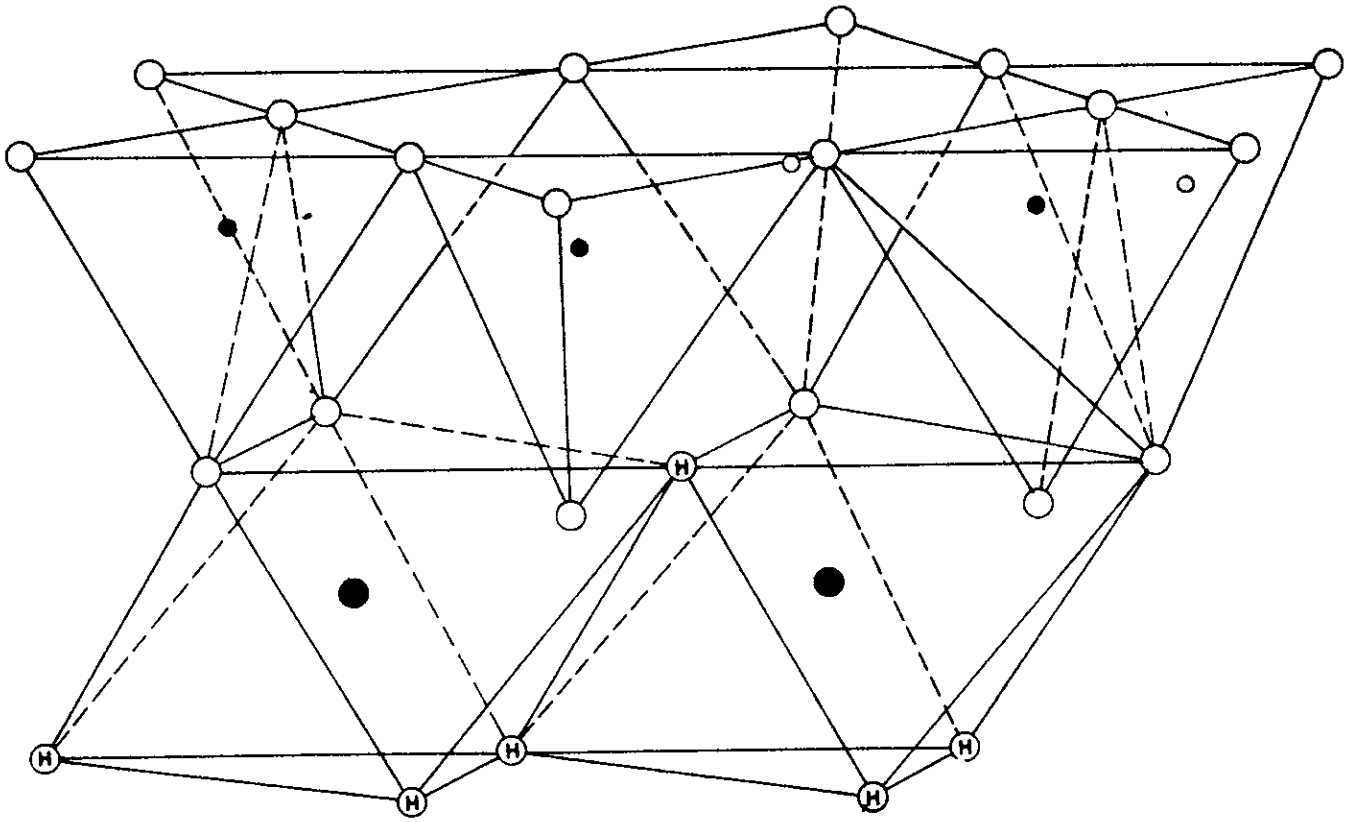
○ & ● Silicons



SHEET STRUCTURE OF SILICA TETRAHEDRONS

Figure 2

ClibPDF - www.fastio.com



KAOLINITE STRUCTURE

Ⓗ Hydroxyls

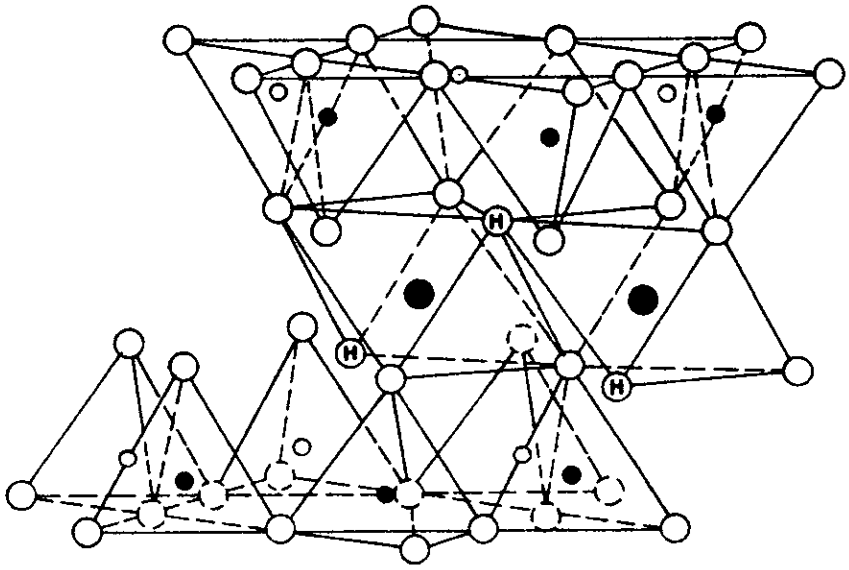
○ Oxygens

● Aluminums

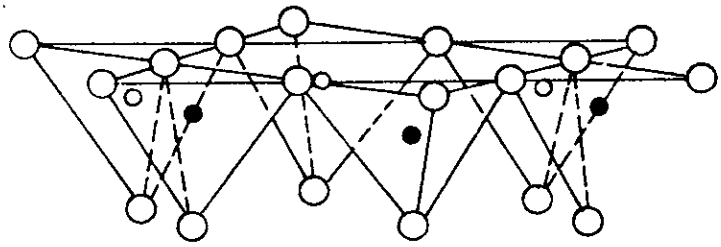
○ & ● Silicons

Figure 3

ClibPDF - www.fastio.com



EXCHANGEABLE CATIONS $_nH_2O$



MONTMORILLONITE STRUCTURE

○ Oxygens

Ⓗ Hydroxyls

● Aluminum

○ & ● Silicon & Occasionally Aluminum

Figure 4

ClibPDF - www.fastio.com

tetrahedrons of each silica sheet and one of hydroxyl layers of the octahedral sheet form a common layer (See Fig. 4). The layers are continuous in the a and b directions, and are stacked one above the other in the c direction. In the stacking of the silica-alumina-silica units, oxygen layers of each unit are adjacent to oxygen layers of the neighboring units with the consequence that there is a very weak bond, and an excellent cleavage between them. Water can enter between the unit layers causing the lattice to expand in the c direction.

The Illite Group

The illitic clay minerals have a basic structural unit consisting of a layer composed of two silica tetrahedral sheets with a central octahedral sheet. The tips of the tetrahedrons in each silica sheet point toward the center of the unit and are combined with the octahedral sheet in a single layer with suitable replacement of the hydroxyl ion by oxygen. The unit is the same as for montmorillonites except that some of the silicons are always replaced by aluminums and the resultant charge deficiency is balanced by potassium ions. The unit layers extend indefinitely in the a and b directions and are stacked in the c direction. (See Fig. 5).

Mixed Layer Clays

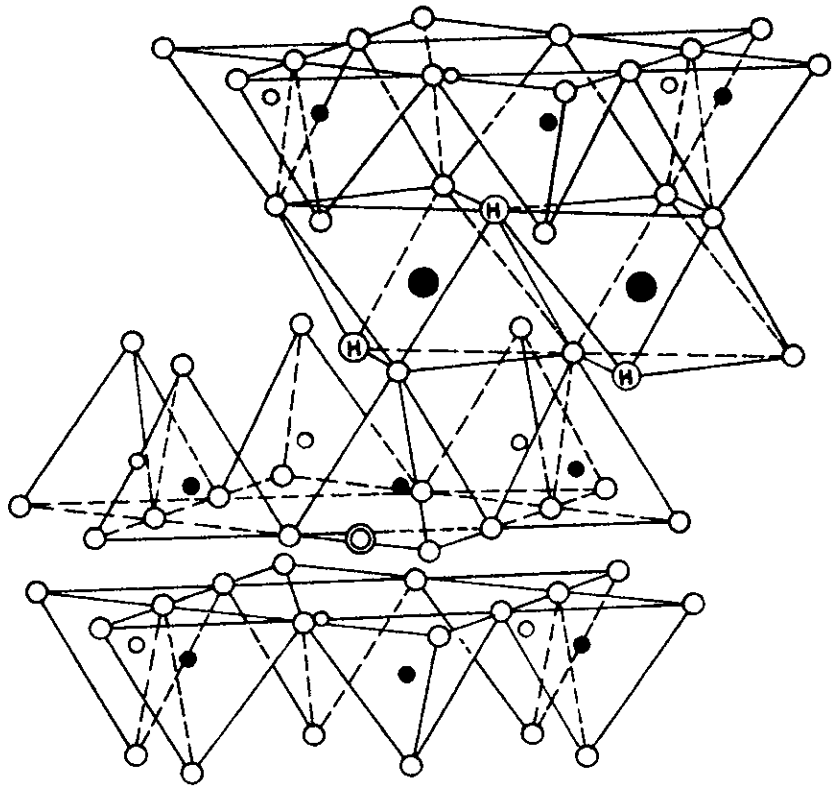
Many clays are composed of more than one clay mineral. The mixture may occur in several ways. It may be one of discrete clay-mineral particles with no preferred geometric orientation of one particle with respect to its neighboring clay-mineral particles. Another type of mixture is the inter-stratification of the layer clay minerals in which individual layers are of the order of a single or a few aluminosilicate sheets. Another kind of mixed-layer structure is one with random irregular interstratification of layers with no uniform repetition of layers.

Differential Thermal Characterisitics of Clay Minerals

Kaolinites

The kaolinite group of clay minerals, kaolinite, dickite, and nacrite, differ structurally only in the manner of stacking of sheets. Water is not adsorbed between the sheets of these minerals since there is relatively strong hydrogen bonding between any two adjacent sheets. The thermal reactions of the kaolin minerals reflect the structure. The only peak given by kaolinitic clay minerals below 500°C is the adsorbed water peak about 150°C. Impurities in the clay mineral may cause irregularities in the 100° - 200°C range. The major endothermic peak of the kaolin family occurs between approximately 600° and 700°C. It is produced by the decomposition of the lattice to form amorphous alumina. All kaolinitic

ClibPDF - www.fastio.com



ILLITE STRUCTURE

○ Oxygens

Ⓗ Hydroxyls

● Aluminum

◎ Potassium

○ & ● Silicons

Figure 5

ClibPDF - www.fastio.com

minerals show an exothermic peak at about 980°C. This peak is usually sharp and results from the recrystallization of amphorous alumina to form gamma alumina. Poorly crystallized or interlayed kaolinites do not show the sharp exothermic peak as do well crystallized kaolinites. There is also a tendency for the exothermic peak to be lowered in impure or poorly crystalline materials.

Montmorillonites

Thermal curves of the montmorillonite group have a number of common features. The first is an endothermic peak or peaks occurring between 100°C. and 250°C. These peaks are mainly dependent on the humidity in which the sample was maintained before heating. Final destruction of the montmorillonite lattice begins about 600°C., and is characterized by an endothermic peak. The decomposition is due to loss of the hydroxyl groups in the form of water. The decomposition reaches a maximum about 700°C. It various somewhat, depending on the type of montmorillonite. Nontronites have a decomposition temperature 100° to 200° lower, and hectorite has a decomposition temperature some 100° higher. This is related directly to the strength of the chemical bonds with the hydroxyl. An increase in ferric ions seems to lower the decomposition temperature. The third endothermic peak in the montmorillonite group corresponds to the final breakdown of the montmorillonite lattice and characterizes the group. It occurs about 900° C., and is followed by a sharp exothermic effect which varies in shape and temperature. The final exothermic peak is attributed to the formation of spinel, and largely determined by the chemical composition of the octahedral layer. In nontronites, with high iron contents, this occurs at a lower temperature due to the weakness of the iron-hydroxyl bond.

Illite Group

Thermal curves of illite clay minerals show an initial endothermic reaction caused by loss of interlayer water between 100°C. and 250°C. A second endothermic peak occurs between the temperatures of 350°C. and 600°C. It is caused by the loss of hydroxyl water. The illite lattice is completely destroyed about 800°C. - 850°C. After the destruction of the lattice around 850°C., spinel crystallizes, and grows in particle size up to 1100°C. The amplitude of the third endothermic peak is indicative somewhat of the chemical composition. The final reaction usually gives a characteristic endothermic - exothermic couple which is typical of illite.

Thermal Characteristics of some Non-Clay Minerals

Quartz

A thermal curve of quartz is characterized by a sharp endothermic reaction at 573°C. At this temperature, quartz undergoes a structural change from the alpha state

ClibPDF - www.fastio.com

(Rhombohedral-trapezohedral tetartohedral) to the beta state (Rhombohedral-trapezohedral hemihedral). When cooled, quartz inverts, with a characteristic exothermic peak.

Carbonate Group

All members of the carbonate group display characteristic thermal reactions when heated. The chemical composition strength of bonding and state of crystallization are indicated by the varying temperature at which the reactions occur.

Calcite is characterized by one sharp endothermic peak starting about 900°C. The size of the peak varies, depending on the concentration of calcite. Dolomite is characterized by two endothermic peaks: one occurring between 750°C. - 800°C., and the other between 900°C. - 950°C.

Siderite ($FeCO_3$) decomposes about 550°C. with a sharp endothermic peak followed by a sharp exothermic peak. Dilution of the caronates tends to lower the temperature at which the reaction occurs.

Limonite

Limonite, $2Fe_2O_33H_2O$, is a common constituent of soils, and frequently occurs with clay minerals. Its thermal curve is characterized by an endothermic reaction occurring between 300°C. and 400°C.

Gypsum

Gypsum ($Ca\ SO_4 \cdot 2H_2O$) is a commonly occurring mineral in certain soil types. Its thermal curve is characterized by an endothermic reaction occurring between 100°C - 250°C.

Organic Matter

Organic matter decomposes in two stages. The first stage is marked by an exothermic peak at about 300°C.; this results from the combustion of the organic matter, and the formation of carbon. In some instances, another exothermic peak occurs at about 500°C., which is caused by the oxidation of the carbon previously formed.

Clay Minerals Found in Soils

The clay mineral component of a soil is determined by (1) climatic conditions, (2) topography, (3) bedrock type and (4) geological history.

A basic igneous rock (diabase, diorite, gabbro) can form either a montmorillonitic clay mineral or a kaolinitic clay mineral when it weathers. If the drainage is poor, or rainfall low, magnesium will remain in the weathering zone after breakdown of the parent rock, and result in the formation of a montmorillonitic clay mineral. High rainfall and

ClibPDF - www.fastio.com

good drainage will result in removal of the magnesium, and the formation of a kaolinitic clay mineral. Under conditions of relatively rapid leaching, a montmorillonite may form in the initial stage, and kaolinite in a later stage. Long continued weathering can result in the breakdown of all clay minerals, and production of a silica rich soil containing no aluminum or iron.

An acid igneous rock (granite, rhyolite, granodiorite) will yield illite and montmorillonite when weathered under conditions permitting potash and magnesia to remain in the soil zone. Low magnesium content will result in formation of an illite; low potash content will result in formation of a montmorillonite. Kaolinite clay minerals result from rapid removal of potash and magnesia.

The clay mineral components present in calcareous sediments are illites and montmorillonites. When calcareous sediments are weathered, there is no alteration of the silicates until all the carbonate is broken down, and calcium removed from the environment.

Studies of recent marine sediments show that illitic clay minerals are common constituents. The climatic conditions of California, especially in the Central Valley, and Coastal Areas seem to favor development of illitic clay minerals.

ClibPDF - www.fastio.com

DIFFERENTIAL THERMAL ANALYSIS EQUIPMENT

The differential thermal analysis apparatus used in the Materials and Research Department is comprised of five units:

1. Furnace and sample holding equipment
2. Preamplification unit
3. Temperature - differential temperature recording unit
4. Program control unit
5. Power supply unit

Furnace

Two identical furnaces are used. They are Lindberg Model CF-1 electrical resistance furnaces. Each is constructed so as to heat a combustion chamber 15" in length and 6" in diameter from room temperature to 1600°C. Heating is provided by resistance type Globar elements spaced around the heating chamber. The current to the heating elements is controlled by a transformer in the base of the furnace unit. The temperature of the heating chamber is indicated by a platinum-platinum 10% rhodium thermocouple attached to an indicating pyrometer.

The furnaces are mounted on flanged wheels and are slid over the sample holder.

Sample Holder

The samples to be tested are held in a nickel sample holder having dimensions 1-3/4" x 1" x 3/4". The sample holder contains two wells, 5/16" wide and 1/2" deep for the unknown material and the inert material and each well is tapped with a 1/8" diameter hole for insertion of the thermocouple. Midway between the two sample wells is a third hole for the reference thermocouple.

The differential thermocouple is made of B & S 28 gauge chrome-chrome alumel wire, and the reference thermocouple is made of B & S gauge platinum-platinum 10% rhodium wire. The thermocouples are insulated by using alundum spaghetti. The differential thermocouple circuit is equipped with two plastic molded tabular condensers to filter stray currents and the leads are covered with metallic shielding which is grounded to remove stray currents.

The sample block is held in the oven by an alundum tube which is fitted into a sleeve and held upright by an aluminum channel beam.

ClibPDF - www.fastio.com

Stabilized D-C Amplifier

The signal from the differential thermocouple is of low intensity, and it is necessary for it to be amplified. By use of the Leeds and Northrup stabilized direct current amplifier, the direct current from the thermocouple is changed to an alternating current by a contact type synchronous input converter. The alternating current is amplified and changed to direct current by an output converter synchronized with the input converter, filtered and fed into the speedomax.

Temperature-Differential Temperature Recording Unit

The Leeds & Northrup speedomax Model G X-Y recorder contains two standard speedomax electrical balancing systems; one, recording differential temperature on a horizontal axis, and one recording furnace temperature on a vertical axis. The pen moves in a horizontal direction recording variations in temperature differential, as the chart is moved vertically by a reversing motor whose action is controlled by the rate of furnace temperature increase.

The two balancing systems operate in the same manner. From an initial condition of balance in the direct current potentiometer circuits (thermocouples) the slightest change in input voltage creates an unbalance potential, causing a current to flow. The current passes through a converter in which contacts alternately reverse the direction of this current through the center-tapped primary of a transformer, and an alternating voltage is induced in the transformer secondary. An electronic tube amplifier amplifies the alternating current voltage and energizes one winding of the two-phase balancing motor. The motor accelerates, moving the contact along the measuring slidewire and the pen across the chart to the new balance position.

At the new balance position, the potential of the poteniometer circuit is equal and opposite to the input voltage. There is then no potential difference in the system until the input voltage changes. The balancing motor is revesible and it can operate to move the slidewire contact in the proper positioning direction for an increasing voltage or in the reverse direction for a decreasing voltage.

The signal from the differential thermocouple is passed through a stabilized direct current indicating amplifier before passing through the speedomax and being recorded.

Program Control Unit

The rate of furnace heating may be controlled by either of two ways (1) manually, by hand setting the control point setter on the Leeds and Northrup Speedomax or automatically by use of a thermocouple operating through the Leeds and Northrup Speedomax and the Leeds & Northrup Position

ClibPDF - www.fastio.com

adjusting control unit. The control is equipped with a selector switch and various heating increase rate may be selected.

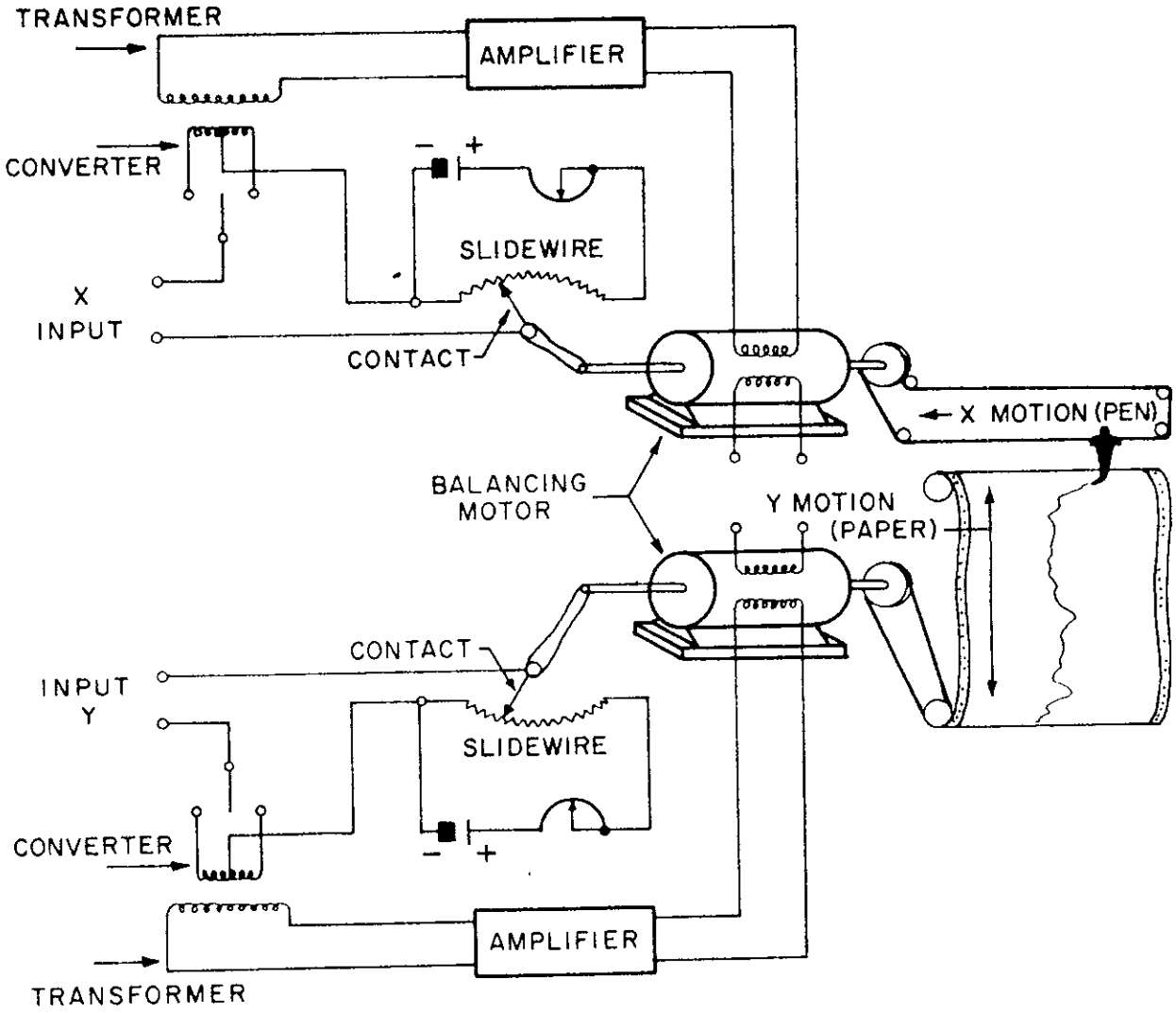
The position adjusting control unit is basically a contact on a control slidewire. As the relative position of the contact on the slidewire is changed, the position of the value slidewire changes, and causes one of three possible occurances: A signal having a positive polarity, a signal having a negative polarity or no signal. The signal is fed into an amplifier detector and depending on the polarity of the signal, operates a reversible valve drive motor which drives the valve, feeding current into the furnace and causing the oven to heat until the X-Y signal ceases.

Power Supply Unit

The power supply to the furnace transformer is regulated by General Radio V-20-M Variac operated by a motor-drive unit controlled by the program controller. The Variac is rated at 3.5 KVA - 115 volts.

A diagrammatic representation of the temperature - differential temperature recording system is shown in Figure #7.

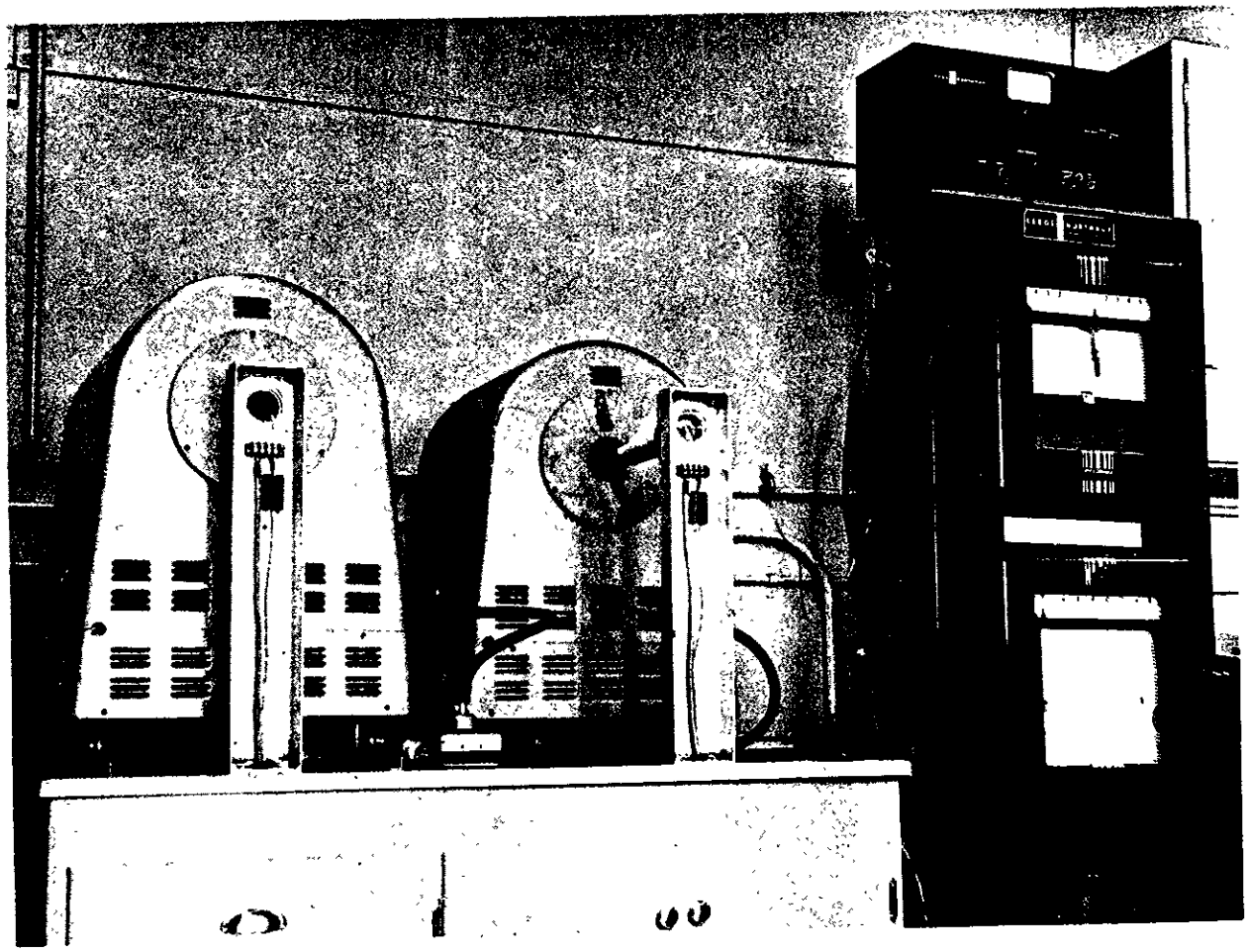
ClibPDF - www.fastio.com



DIAGRAMMATIC REPRESENTATION OF TEMPERATURE-DIFFERENTIAL TEMPERATURE RECORDING SYSTEM

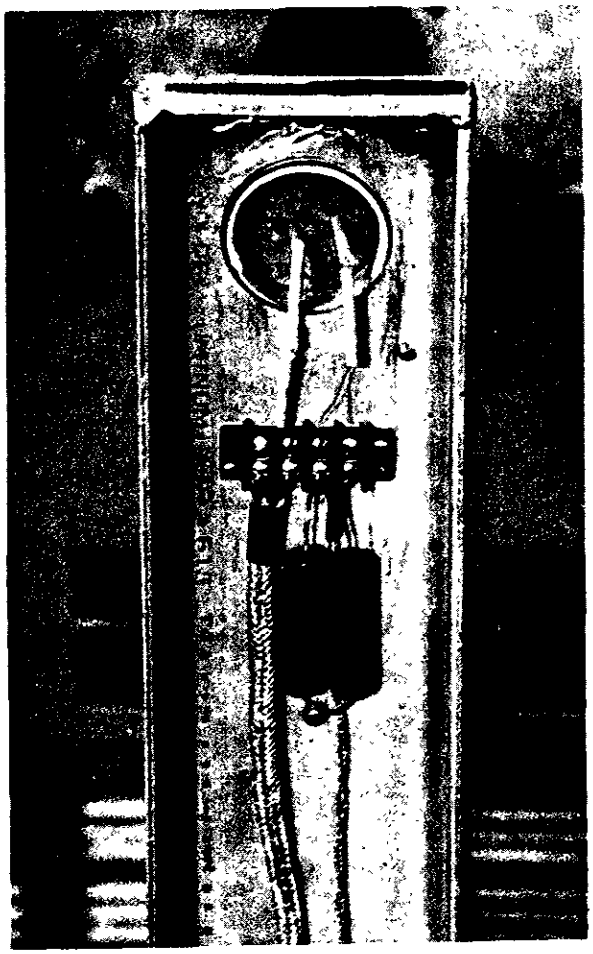
Figure 6

ClibPDF - www.fastio.com

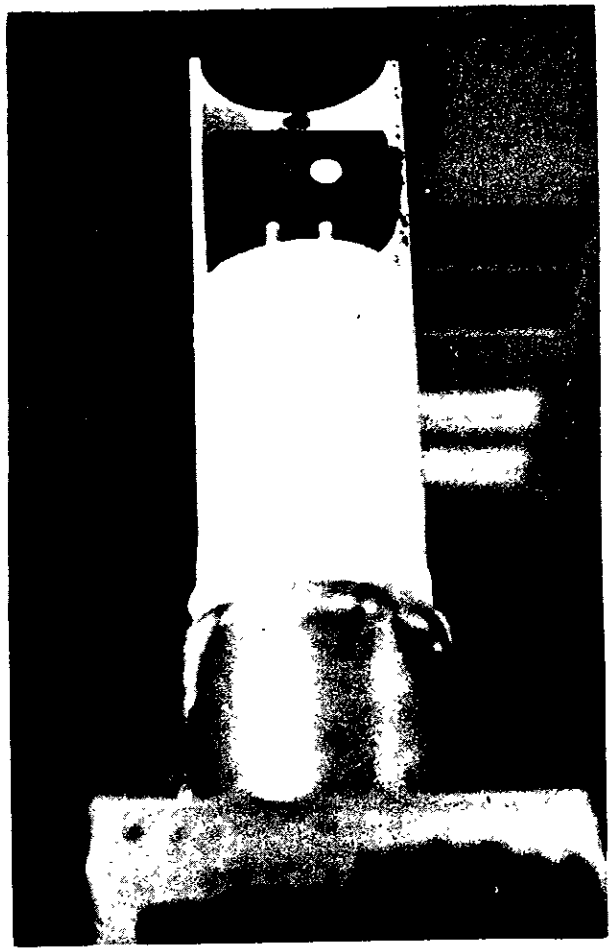


DIFFERENTIAL THERMAL ANALYSIS EQUIPMENT

ClibPDF - www.fastio.com

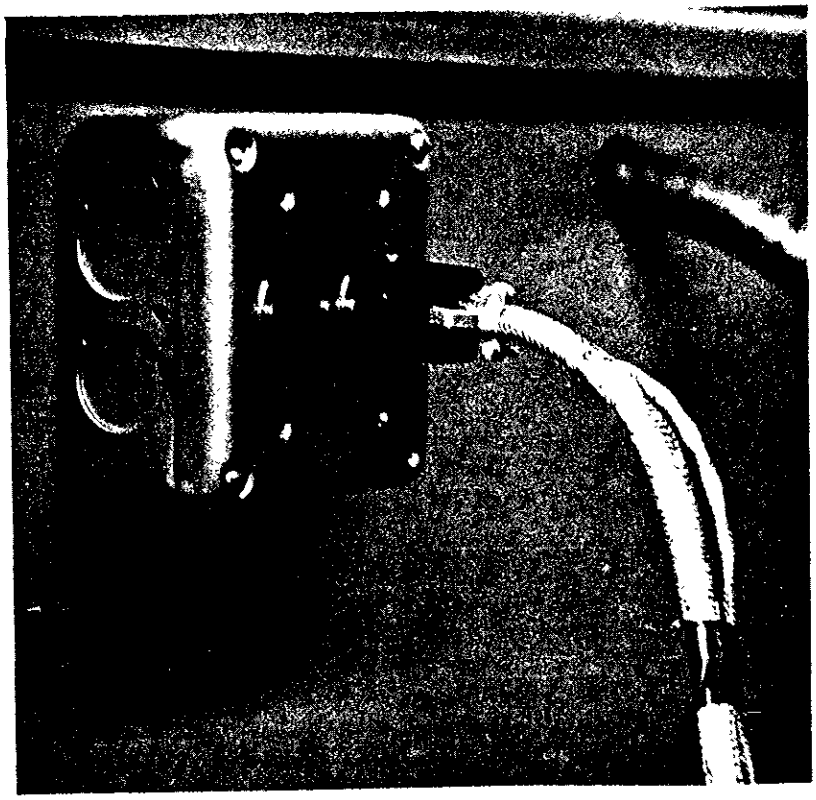


ELECTROLYTIC CONDENSERS FOR FILTERING STRAY CURRENTS



NICKEL SAMPLE HOLDER IN ALUNDUM TUBE

ClibPDF - www.fastio.com



SWITCHING ARRANGEMENT FOR OVENS

ClibPDF - www.fastio.com

THERMOGRAMS OF SELECTED CALIFORNIA SOIL SAMPLES

Differential thermograms of soils and construction materials used in various parts of the State are described in the following section. The sample site locations are indicated on a generalized soils map of California (Plate I). The thermograms are grouped and discussed by highway districts. The thermograms are designated by district, county, route and section.

The illustrations are half size and were prepared by means of a pantograph. The principal clay mineral is indicated under the country, route, and section designation.

The mineralogical constituents of the soils were identified by comparing the thermograms of the California Soils with thermograms published by various laboratories. The reference most used was Reference Clay Minerals, American Petroleum Institute, Research Project 49.

In some instances, chemical treatment of the soil was necessary to identify some of the constituents. In cases when this was done, several thermograms are shown for each material, and the nature of the chemical treatment is indicated.

District I - Fig. 7

I-Men-1-Uki,C

This sample contains an illitic clay mineral and quartz. The illitic nature of the clay mineral in this sample is indicated by the weak endothermic reaction between 100-160°C. marking the loss of adsorbed water, followed by the exothermic peak at 350°C. and the endothermic peak about 530°C. The exothermic dome from 880° to 950°C. indicates the formation of gamma aluminous oxide, and the final destruction of the illitic lattice. The material was taken from a pit on the Russian River.

District II -

II-Sha-28-C

This sample contains an illitic clay mineral. It was taken from Burney Creek. The illitic nature is indicated by the weak endothermic reaction from 100-160°C. marking the loss of adsorbed water, the endothermic peak at 330°C., and the endothermic peak at 510°C. The final destruction of the illitic lattice is indicated by the rather indistinct exothermic dome from 890°C. to 960°C.

ClibPDF - www.fastio.com

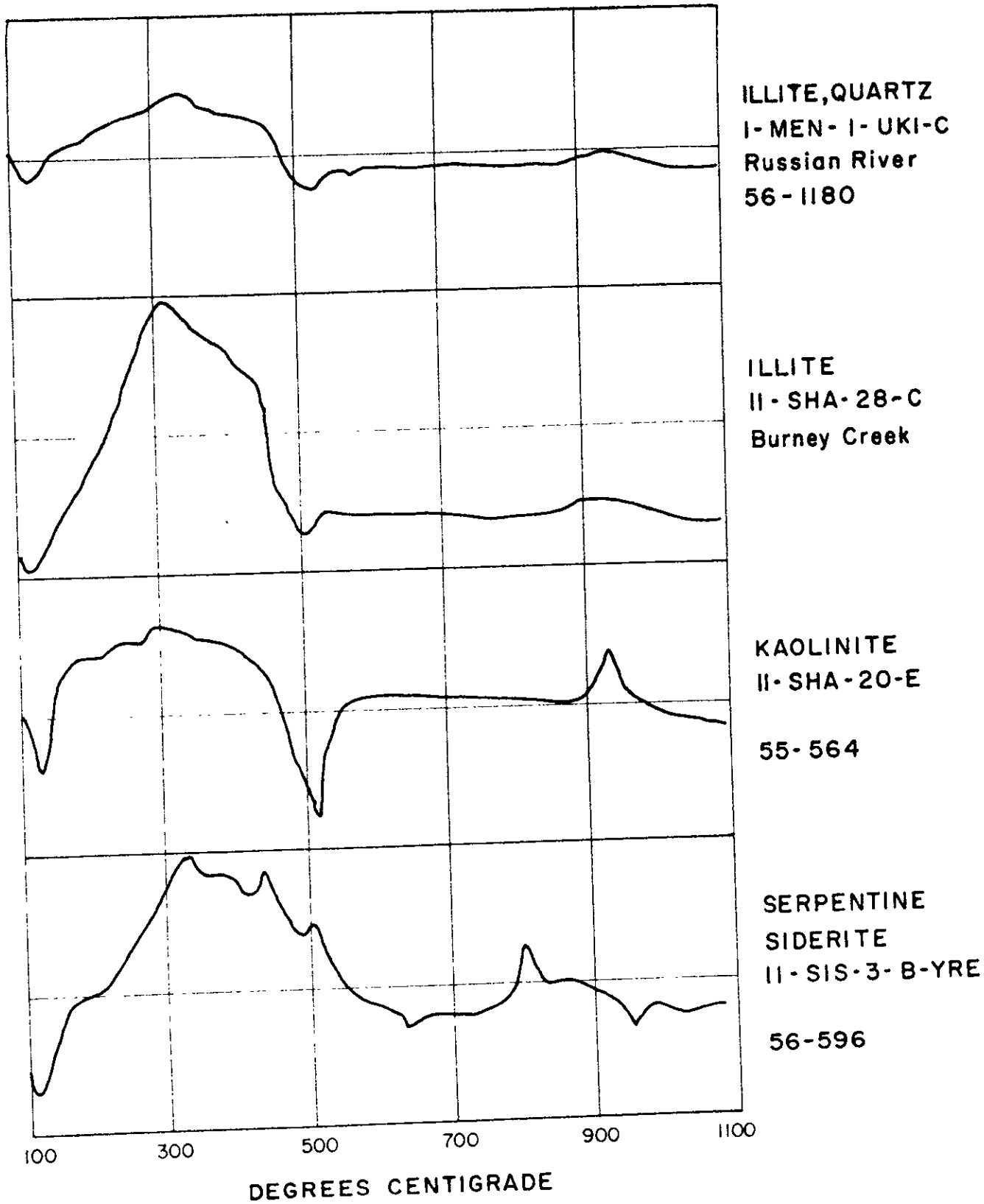


Figure 7

ClibPDF - www.fastio.com

II-Sha-20-E

This sample contains a clay mineral of the kaolin group. The endothermic peak occurs about 530°C. and the exothermic peak about 940°C. Both of these temperatures are rather low for a kaolin, and would indicate that the kaolin is in a poorly cystalline state. The flexures in the thermograph between 200° and 500°C. are due to the presence of iron oxides and organic matter.

II-Sis-3-B,Yre

This sample is rather unusual in that the soil constituents are not clay minerals. The predominate constituent is serpentine; this is indicated by the sharp endothermic reaction at 640-660°C. and the sharp exothermic reaction at 820° C. The series of endothermic peaks between 300° and 500°C. is caused by siderite and organic matter.

District III - Figs. 8, 9 & 10

III-Pla

This material is of volcanic origin. It is highly weathered, and broke down completely when immersed in water. The clay mineral present is of the kaolin group as indicated by the sharp endothermic peak at 580°C. and the sharp exothermic peak at 970°C.

III-Col-7-C

This sample contains both illite and montmorillonite as well as quartz. The presence of illite is indicated by the endothermic bend about 540°C. and the exothermic dome from 860° to 940°C. The presence of montmorillonite is indicated by the sharp endothermic reaction at 710-730°C. The quartz is indicated by the sharp endothermic reaction at 573°C.

Capell Creek Aggregate

This material is a sample of subbase material submitted by the U.S. Bureau of Reclamation. It was used in road construction of rerouted state routes necessitated by the construction of Monticello dam.

The material contains an illitic clay mineral as indicated by the endothermic peak about 550°C. and the broad exothermic dome from 870° to 970° C. The presence of serpentine is indicated by the sharp endothermic peak at 640-660°C. and the sharp exothermic peak at 820°C. Quartz is indicated by the sharp endothermic peak at 573°C.

ClibPDF - www.fastio.com

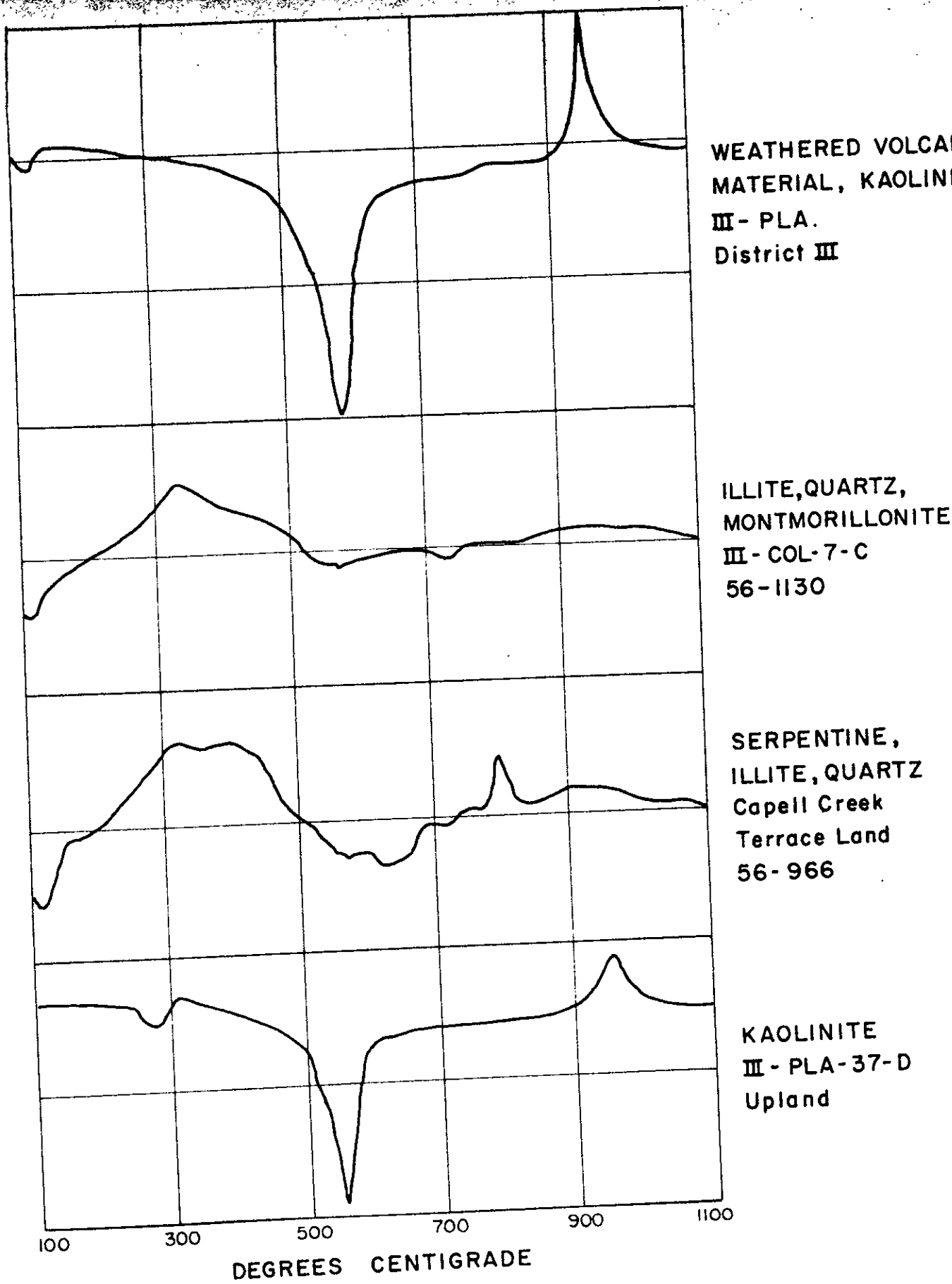


Figure 8

ClibPDF - www.fastio.com

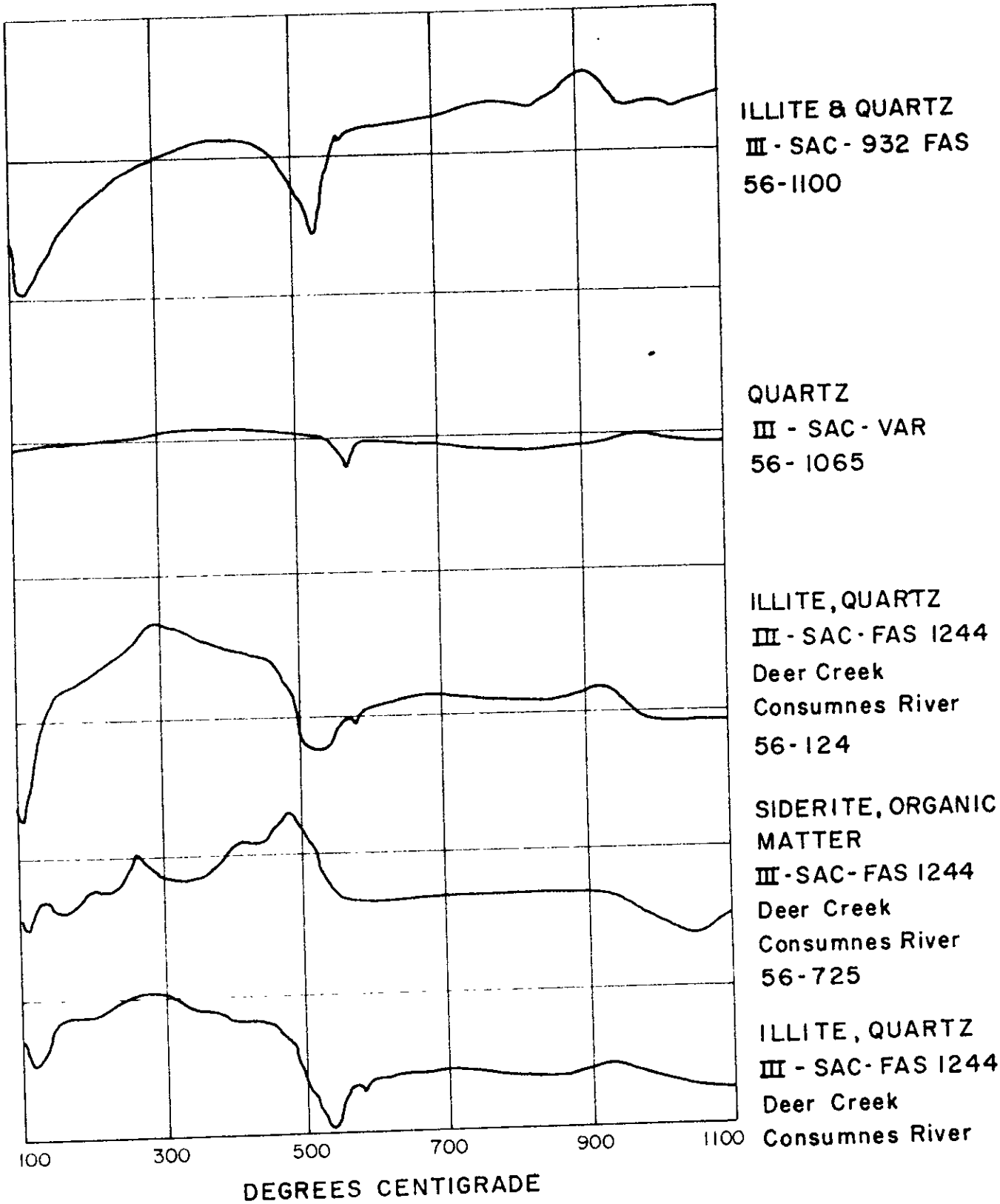


Figure 9

ClibPDF - www.fastio.com

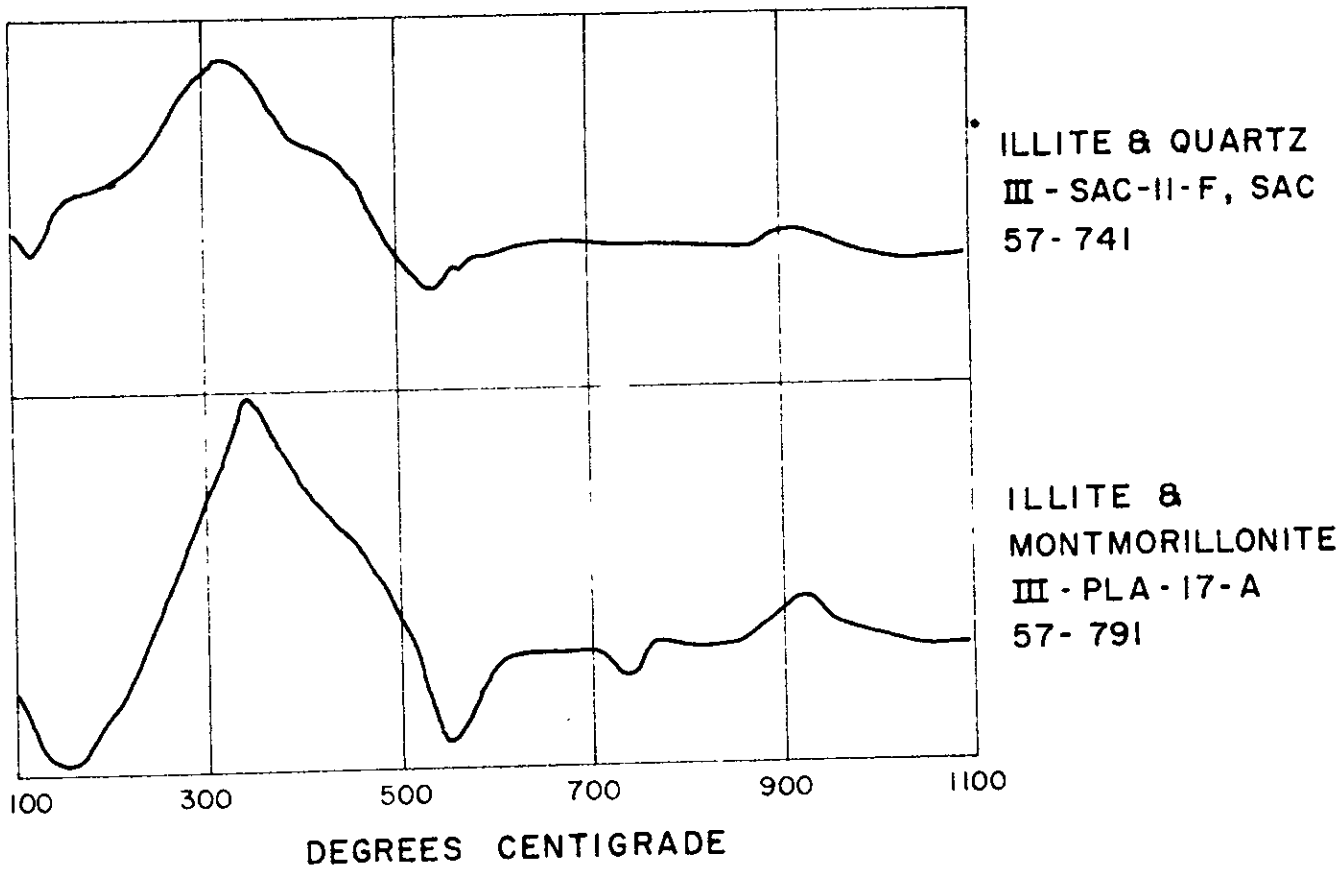


Figure 10

ClibPDF - www.fastio.com

III-Pla-37-D

The clay material in this sample is of a kaolinitic nature. The sharp endothermic peak at 570°C. and the sharp exothermic peak at 980°C. are characteristic of kaolin.

III-Sac-932-FAS

This sample contains illite and quartz. The endothermic peak occurring about 530°C. is not too typical of illite, but the exothermic dome from 870° to 940°C. is very typical of illite. The sharp endothermic reaction at 573°C. indicates quartz.

III-Sac-Var

The only thermally reactive, identifiable material in the sample is quartz. The quartz inversion is very strong. The rest of the line is nearly straight. There is a slight suggestion of an exothermic dome about 1000°C., but it is not pronounced enough for identification of any clay mineral.

III-Sac-FAS 1244

These three samples are of material from the Deer Creek - Cosumnes River drainage area. Two samples (56-724 & 726) contain illite and quartz as indicated by endothermic peaks at about 540°C. and exothermic domes at 880 to 930°C. the presence of quartz is indicated by the sharp endothermic peak at 573°C. The third sample (56-725) contains siderite (iron carbonate) and organic matter.

III-Sac-11-F,Sac

This sample contains illite and quartz. The illite seems to be fairly well crystallized as indicated by the endothermic peak from 100-160°C. marking loss of adsorbed water. The exothermic peak about 350°C., the endothermic peak about 540°C. and the exothermic dome from 890° t0 930°C. The presence of quartz is indicated by the sharp endothermic peak at 573°C.

III-Pla-17-A

This sample contains illite as indicated by the endothermic peak at 550°C. and the exothermic peak at 920°C.; and montmorillonite as indicated by the sharp endothermic peak at 730-750°C.

District IV - Figs. 11 & 12

IV-CC-69-N,14-Richmond

This sample contains illite and quartz. The illite seems to be fairly well crystallized as indicated by the

ClibPDF - www.fastio.com

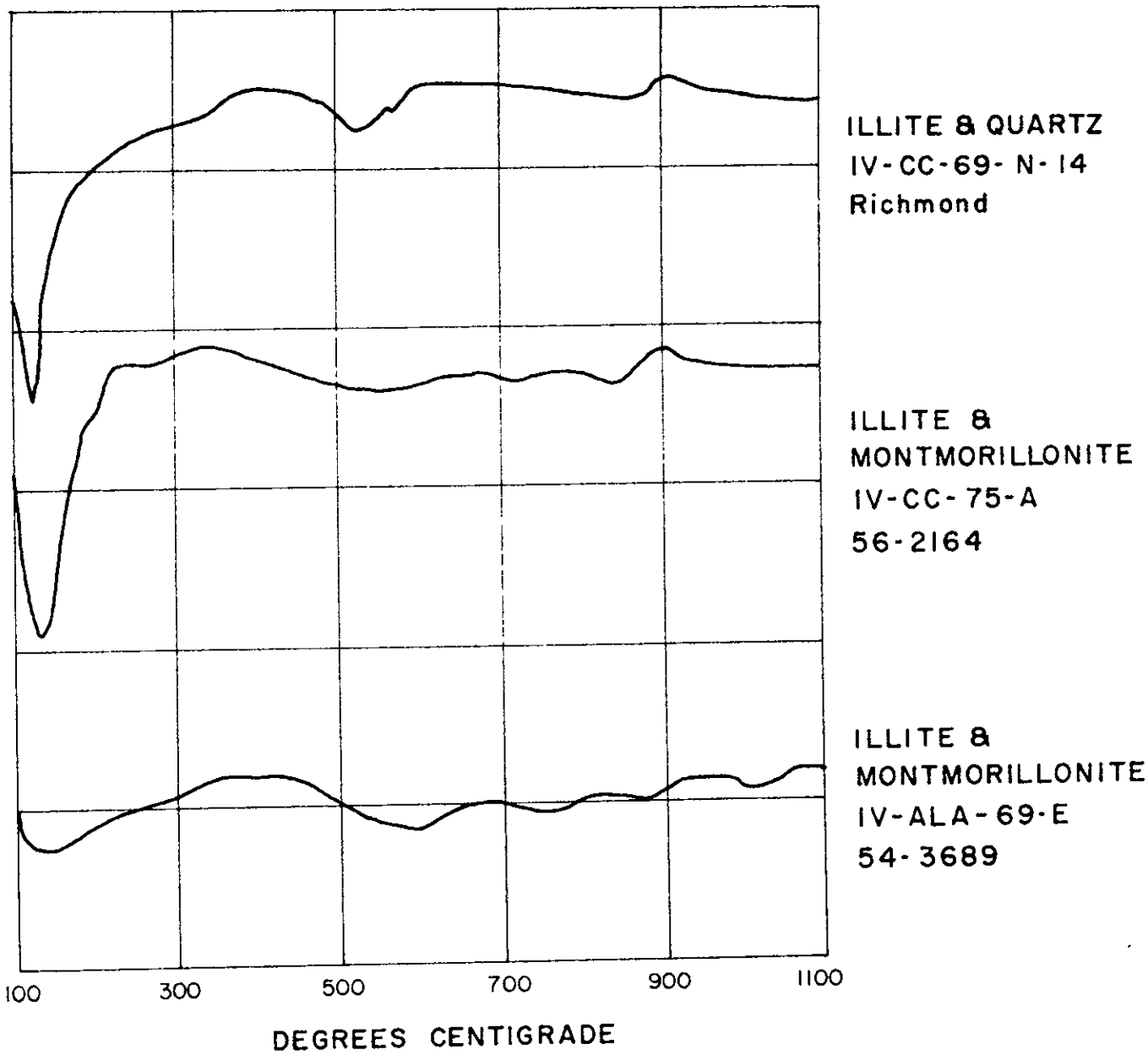


Figure 11

ClibPDF - www.fastio.com

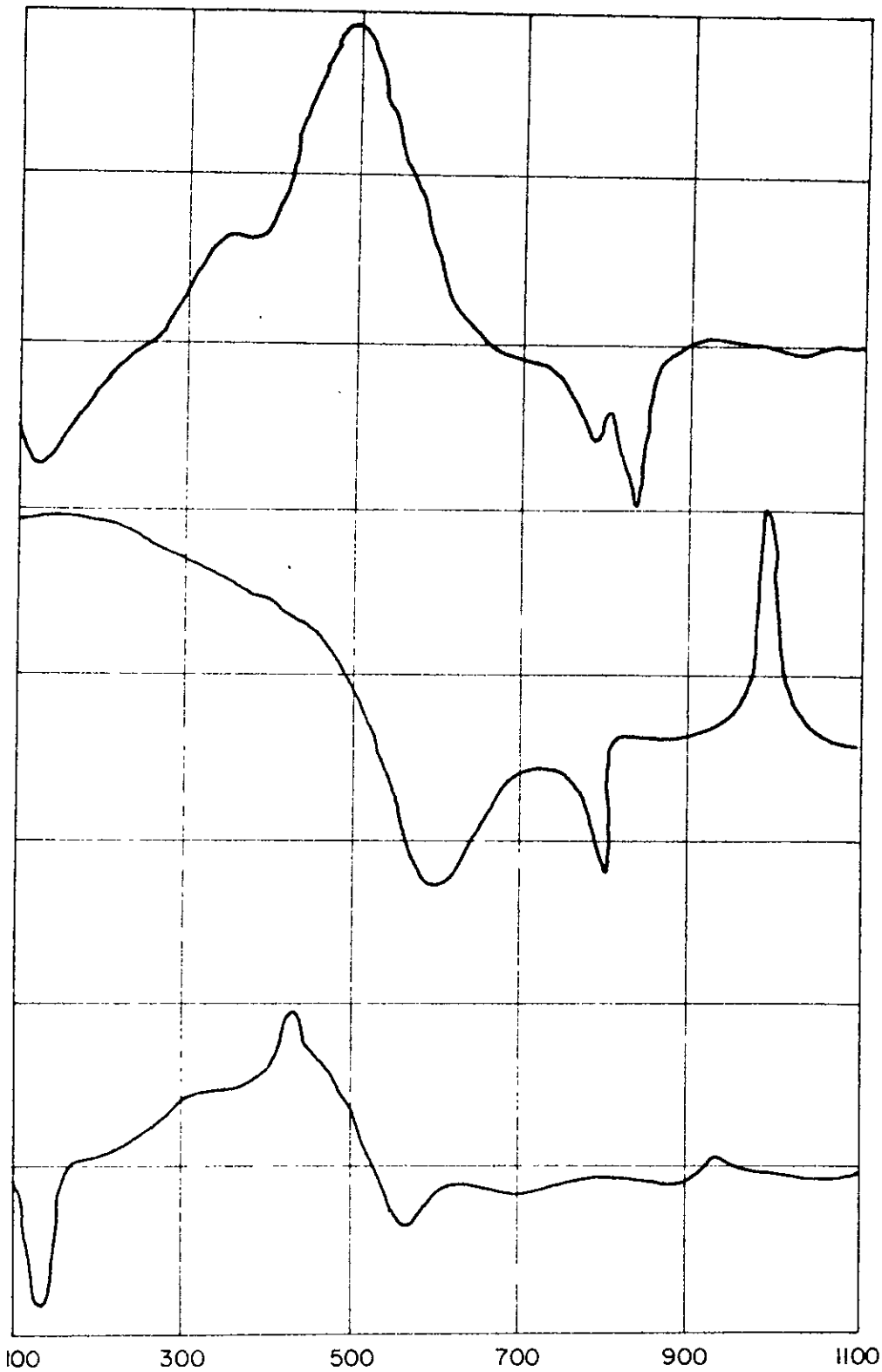


Figure 12

ClibPDF - www.fastio.com

sharp endothermic peaks from 100° to 150°C. The exothermic peak at 400°C. The endothermic peak at 530°C. and the exothermic dome at 900°C. The presence of quartz is shown by the sharp peak at 573°C.

IV-CC-75-A

This sample contains a poorly developed illitic clay mineral and quartz. The presence of illite is indicated by a broad flexure from 450° to 600°C. and a sharp doublet from 850° to 930°C. There is a very slight endothermic flexure indicative of quartz at 573°C.

IV-Ala-69-E

This sample contains illite and montmorillonite. The illite is not too well crystallized as indicated by the broadness of the endothermic peak at about 550°C. and the doublet and exothermic dome from 880°C. to 1000°C. The presence of montmorillonite is indicated by the endothermic peak at 750°C.

IV-SCr-56-D

This sample is of an alteration product fround in the granite at Wadell Bluffs. The clay mineral present is montmorillonite. The presence of siderite (iron carbonate) is indicated by the exothermic peak at 500°C.

IV-Mrn-1-C,D,Saus

This sample contains a kaolinitic clay mineral and calcite. The endothermic peak occurring about 600° is broad and poorly defined, and the exothermic peak at 980°C. is very sharp. The presence of calcite is indicated by the sharp endothermic peak about 800°C.

IV-CC-Carquinez

This material is an illitic clay taken from a slippage plane. The illitic nature is clearly defined by the sharp endothermic peak at 550°C. and the doublet at 900°C. The presence of quartz is indicated by the endothermic peak at 573°C.

District V - Fig. 13 & 14

V-SLO-33-B

This material is a montmorillonitic clay. The sharp endothermic reaction at 670°C. and the lesser endothermic peak at 730°C. are indicative of montmorillonite.

ClibPDF - www.fastio.com

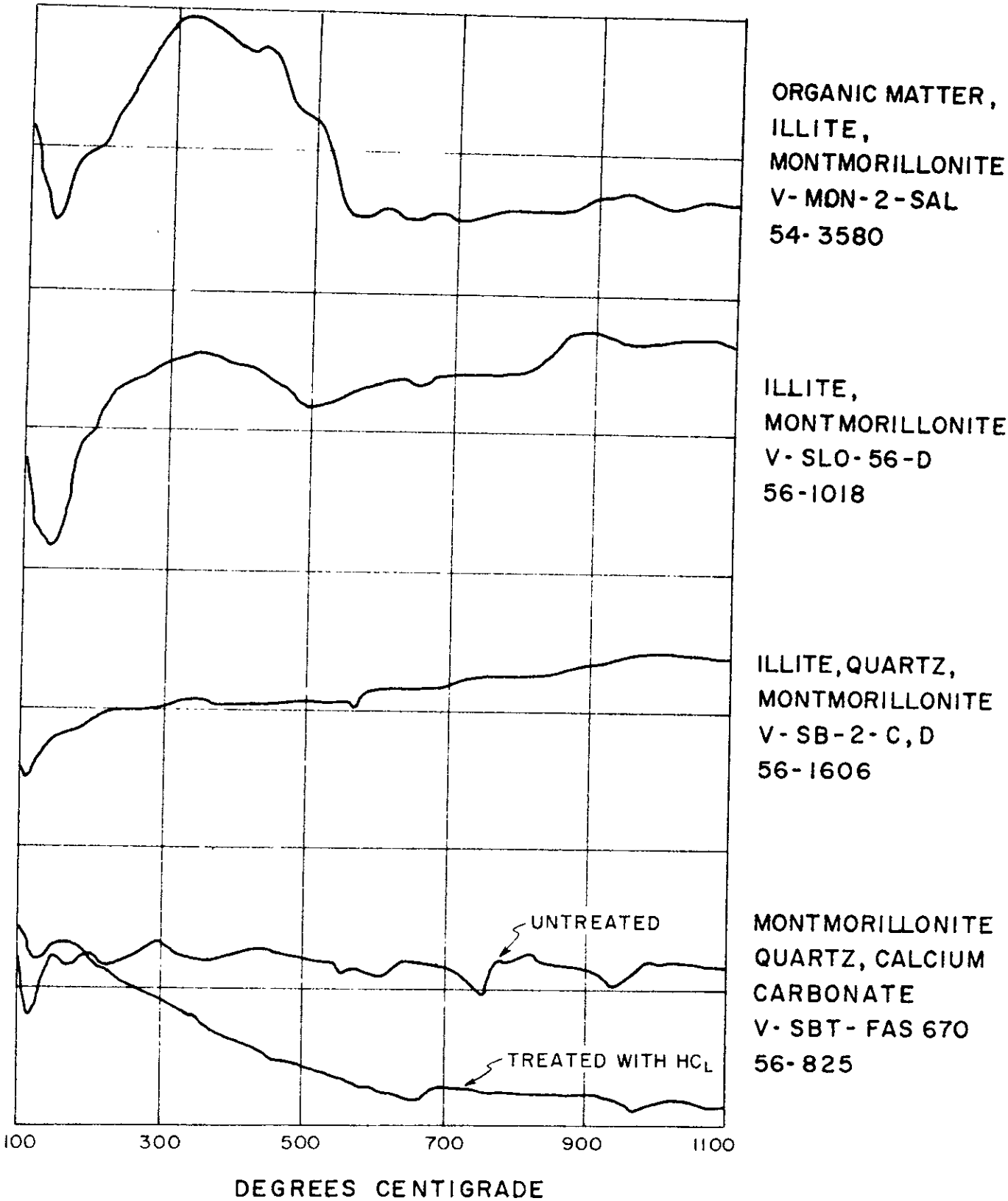


Figure 13

ClibPDF - www.fastio.com

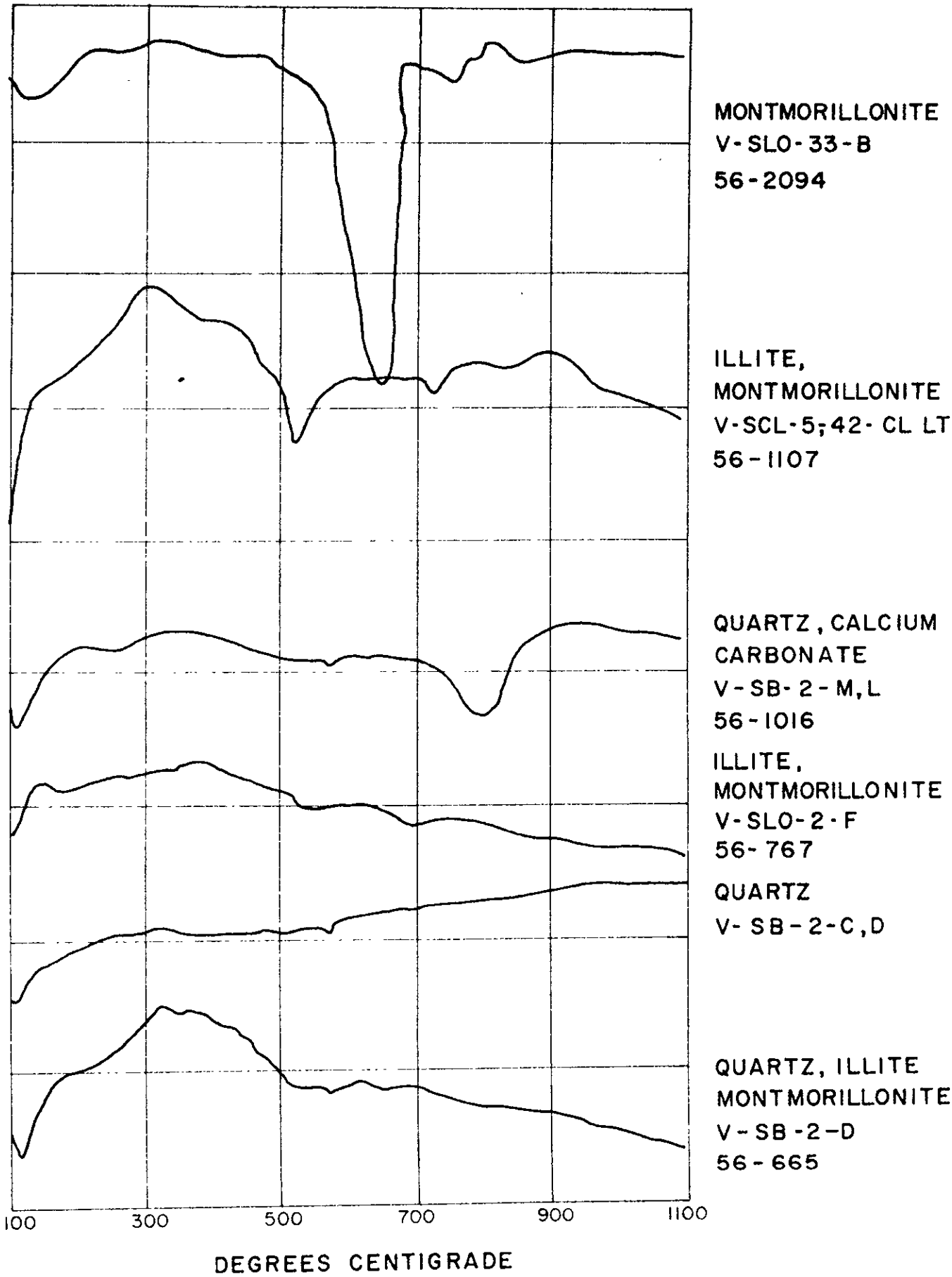


Figure 14

ClibPDF - www.fastio.com

V-SC1-5,42-C1,Lts,D

This soil sample contains two clay minerals, illite and montmorillonite. The illite is well crystallized as indicated by the sharp endothermic peak at 540°C. and the exothermic dome at 910°C. The presence of montmorillonite is clearly indicated by the sharp endothermic peak at 750°C.

V-SB-2-M,L

This sample contains quartz and calcium carbonate. The quartz is indicated by the sharp endothermic reaction at 573°C. and the calcium carbonate by the broad endothermic curve at 800° to 900°C.

V-SLO-2-F

This sample contains illite and montmorillonite. The illite is not too well crystallized as indicated by the lack of definition of the endothermic peak at 550°C. and the almost absent exothermic dome at 910°C. The presence of montmorillonite is indicated by the slight endothermic curve at 700°C. The exothermic peaks between 300° and 500°C. probably indicate siderite and organic matter.

V-SB-2-C,D

This sample contains quartz and some organic matter. The quartz is shown by the endothermic peak at 573°C., and the organic matter is indicated by the slight exothermic peak about 320°C.

V-SB-2-D

This sample contains illite, montmorillonite and quartz. The illite is poorly crystalline as indicated by the lack of peak definition as is the montmorillonite. The quartz makes a strong inversion at 473°C.

V-Mon-2-Sal

Illite, montmorillonite, and organic matter are the constituents of this sample. The illite is fairly well crystallized and the endothermic peak occurs about 550°C. The exothermic dome occurs about 930°-940°C. The montmorillonite characteristics are clearly shown by two endothermic peaks, one at 660°C. and the other at 720°C. The exothermic peaks between 300° and 500°C. are indicative of organic matter.

V-SLO-56-D

This sample contains illite and montmorillonite. The illite seems to be well crystallized as indicated by the strong endothermic peak at 500°C. and the exothermic dome at 900°C. The montmorillonite is indicated by the endothermic peak at 640°C.

ClibPDF - www.fastio.com

V-SB-2-C,D

This sample contains illite and quartz. The illite is poorly crystallized and the thermogram is poorly defined. The quartz endothermic inversion at 573° centigrade is sharp. There is a vague suggestion of montmorillonite at 680° centigrade as indicated by the slight endothermic flexure.

V-SBt-FAS 670

This sample contains montmorillonite, quartz and calcium carbonate. The montmorillonite is indicated by the endothermic peak at 660° centigrade and the doublet formed by loss of adsorbed water between 100° and 200° centigrade. The presence of the calcium carbonate obscured the initial thermogram and some of the material was treated with hydrochloric acid to remove it. Subsequently another thermogram was made and the montmorillonitic nature was more clearly established.

District VI - Fig. 15

VI-Ker-4-Bfd

The constituents of this sample are illite and siderite. The presence of illite is shown by the exothermic dome at 960° centigrade. The strong exothermic peak at 520° centigrade indicates the presence of siderite.

VI-Ker-4-Bfd

This sample contains illite and quartz. The endothermic inversion for the illite occurs somewhat lower than usual at about 510° centigrade and it probably indicates the material is rather poorly crystallized. The quartz inversion is sharp and distinct. The exothermic dome occurs at 900° centigrade, and is fairly well defined.

VI-Ker-4-Bfd

This sample contains illite and quartz. The illite seems to be well crystallized, as indicated by the sharpness of the endothermic peak at 550° centigrade. The exothermic dome occurring about 920° centigrade is well developed. The presence of quartz is indicated by the endothermic peak at 573° centigrade.

VI-Tul-10-B

This sample contains illite, calcium carbonate and quartz. The illite does not seem too well crystallized as the endothermic peak is not sharply defined. The calcium carbonate endothermic is very sharp, and this is indicative of a well developed crystalline state. The presence of quartz is indicated by the endothermic peak at 573° centigrade.

ClibPDF - www.fastio.com

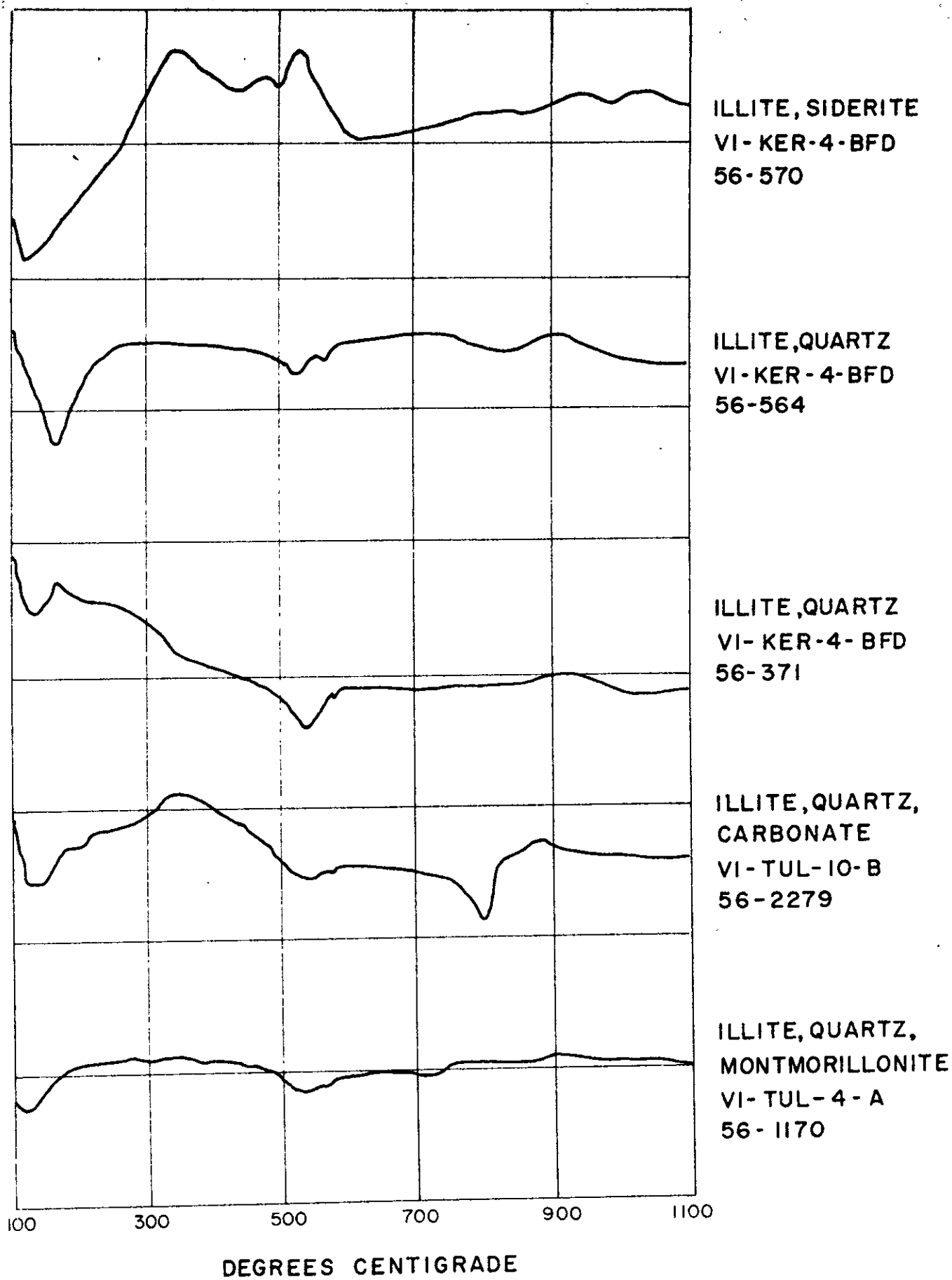


Figure 15

ClibPDF - www.fastio.com

VI-Tul-4-A

Illite, montmorillonite and quartz are the identifiable constituents of this sample. The illite endothermic curve is not too pronounced nor sharp. The endothermic peak for montmorillonite is well defined. The quartz peak is not too prominant.

District VII - Fig. 16

VII-Ven

This sample shows a pronounced, well defined illite endothermic peak, but it occurs at a rather low temperature for illite, about 500° C. The exothermic dome is well developed and occurs at 900° centigrade. The presence of quartz is indicated by a pronounced endothermic peak at 573° centigrade.

VII-Ven

This sample contains illite, montmorillonite and quartz. The endothermic peak for illite occurring about 550° centigrade is not too well defined nor is the exothermic dome at 900° centigrade. The presence of montmorillonite is indicated by endothermic peaks at 700° centigrade and about 850° centigrade. Quartz is indicated by a slight almost indiscernable endothermic peak at 573° centigrade.

District VIII - Fig. 16

VIII-Riv-19-D

This sample contains montmorillonite and quartz. The presence of montmorillonite is indicated by the endothermic peaks occurring at 610° centigrade and at about 830° centigrade. The quartz endothermic peak at 573° centigrade is almost indiscernable.

District X - Fig. 17

X-Sol-7-C

This sample contains a considerable amount of organic matter. The series of exothermic peaks from 200° to 500° centigrade are caused by the oxidization of the organic matter and the burning of the carbon formed by the oxidation. The presence of illite is indicated by the endothermic peak about 540° centigrade and the well defined exothermic dome at 930° centigrade. The lack of definition of the endothermic peak of illite is due to the pronounced exothermic reaction of the oxidization of the organic matter. The presence of quartz is indicated by the endothermic peak at 573° centigrade.

ClibPDF - www.fastio.com

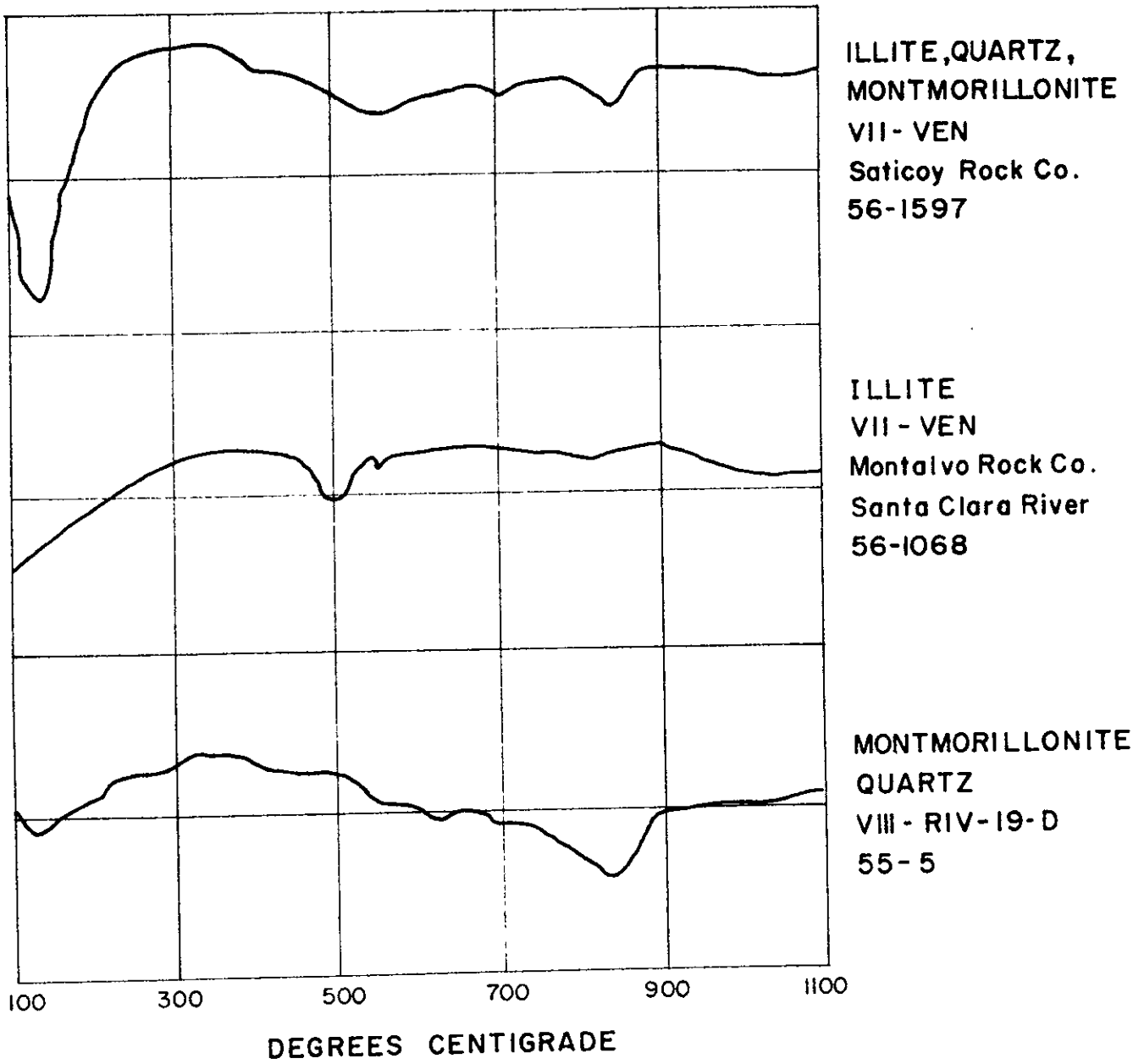


Figure 16

ClibPDF - www.fastio.com

District XI - Fig. 17

XI-SD-199-A

Illite, montmorillonite and quartz are the constituents of this sample. The endothermic peak for illite at about 540° centigrade and the exothermic domed doublet at 900° centigrade are well defined. The presence of montmorillonite is indicated by an endothermic peak at 720° centigrade. The quartz endothermic peak at 573° centigrade is rather vague.

ClibPDF - www.fastio.com

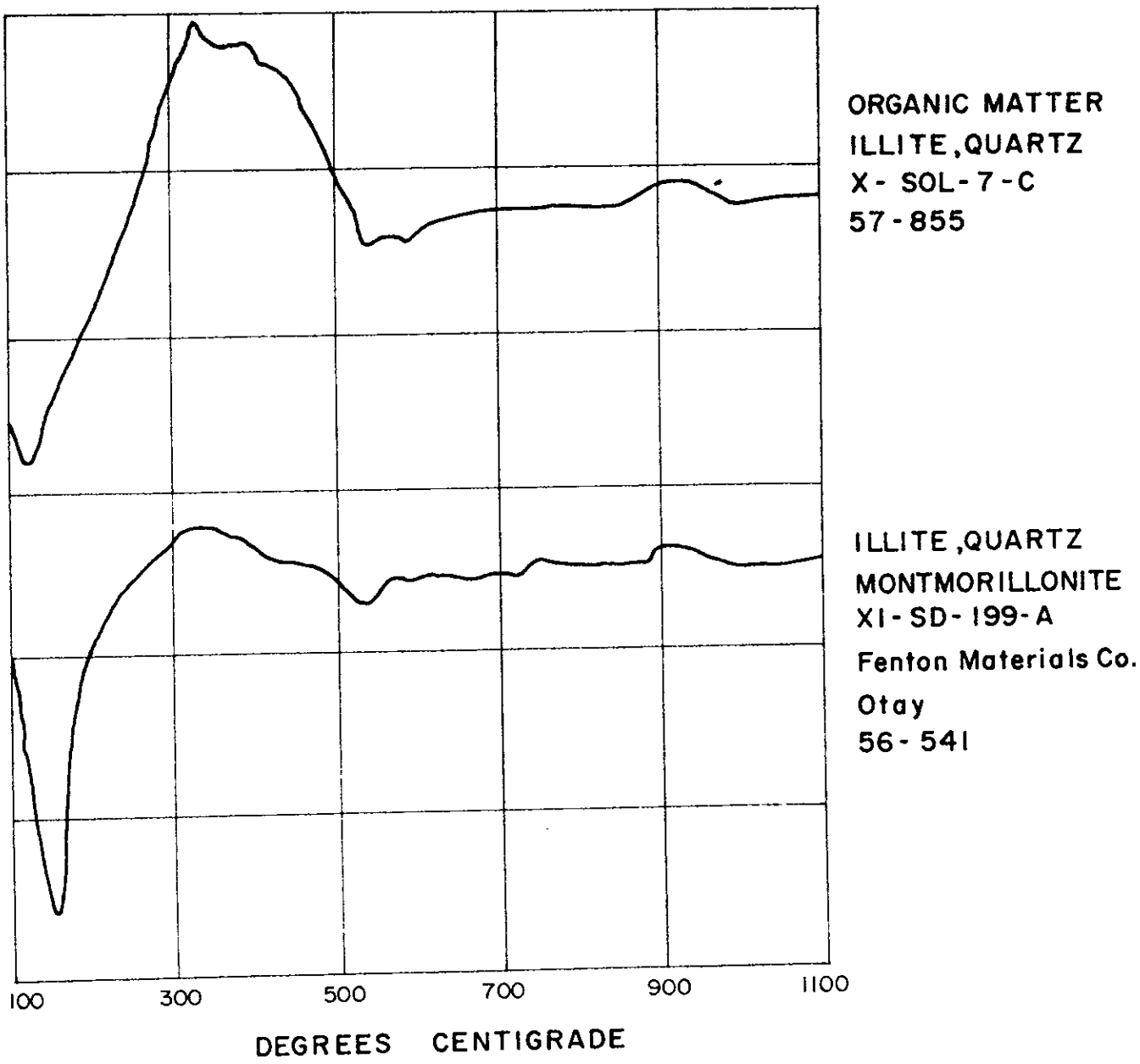


Figure 17

ClibPDF - www.fastio.com

THERMOGRAMS OF SUITES OF SOIL SAMPLES

In the previous section, individual samples from the various districts were described: soils investigations of a portion of the proposed Westside Freeway resulted in the collection of a suite of samples from ten borings and the opportunity to prepare thermograms of several samples from each hole.

The thermograms are presented with the lithological log of each hole and the differences in clay mineral type can readily be noted.

In boring D-1, (Figure 18) all samples analyzed were similar in mineralogical content. All contained illite, and montmorillonite, gypsum and quartz.

The samples from boring D-2 (Figure 19) show considerable variation in the accessory mineral constituents of the soils. All samples contained illite and montmorillonite. Sample 2-B (7-10 depth_ contained gypsum, quartz and a carbonate. Sample 4-A (17-20') contained gypsum and quartz but no carbonate. Sample 6-A (27-30') contained only typsum while sample 8-A (35-36') contained gypsum and quartz.

The material in boring D-3 (Figure 20) seems separable into two groups. The upper, as represented by samples 2A (7-10') and 3A (11-12') being an illite-montmorillonitic soil with gypsum, carbonate and quartz in the 11-12' horizon. The lower group is an illitic clay containing quartz and carbonate with gypsum in the lowest sample 8-I (39-40').

In boring D-4, (Figure 21) all samples except 1A (3-5') contained illite, montmorillonite, gypsum, quartz and carbonate. Sample 1A contained no carbonate.

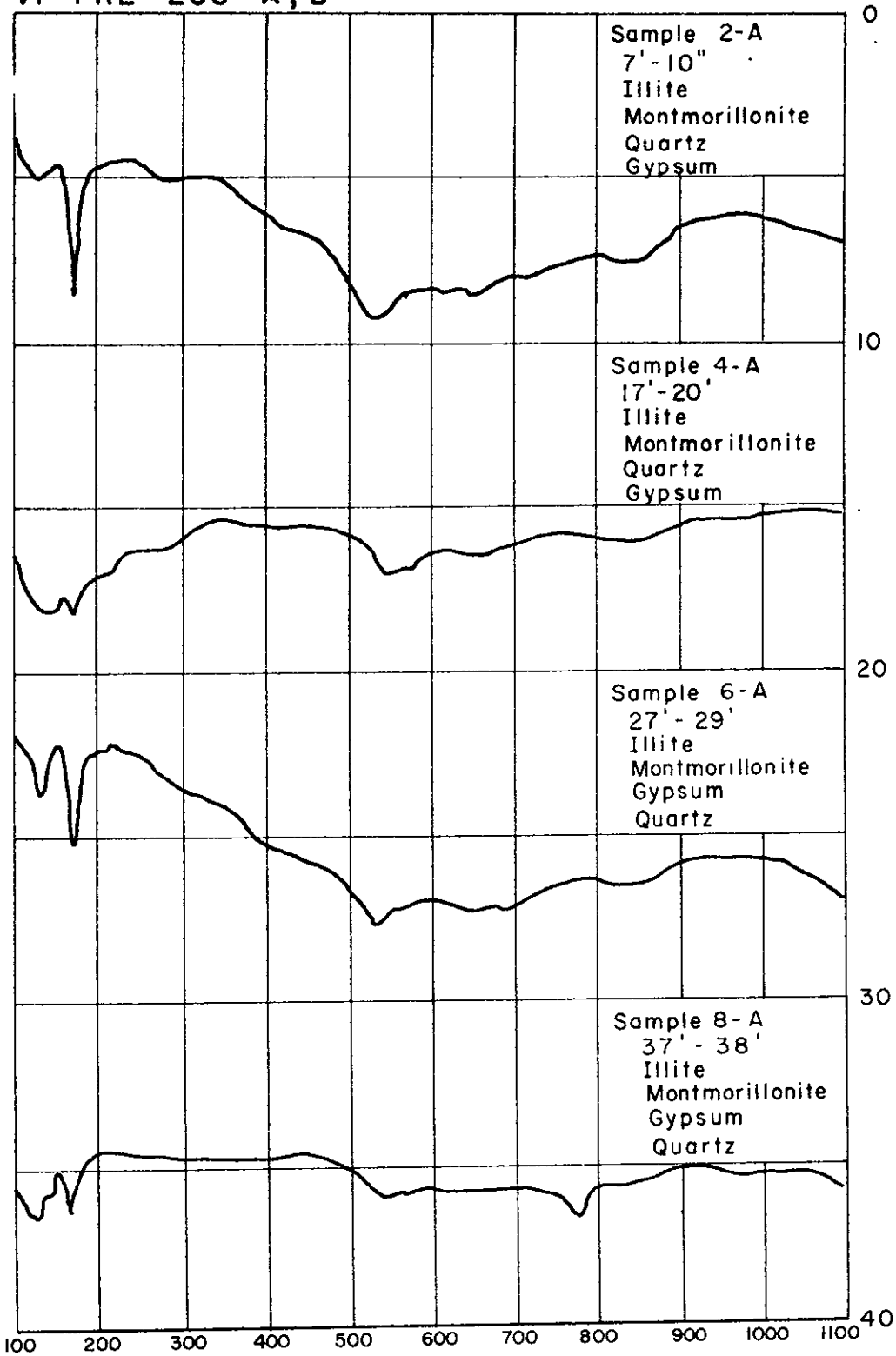
Samples from boring D-5 (Figure 22) were all similar in mineralogical content in that all contained illite, montmorillonite, gypsum, and quartz and carbonate with the exception of the sample from 28-30 feet (9A) which contained no carbonate.

The samples from boring D-6 (Figure 23) are all similar in mineralogical content in that all contain illite and quartz. Gypsum is present in sample 2A (8-9') carbonate in sample 4-III (18-20') both carbonate and gypsum in sample 6-I (28-30') and carbonate, gypsum and montmorillonite in sample 8-I (38-39.3').

All samples from boring D-7 (Figure 24) contain illite and quartz. Sample 3-II (13-15') contains gypsum; sample

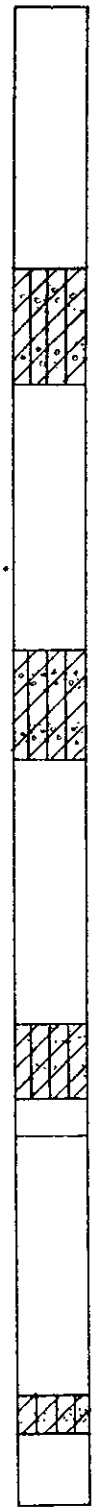
ClibPDF - www.fastio.com

DIFFERENTIAL THERMOGRAMS OF SAMPLES
From Boring D-1 , Job No 2206-T-20
Proposed West Side Freeway
VI-FRE-238-A, B



DESCRIPTIVE LOG

D-1

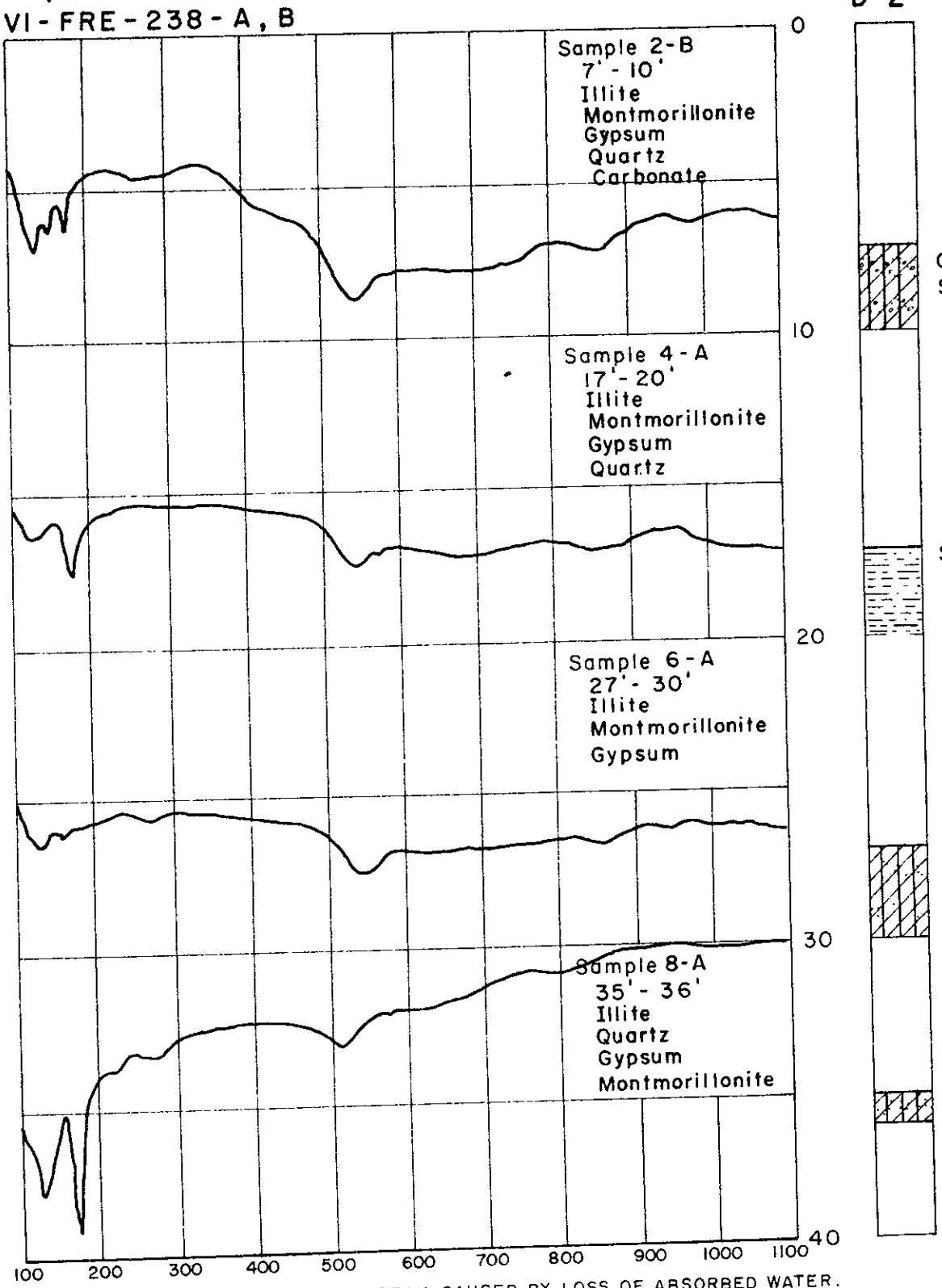


KEY- 100-150°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF ABSORBED WATER.
160-180°C SHARP ENDOTHERMIC PEAK CAUSED BY LOSS OF WATER OF HYDRATION IN GYPSUM.
330-550°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM ILLITE LATTICE.
573°C SHARP ENDOTHERMIC PEAK CAUSED BY QUARTZ INVERSION
640-660°C SLIGHT ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM MONTMORILLIONITE LATTICE.
770-790°C SHARP ENDOTHERMIC PEAK CAUSED BY DECOMPOSITION OF CARBONATE.
850-900°C BROAD ENDOTHERMIC PEAK CAUSED BY DESTRUCTION OF ILLITE & MONTMORILLONITE LATTICE & THE FORMATION OF SPINEL.

Figure 18

ClibPDF - www.fastio.com

DIFFERENTIAL THERMOGRAMS OF SAMPLES
From Boring D-2 , Job No 2206-T-20
Proposed West Side Freeway
VI-FRE-238-A, B



DESCRIPTIVE LOG

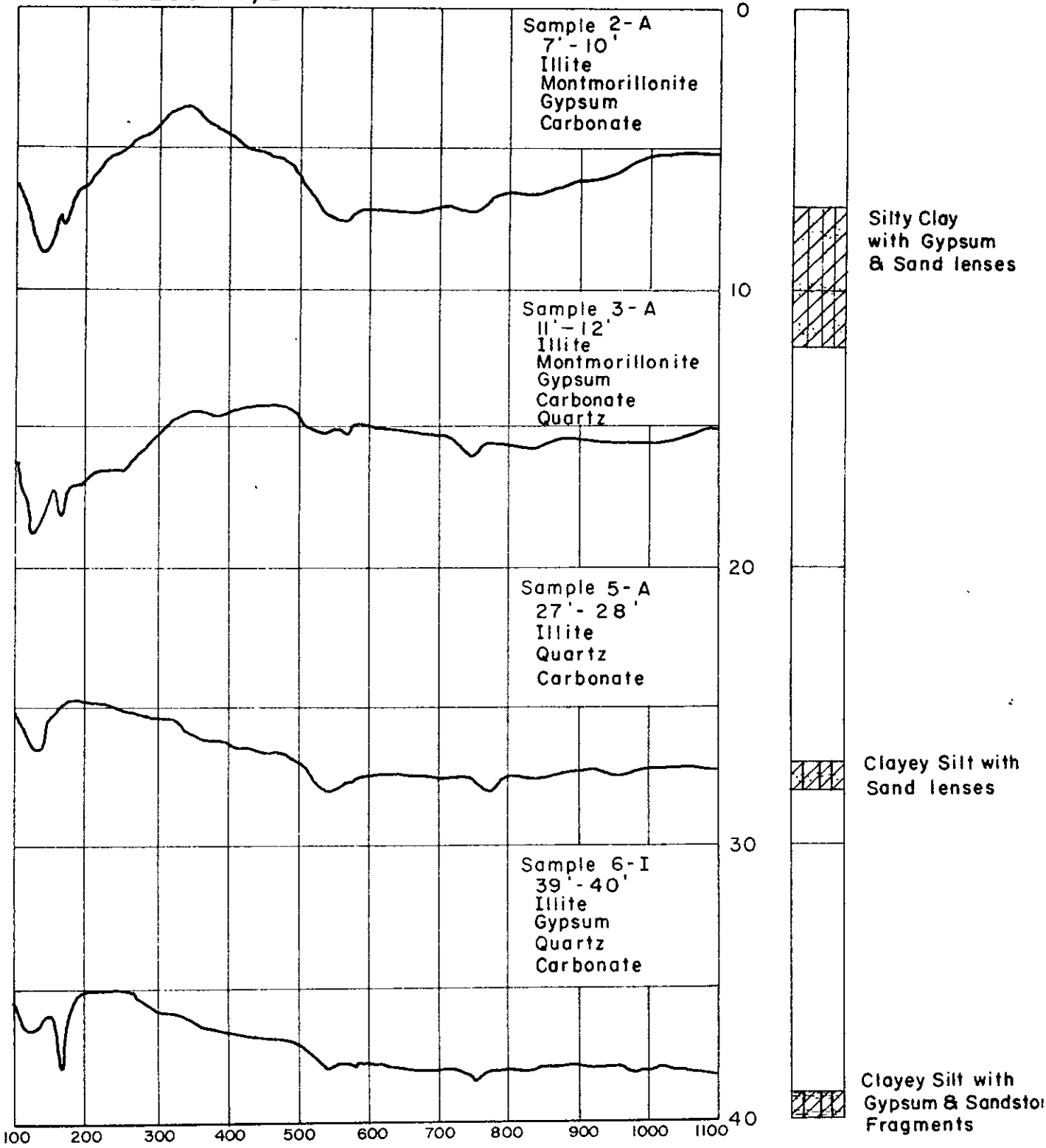
D-2

KEY- 100-150°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF ABSORBED WATER.
160-180°C SHARP ENDOTHERMIC PEAK CAUSED BY LOSS OF WATER OF HYDRATION IN GYPSUM.
330-550°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM ILLITE LATTICE.
573°C SHARP ENDOTHERMIC PEAK CAUSED BY QUARTZ INVERSION
640-660°C SLIGHT ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM MONTMORILLIONITE LATTICE.
770-790°C SHARP ENDOTHERMIC PEAK CAUSED BY DECOMPOSITION OF CARBONATE.
850-900°C BROAD ENDOTHERMIC PEAK CAUSED BY DESTRUCTION OF ILLITE & MONTMORILLONITE LATTICE & THE FORMATION OF SPINEL

Figure 19

ClibPDF - www.fastio.com

DIFFERENTIAL THERMOGRAMS OF SAMPLES
From Boring D-3 , Job No 2206-T-20
Proposed West Side Freeway
VI-FRE-238-A, B

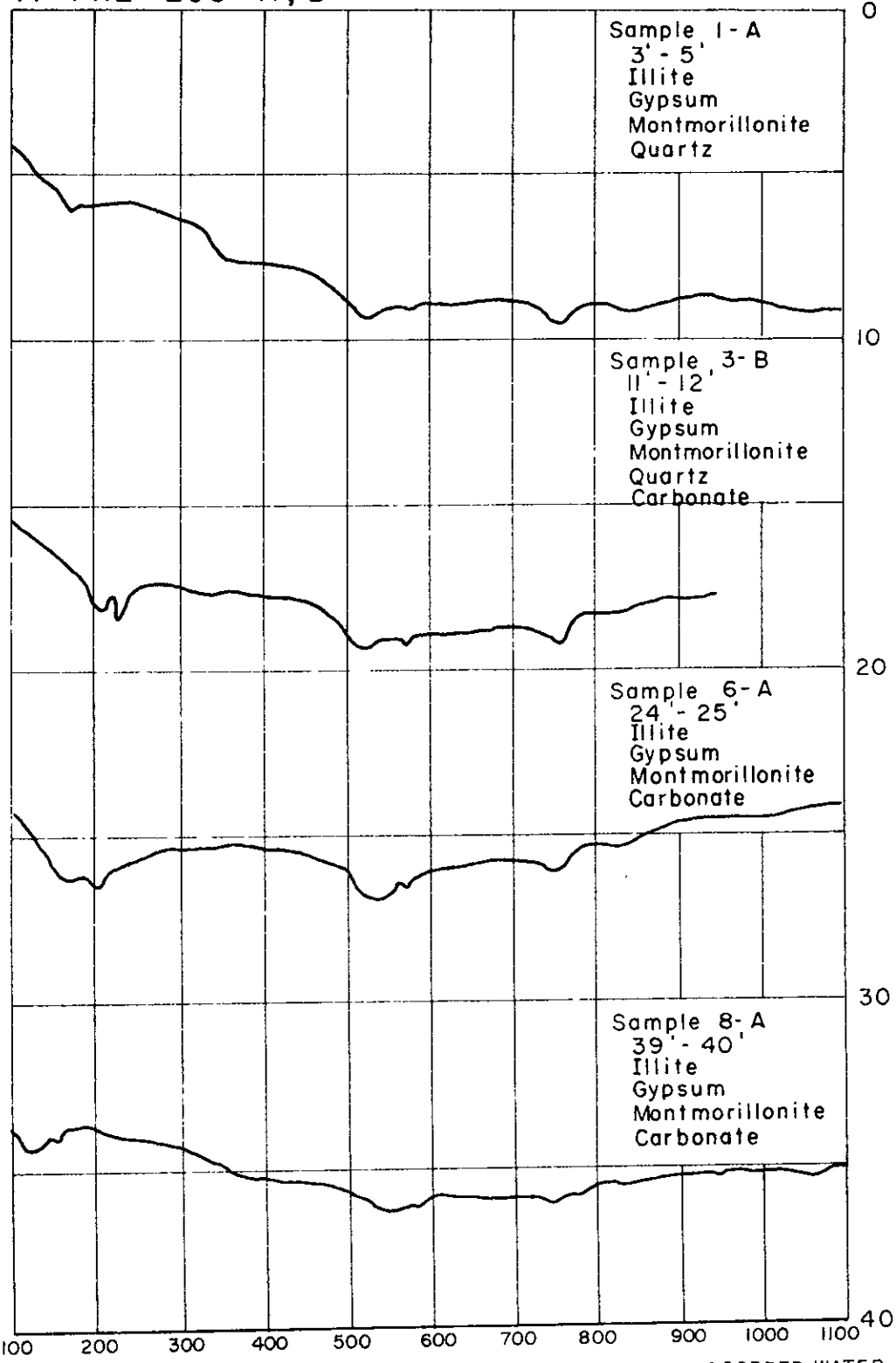


KEY- 100-150°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF ABSORBED WATER.
160-180°C SHARP ENDOTHERMIC PEAK CAUSED BY LOSS OF WATER OF HYDRATION IN GYPSUM.
330-550°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM ILLITE LATTICE.
573°C SHARP ENDOTHERMIC PEAK CAUSED BY QUARTZ INVERSION
640-660°C SLIGHT ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM MONTMORILLIONITE LATTICE.
770-790°C SHARP ENDOTHERMIC PEAK CAUSED BY DECOMPOSITION OF CARBONATE.
850-900°C BROAD ENDOTHERMIC PEAK CAUSED BY DESTRUCTION OF ILLITE & MONTMORILLONITE LATTICE & THE FORMATION OF SPINEL.

Figure 20

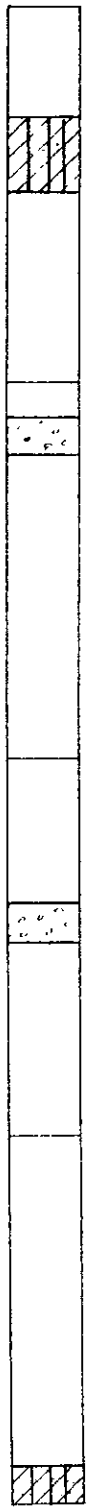
ClibPDF - www.fastio.com

DIFFERENTIAL THERMOGRAMS OF SAMPLES
From Boring D-4, Job No 2206-T-20
Proposed West Side Freeway
VI-FRE-238-A, B



DESCRIPTIVE LOG

D-4

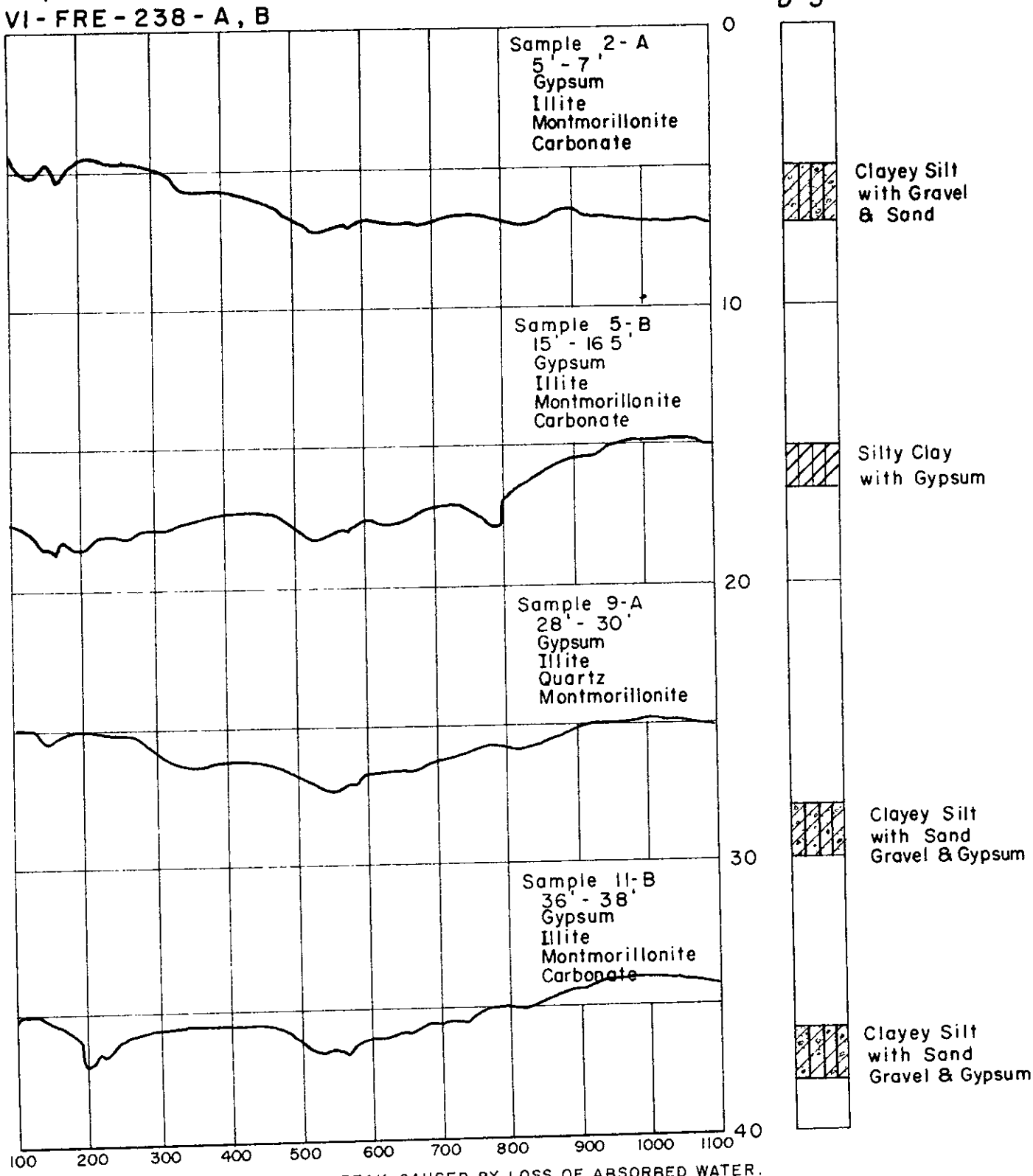


KEY- 100-150°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF ABSORBED WATER.
160-180°C SHARP ENDOTHERMIC PEAK CAUSED BY LOSS OF WATER OF HYDRATION IN GYPSUM.
330-550°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM ILLITE LATTICE.
573°C SHARP ENDOTHERMIC PEAK CAUSED BY QUARTZ INVERSION.
640-660°C SLIGHT ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM MONTMORILLIONITE LATTICE.
770-790°C SHARP ENDOTHERMIC PEAK CAUSED BY DECOMPOSITION OF CARBONATE.
850-900°C BROAD ENDOTHERMIC PEAK CAUSED BY DESTRUCTION OF ILLITE & MONTMORILLONITE LATTICE & THE FORMATION OF SPINEL

Figure 21

ClibPDF - www.fastio.com

DIFFERENTIAL THERMOGRAMS OF SAMPLES
From Boring D-5, Job No 2206-T-20
Proposed West Side Freeway
VI-FRE-238-A, B

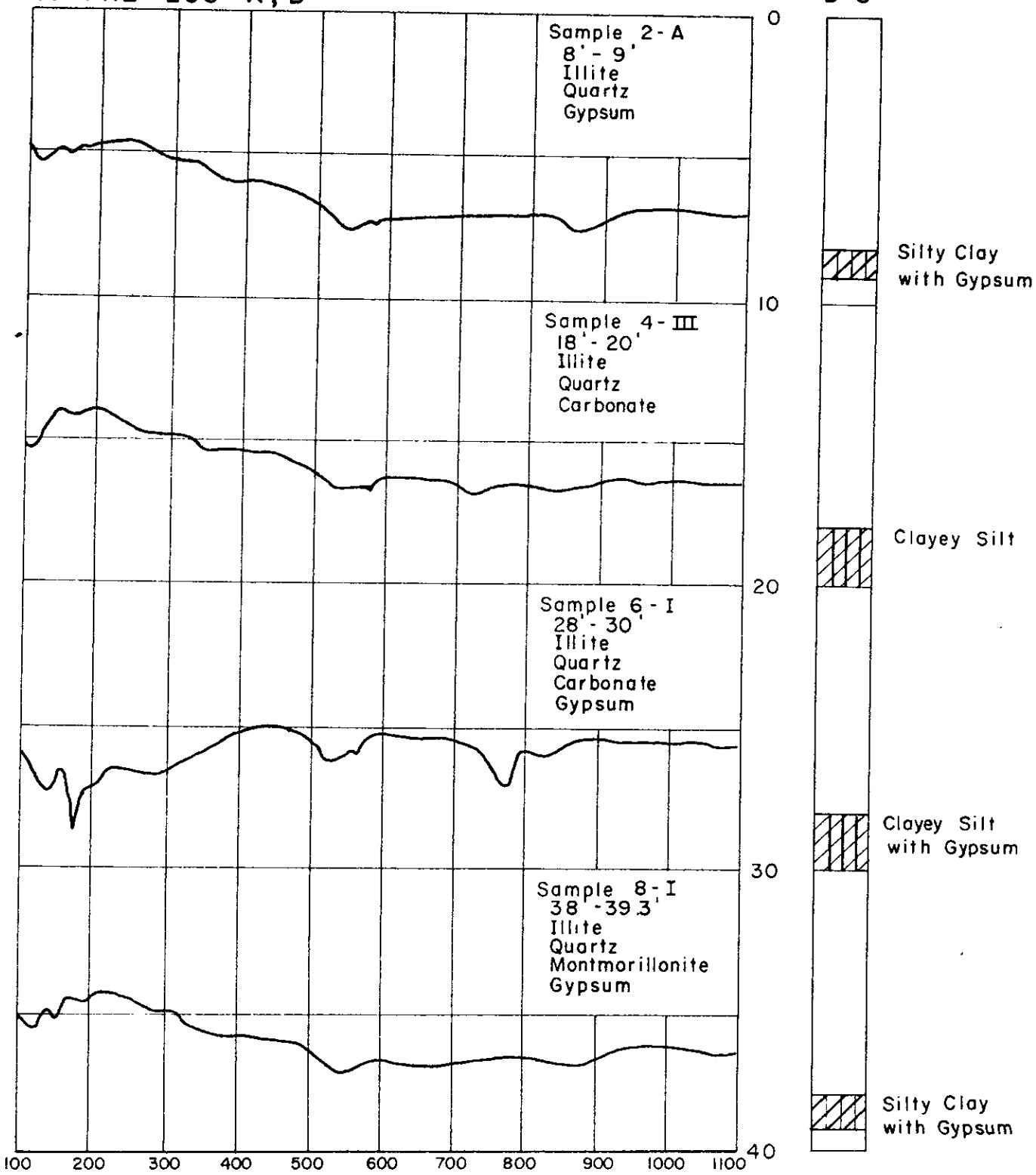


KEY- 100-150°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF ABSORBED WATER.
160-180°C SHARP ENDOTHERMIC PEAK CAUSED BY LOSS OF WATER OF HYDRATION IN GYPSUM.
330-550°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM ILLITE LATTICE.
573°C SHARP ENDOTHERMIC PEAK CAUSED BY QUARTZ INVERSION
640-660°C SLIGHT ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM MONTMORILLIONITE LATTICE.
770-790°C SHARP ENDOTHERMIC PEAK CAUSED BY DECOMPOSITION OF CARBONATE.
850-900°C BROAD ENDOTHERMIC PEAK CAUSED BY DESTRUCTION OF ILLITE & MONTMORILLONITE LATTICE & THE FORMATION OF SPINEL.

Figure 22

ClibPDF - www.fastio.com

DIFFERENTIAL THERMOGRAMS OF SAMPLES
From Boring D-6, Job No 2206-T-20
Proposed West Side Freeway
VI-FRE-238-A, B



KEY- 100-150°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF ABSORBED WATER.

160-180°C SHARP ENDOTHERMIC PEAK CAUSED BY LOSS OF WATER OF HYDRATION IN GYPSUM.

330-550°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM ILLITE LATTICE.

573°C SHARP ENDOTHERMIC PEAK CAUSED BY QUARTZ INVERSION.

640-660°C SLIGHT ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM MONTMORILLIONITE LATTICE.

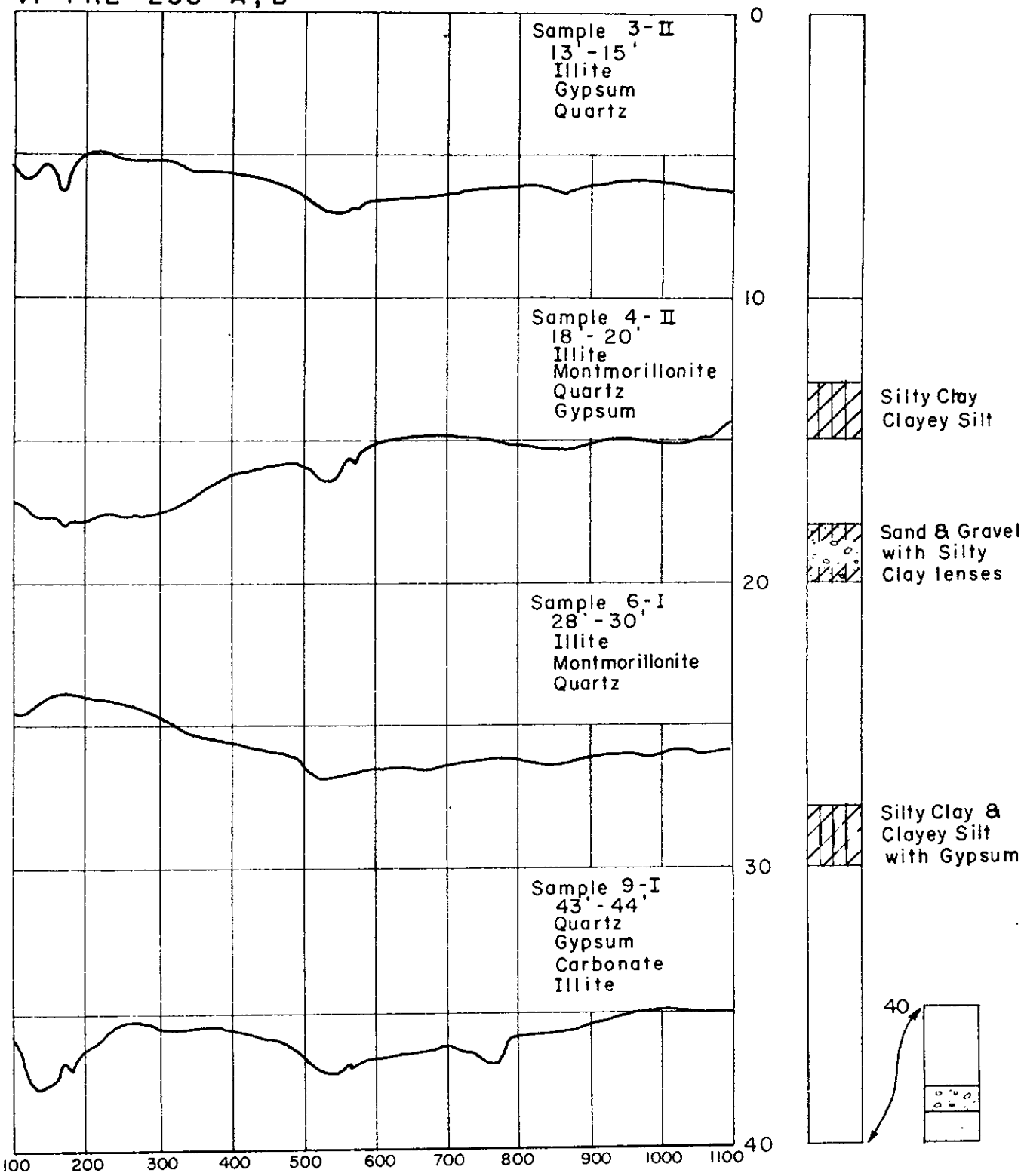
770-790°C SHARP ENDOTHERMIC PEAK CAUSED BY DECOMPOSITION OF CARBONATE.

850-900°C BROAD ENDOTHERMIC PEAK CAUSED BY DESTRUCTION OF ILLITE & MONTMORILLONITE LATTICE & THE FORMATION OF SPINEL.

Figure 23

ClibPDF - www.fastio.com

DIFFERENTIAL THERMOGRAMS OF SAMPLES
From Boring D-7 , Job No 2206-T-20
Proposed West Side Freeway
VI-FRE-238-A, B

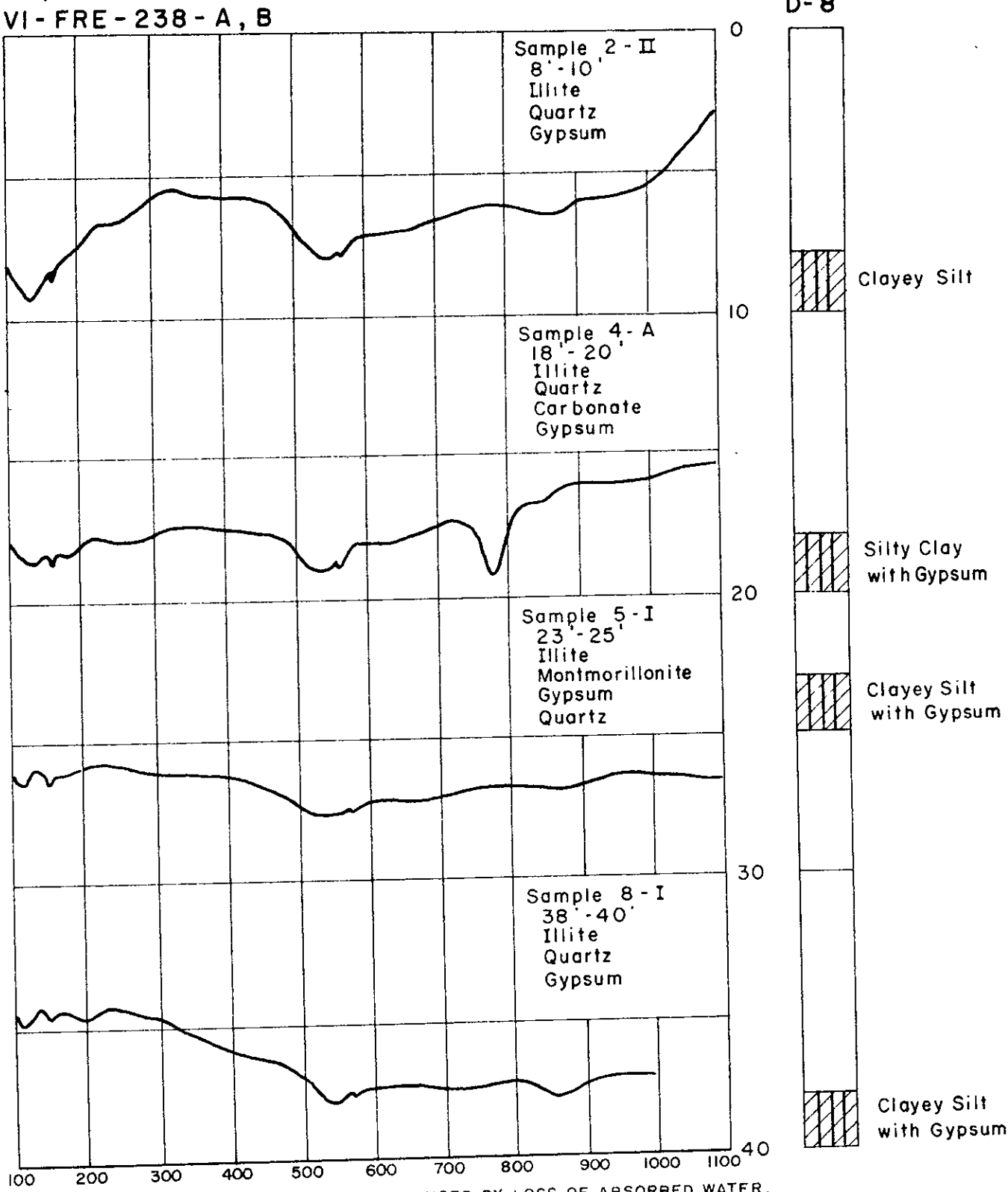


KEY- 100-150°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF ABSORBED WATER.
160-180°C SHARP ENDOTHERMIC PEAK CAUSED BY LOSS OF WATER OF HYDRATION IN GYPSUM.
330-550°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM ILLITE LATTICE
573°C SHARP ENDOTHERMIC PEAK CAUSED BY QUARTZ INVERSION.
640-660°C SLIGHT ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM MONTMORILLIONITE LATTICE.
770-790°C SHARP ENDOTHERMIC PEAK CAUSED BY DECOMPOSITION OF CARBONATE.
850-900°C BROAD ENDOTHERMIC PEAK CAUSED BY DESTRUCTION OF ILLITE & MONTMORILLONITE LATTICE & THE FORMATION OF SPINEL.

Figure 24

ClibPDF - www.fastio.com

DIFFERENTIAL THERMOGRAMS OF SAMPLES
From Boring D-8, Job No 2206-T-20
Proposed West Side Freeway
VI-FRE-238-A, B

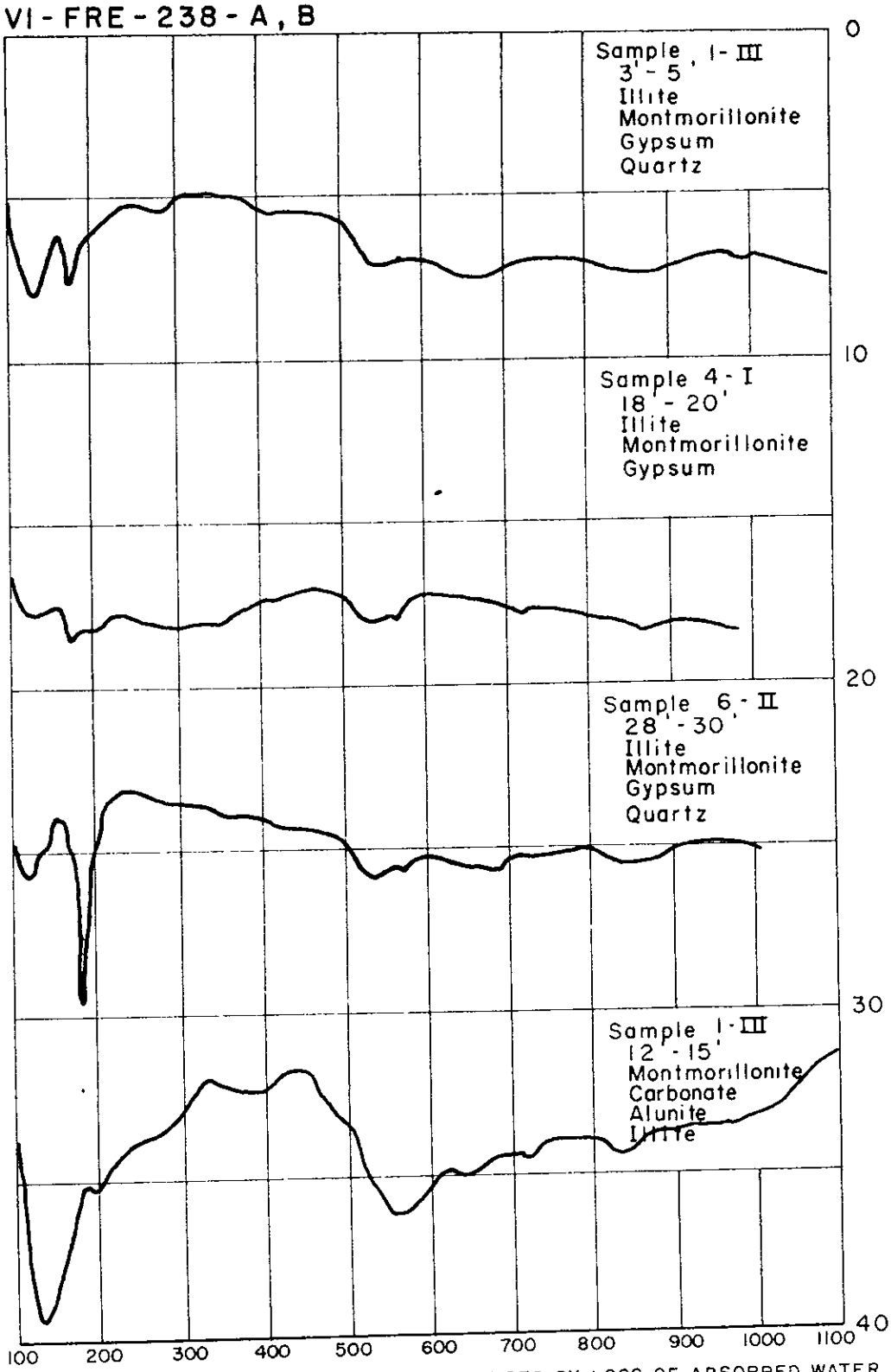


KEY- 100-150°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF ABSORBED WATER.
160-180°C SHARP ENDOTHERMIC PEAK CAUSED BY LOSS OF WATER OF HYDRATION IN GYPSUM.
330-550°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM ILLITE LATTICE
573°C SHARP ENDOTHERMIC PEAK CAUSED BY QUARTZ INVERSION
640-660°C SLIGHT ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM MONTMORILLIONITE LATTICE.
770-790°C SHARP ENDOTHERMIC PEAK CAUSED BY DECOMPOSITION OF CARBONATE.
850-900°C BROAD ENDOTHERMIC PEAK CAUSED BY DESTRUCTION OF ILLITE & MONTMORILLONITE LATTICE & THE FORMATION OF SPINEL.

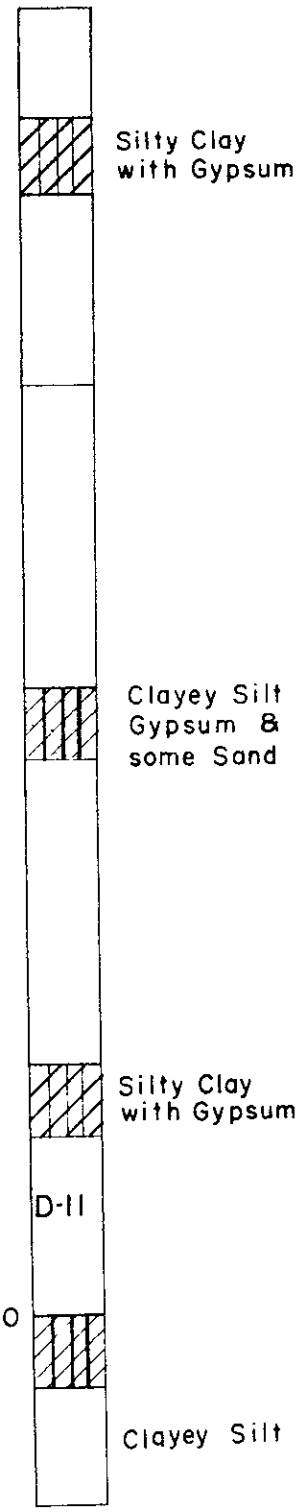
Figure 25

ClibPDF - www.fastio.com

DIFFERENTIAL THERMOGRAMS OF SAMPLES
From Boring D-9 , Job No 2206-T-20
Proposed West Side Freeway
VI-FRE-238-A, B



DESCRIPTIVE LOG



KEY- 100-150°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF ABSORBED WATER.
160-180°C SHARP ENDOTHERMIC PEAK CAUSED BY LOSS OF WATER OF HYDRATION IN GYPSUM.
330-550°C BROAD ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM ILLITE LATTICE
573°C SHARP ENDOTHERMIC PEAK CAUSED BY QUARTZ INVERSION
640-660°C SLIGHT ENDOTHERMIC PEAK CAUSED BY LOSS OF HYDROXYL ION FROM MONTMORILLIONITE LATTICE.
770-790°C SHARP ENDOTHERMIC PEAK CAUSED BY DECOMPOSITION OF CARBONATE.
850-900°C BROAD ENDOTHERMIC PEAK CAUSED BY DESTRUCTION OF ILLITE & MONTMORILLONITE LATTICE & THE FORMATION OF SPINEL.

Figure 26

ClibPDF - www.fastio.com

4-II (18-20') contains montmorillonite and typsum. Sample 6-I (28-30') contains montmorillonite and sample 9-I (43-44') contains gypsum and carbonate.

The basic mineralogical composition of boring D-8 (Figure 25) is illite, quartz and gypsum. Sample 4A (18-20') contains carbonate. Sample S-1 (23-25') contains montmorillonite.

All materials in boring D-9 (Figure 26) contain illite, montmorillonite, gypsum and quartz.

Only one sample was obtained in boring D-11 (Figure 25). It contained illite, montmorillonite, and carbonate as well as alunite, an alteration product of gypsum.

Miscellaneous Materials

Waste material from a World War II magnesium plant located near Modesto was used as subbase material for a portion of X-SJ-4-E. Some distress was noted, and samples of the material were sent to the Materials and Research Department. Differential thermal analysis indicated that gibbsite, a magnesium hydroxide was present, and being formed by reaction of metallic magnesium in the waste material with water. Several tests were made. The results are shown in Figure 27. The first test was made on sack run material. The pronounced endothermic peak at 440° centigrade occurs when the hydroxyl ion is driven from the gibbsite by heating. The pronounced exothermic peaks at 610 - 630° centigrade are the result of the oxidation of the metallic magnesium. The second test was made of a white powder occurring around the metallic magnesium. It gave a similar but more pronounced curve than the first test. Test three was made on a white powder formed by the oxidizing of metallic magnesium with a blow pipe. The endothermic peak is not as well defined as in the first two tests. This is thought due to the lack of crystallinity of the material, but the exothermic peak occurring at 600° C. is present and very well developed. The wider extent of the peak is thought due to the greater amount of magnesium present in the material.

A series of samples from piezometer borings in Contra Costa County was examined by differential thermal analysis. The samples showed considerable variation in mineralogical content.

In boring D-1 - 7-B (Figures 28 & 29) mineralogical constituents identifiable are: siderite, montmorillonite and illite. There also seems to be a considerable amount of organic matter present.

Sample D-2 - 3A contains illite, and montmorillonite. The endothermic deflections are pronounced as is the exothermic illitic dome.

ClibPDF - www.fastio.com

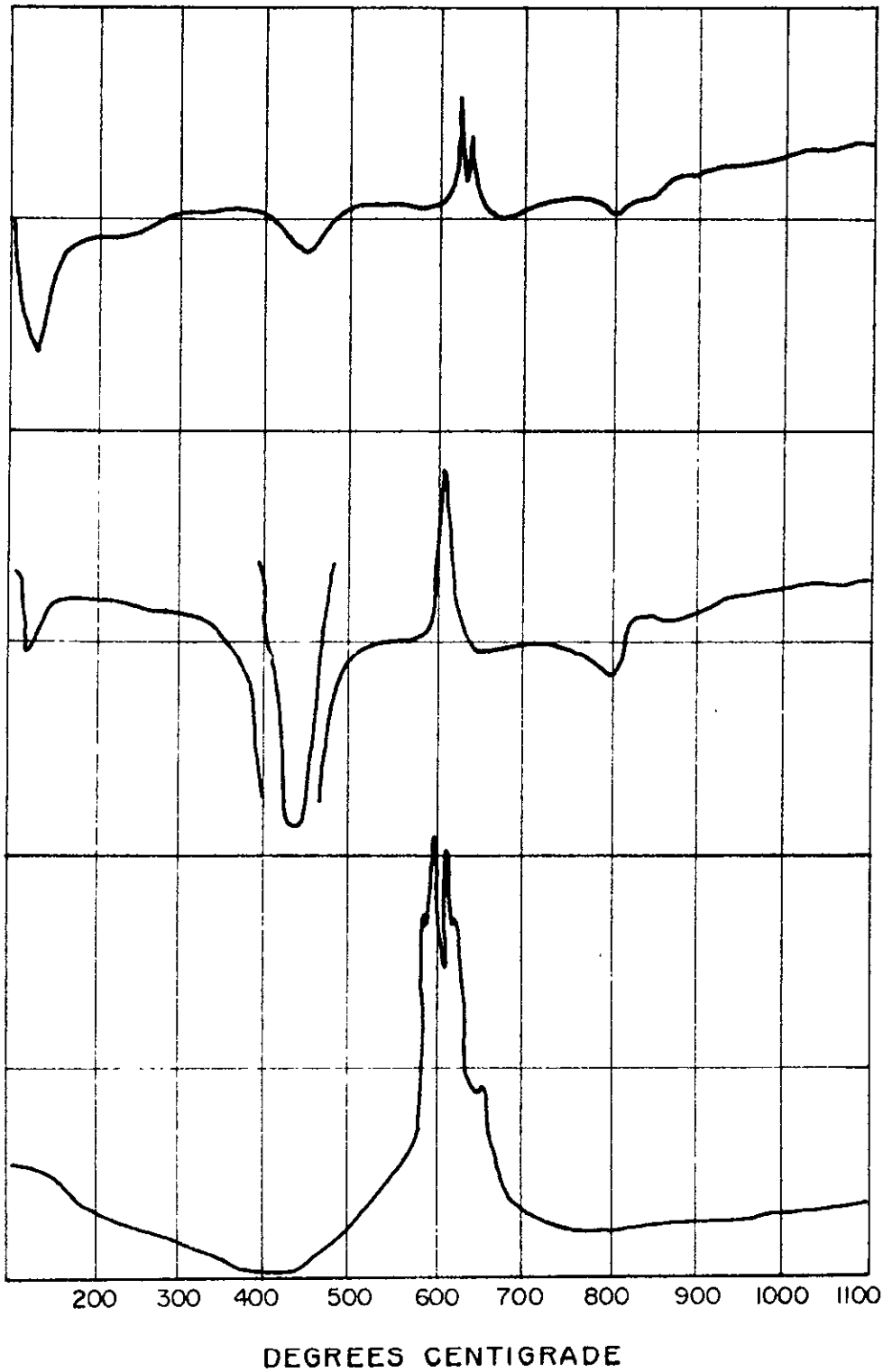


Figure 27

ClibPDF - www.fastio.com

Sample D-6 - 4A shows a definite quartz inversion at 573° centigrade, and also a slight suggestion of an illite endothermic reaction as well as a slight exothermic dome.

Sample D-8 - SIII contains siderite, montmorillonite and illite. The endothermic reaction of illite is obscured by the siderite exothermic peak occurring at 550°C., but the exothermic dome at 900°C. is present. A slight endothermic and curve between 600° and 700° C. indicates the presence of montmorillonite.

Sample D-9 - 2A contains illite, and montmorillonite. The illite endothermic peak occurring about 500°C. is not too well developed, and it occurs at a rather low temperature. The exothermic dome is not too pronounced and it seems to indicate a poorly developed illite. The montmorillonite endothermic curve occurs between 600° and 700°C. It is rather poorly defined .

Sample D18 - 3VIII contains illite, montmorillonite, quartz and siderite. The 500° - 600°C. endothermic curve for illite is absent but the exothermic dome occurs at 900°C. A strong quartz inversion occurs at 573°C. The presence of siderite is indicated by anexothermic peak at 450°C. Montmorillonite is indicated by the endothermic peak at 640 - 670°C.

Sample D18 - 5II consists of illite and montmorillonite. It has a characteristic exothermic peak at 450°C. an endothermic peak at 550°C. and an exothermic dome at 900°C. The presence of montmorillonite is indicated by the ill defined endothermic peak from 640° - 700°C.

Sample D29 - 1III contains illite and siderite. The 550°C. endothermic peak is absent, but the presence of a strong exothermic dome at 900°C. is indicative of illite. The siderite exothermic peak at 530°C. marks the illite endothermic peak completely.

Sample D-32 - 4A contains illite and montmorillonite. The illite curve has well developed inflections and it is very definitive. The montmorillonite curve is definite in the 650° - 700°C. range.

Sample D74 - 10A contains illite and montmorillonite. The illite endothermic curve at 550°C. is well defined as is the exothermic doublet and dome at 900°C. The montmorillonitic endothermic peak at 640 - 700°C. is prominate.

Sample D-77 - 3A contains illite. The illite endothermic curve at 550°C. is well defined as is the exothermic doublet and dome at 900°C.

ClibPDF - www.fastio.com

Sample D-77 - 6A contains illite and quartz. The illite endothermic curve at 550°C. is well defined as is the exothermic doublet and dome at 900°C. The quartz inversion at 573°C. is pronounced.

ClibPDF - www.fastio.com

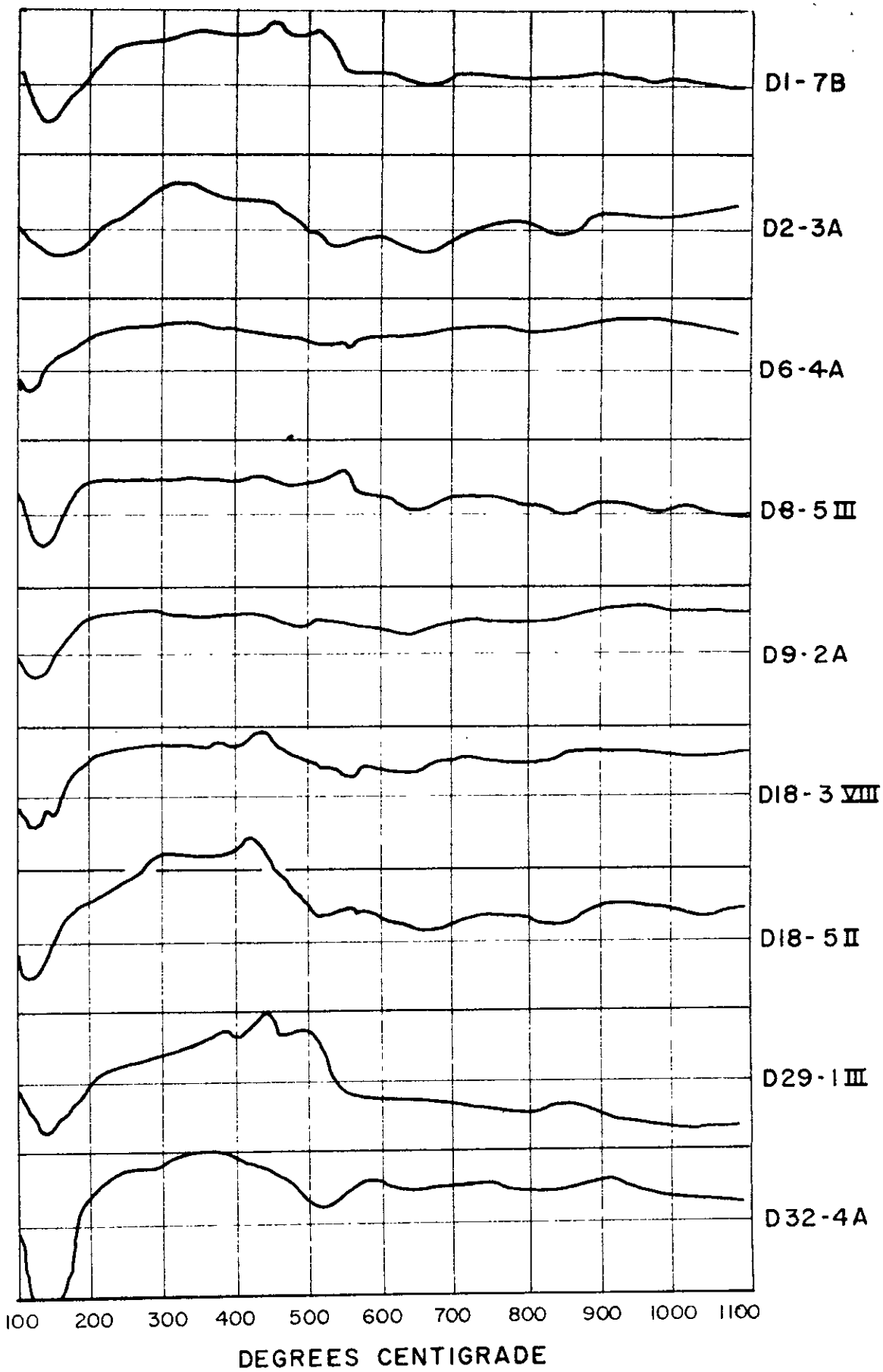


Figure 28

ClibPDF - www.fastio.com

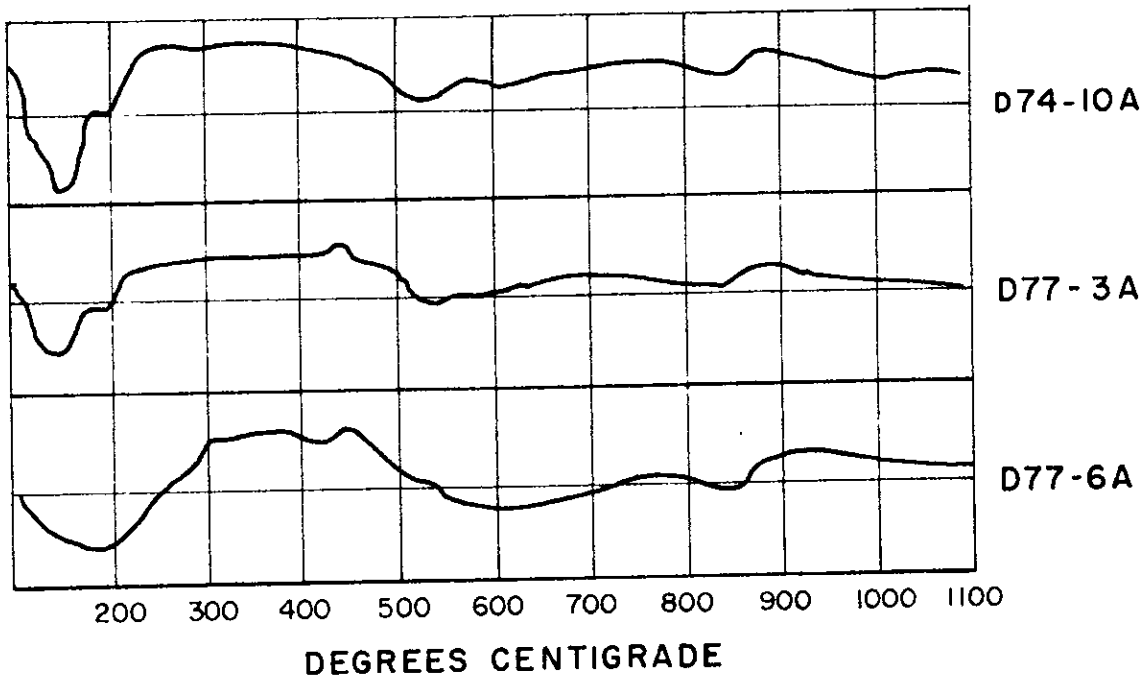


Figure 29

ClibPDF - www.fastio.com

REFERENCE CHART OF CLAY MINERALS

COMPILED FROM CLAY MINERALS, AMERICAN PETROLEUM INSTITUTE

RESEARCH PROJECT 49

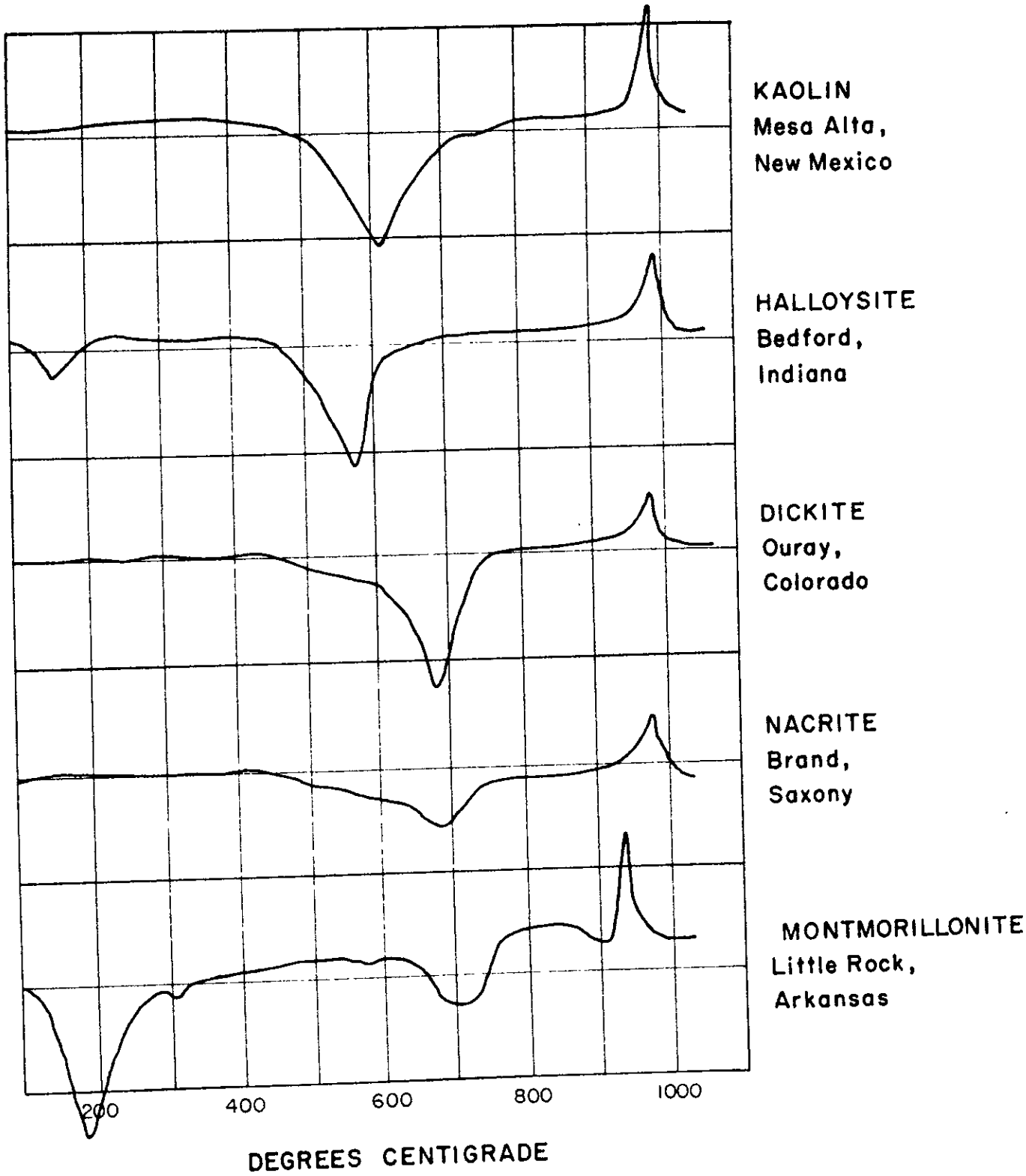


Figure 30

ClibPDF - www.fastio.com

REFERENCE CHART OF CLAY MINERALS

COMPILED FROM CLAY MINERALS, AMERICAN PETROLEUM INSTITUTE RESEARCH PROJECT 49

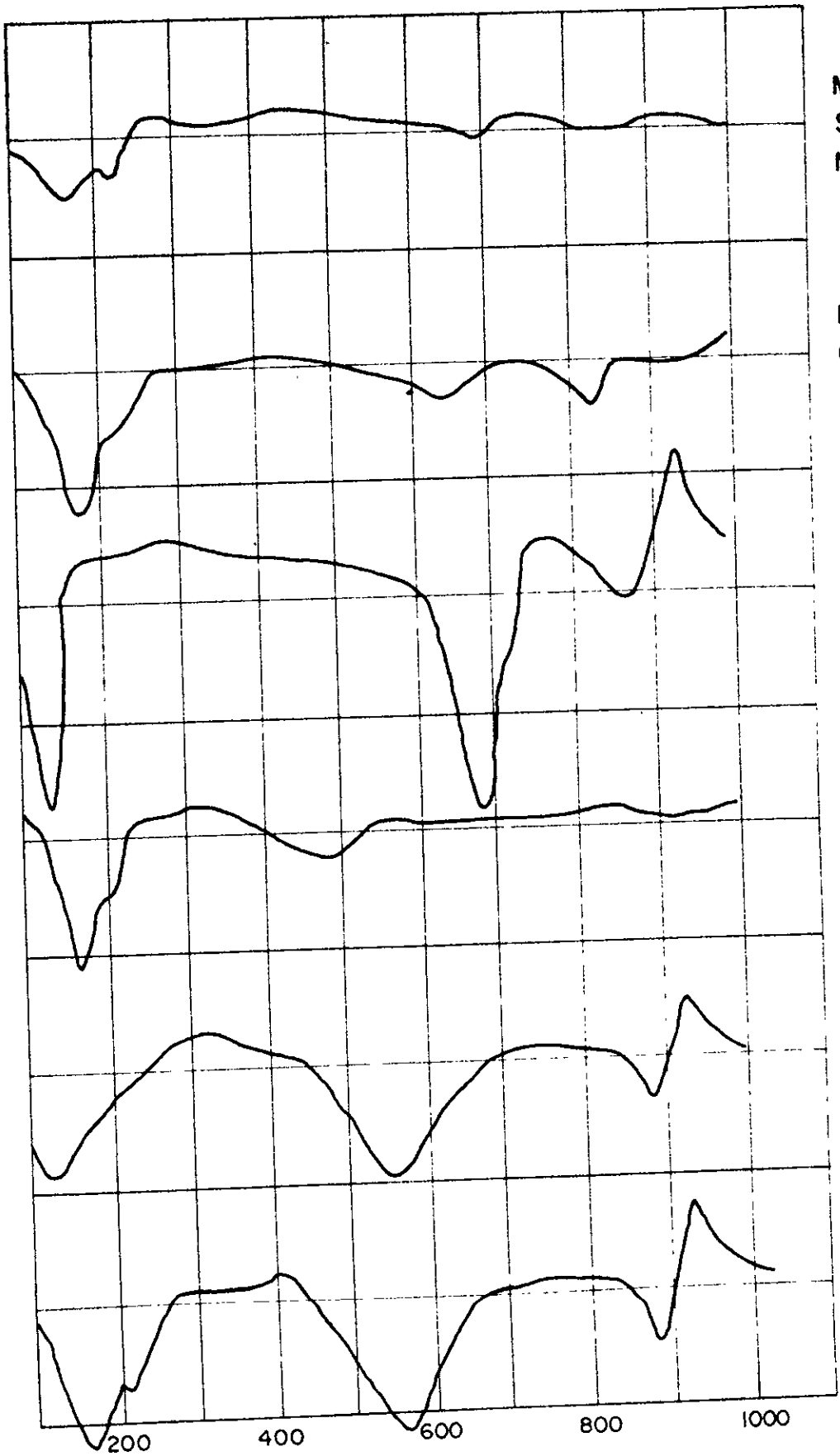


Figure 31

ClibPDF - www.fastio.com

QUANTITATIVE DETERMINATIONS OF CLAY MINERALS

The quantitative determination of the amount of a clay mineral present in a soil sample has not been discussed in this report. A survey of the literature on the subject indicates most workers feel that quantitative differential thermal analysis is not too reliable, and that X-ray techniques furnish a more reliable and rapid method of quantitative determination.

Quantitative differential thermal analysis seems most workable in mixtures of two or three chemicals in an inert substance. In industry, it is used as a rapid method of determining the amount of kaolin in raw material for the ceramics industry, and as a quality control on raw materials. The literature on differential thermal analysis contains numerous series of articles in which a writer will describe a method of quantitative differential thermal analysis, and several other writers will write in great detail relating how it does not work as described by the initial writer.

Among the various papers on the theory of quantitative differential thermal analysis, several features are more or less common (1) conductivities and heat capacities are considered as constant and (2) differential temperature is plotted against time or reference temperature. These two assumptions are necessary for development of an operating method but the assumptions could be fallacious in that there is a possibility of a change in heat capacity during and after a phase transformation as in dehydration where some mass is lost. Heat conductivities are assumed constant, and if the heat exchange occurs under black body environment, conduction is more or less independent of the material. The size of the sample is of great importance: due to the complications introduced by heat conduction, the dimensions of sample should be as small as feasible.

Conditions inherent in any soil sample offer the greatest problems in quantitative differential thermal analysis. The materials vary widely in crystallinity and particle size. Variations in percentage composition of 5 to 30 percent as described in the literature due to difference in particle size and crystallinity of the same material. Coatings of the grains of minerals can cause a difference of as high as 50 percent. The presence of extraneous material such as sodium chloride has cause actual changes in the apparent ratio of clay mineral to inert substances in a soil.

Conclusions

Differential thermal analysis offers a rapid method of determining the type of a clay mineral or other thermally active substance present in a soil sample. Expansive clays

ClibPDF - www.fastio.com

can be readily identified. The crystalline condition of some of the clay mineral constituents of soils can be determined and behavior of soils in construction can be explained.

Examination of suites of samples from borings can permit the grouping of zones of similar soil constituents and the possible explanation of soil behavior factors such as permeability and bearing.

ClibPDF - www.fastio.com

BIBLIOGRAPHY

GRIM, Ralph E. - Clay Mineralogy

KERR, Paul F. (Editor) Preliminary Reports Reference Clay Minerals American Petroleum Institute Research Project 49

LAMBE, T. William - Differential Thermal Analysis

PASK, Joseph A. & TURNER, Mort D. - Clays & Clay Technology

ROWLAND, Richards A. - Differential Thermal Apparatus Analysis

ROWLAND, Richards A. & JONAS, Edward C. - Variations in Differential Thermal Analysis Curves of Siderite

SMOTHERS, W. J. & CHIANG, Y. - Differential Thermal Analysis

SOVERI, U. - Differential Thermal Analyses of Some Quaternary Clays of Fennoscaudia

SWINGFORD, Ada & PLUMMER, Norman (Ed) - Clay and Clay Minerals

ClibPDF - www.fastio.com