MINERALOGICAL STUDIES OF GONDWANA SEDIMENTS FROM KORBA COALFIELD, MADHYA PRADESH, INDIA. PART III—CLAY MINERALOGY

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ABSTRACT

A Lower Gondwana succession of Korba Coalfield is studied for clay minerals in the clay fraction ($<2\mu$) by X-ray diffraction analysis and semi-quantitative estimation has been done to elucidate the climatic history of the sequence.

On the basis of clay mineral assemblage and lithology five mineralogical zones with specific palaeoclimates are established. Talchir sediments (zone A) of glacial environment are characterized by montmorillonite as main clay mineral, formed during pre-glaciation period. Lower-most part of the Barakar succession (zone B) is marked by the abundance of montmorillonite, formed during post-glaciation period in a cold temperate, humid climate. In zone C) montmorillonite in minor amounts occurs with kaolinite; climate is temperate. In zone D) lithology controls the clay mineralogy. Sandstones (well-drained channel sand facies) contain kaolinite along with chlorite and illite; shales (poorly drained flood basin facies) contain kaolinite and mixed layer mineral. For this zone a warm temperate to subtropical climate is suggested. The topmost zone (zone E) contains kaolinite in dominance with minor amounts of illite and chlorite formed in tropical, humid climate.

INTRODUCTION

Gondwana sediments represent thick succession of detrital sediments deposited in isolated, faulted basins. The sediments are dominantly sandstones with shale and coal intercalations. In these faulted basins sedimentation started in glacial environment with very cold climate; later on climate became warmer, well suited for the establishment of vegetation, which provided material for the genesis of coal. Along with the change in climate, domain of sedimentation also changed, i.e. sedimentary environment changed from glacial to fluvial. This change in climate and domain of sedimentation is well expressed in the lithology as well as in the vegetational history. Actually, the subdivision of basal part of Lower Gondwanas, i.e. Talchir and Barakar is based on this change in climate and domain of sedimentation: Talchir sediments are mainly glacial deposits, while the Barakar sediments represent fluvial deposits.

A detailed study of the mioflora in a borehole succession of Korba Coalfield revealed that during deposition of the Talchir and Barakar sediments climate changed with time (Bharadwaj & Srivastava, 1973). General mineralogical studies also suggest that change from glacial to fluvial environment is well-marked (Singh & Sharma, 1973), so does the study of quartz grain surface features (Singh, 1974).

Clay minerals are formed mainly during soil genesis and weathering, which are directly controlled by climatic conditions. Thus, study of clay minerals in a geological succession may provide clues to the palaeo-climates. It was provoking to study the control of climate on the clay mineralogy of the Gondwana sediments of Korba Coalfield. The main aim of the present investigation has been to study, with the help of X-ray diffraction analysis,

Geophytology, **4** (2) : 196-220

the distribution of clay minerals in the Gondwana sediments of Korba Coalfield, and to obtain an insight in the control of climate on the genesis of clay minerals.

MATERIAL

The samples studied for clay minerals were obtained from a borehole of Korba Coalfield (Borehole No. NCKB-19). Same profile has been also studied for other parameters (Bharadwaj & Srivastava, 1973; Singh & Sharma, 1973; Singh, 1974). The samples were taken from different lithological units of the palynological zones (see Singh & Sharma, 1973).

It is believed that all the samples studied are devoid of any weathering effects during modern times. The shallowest sample was taken at a core depth of 18.28 m. Weathering processes alter the clay minerals; it has been observed in several studies that samples from subsurface and outcrop show quantitative or even qualitative differences in the clay mineral composition (e.g. Glass, 1958; Okrajek, 1965). In the present study effect of weathering during modern times can be regarded as negligible.

Study of Clay Minerals

Historical—Study of fine-grained fraction (clay fraction) of the sediments has attracted the scientists from the early days. The earliest works were based on chemical analysis (for example, Wülfing, 1900; Vogt, 1906). Later, after the introduction of X-ray diffraction technique detailed study of clay started. But the major work has been done during sixties and seventies which has contributed much to our understanding of clay minerals. Brindley (1951), Brown (1961), Grim (1968) and Millot (1970) are the important books on the various aspects of clay mineralogy.

During the study of clay minerals a major controversy came into existence: detrital vs. neoformed clays, whether clay minerals are always detrital in origin, or whether they are always neoformed in the place of sedimentation, or later during diagenesis. It can be said that both the processes exist in nature, but the detrital clay minerals are much more abundant than the neoformed clay minerals. Moreover, process of transformation is also active which causes transformation of land derived material into new minerals under the influence of depositional environment.

Genesis of Clay Minerals—Clay minerals are the most stable alumino-silicates on the Earth's surface, and they are extensively formed within hydrosphere. During weathering various alumino-silicates, e.g. felspars, mica, pyroxenes are broken and changed to various clay minerals, depending upon the weathering conditions. As long as the physico-chemical conditions of the hydrosphere remain unchanged, clay minerals remain stable. Upon burial, during metamorphism clay minerals are changed to other alumino-silicates, e.g. transformation of montmorillonite into illite, and ultimately to sericite.

Processes of Weathering and Soil Formation—On the continents, clay minerals are primarily produced during weathering and soil formation as a result of alteration of silicates. For the proper understanding of genesis of clay minerals a clear insight into the geochemistry of weathering and pedogenesis is absolutely necessary. The main phenomena during weathering is hydrolysis, which involves the phenomena of ion exchange between two media, water and silicate. H^+ ions of water cause expulsion of cations from the silicate structure. Thus degree of leaching would depend upon the availability of H^+ ions from water. Other important factors controlling the clay mineral genesis during weathering and pedogenesis are lessivage and vegetation. The term lessivage denotes a complex of processes involved in removing material from the upper part of soil profile; it includes both mechanical transport and removal of ions in solution (MILLOT, 1970). Role of vegetation is very important during soil genesis. If vegetation is dense, erosion is restricted, development of soil profile is well-marked, soluble ions are quickly removed by percolating waters. If vegetation is destroyed the soil cover is eroded, and the material is deposited in the basin of sedimentation. All these factors, i.e. percolating waters, lessivage, and vegetation are directly controlled by climate, making the climate to be the prime factor controlling the clay mineral genesis on the continents. During weathering water and temperature are the main controlling factor. It leads to the fact, that weathering is more complete in tropical and temperate climates than in glacial and periglacial climates. During weathering process following set of clay minerals are produced (MILLOT, 1970):

(i) Clay minerals neoformed from silicates of parent rock: illite and chlorite. Illite is derived from sericite, felspars and alumino-silicates. Chlorite is derived from ferromagnesian minerals.

(ii) Clay minerals transformed from earlier ones: vermiculite, mixed layer mineral, montmorillonite. Illite and chlorite alter into various mixed layer minerals and to montmorillonite.

(iii) Clay minerals neoformed from the products of hydrolysis under special climatic conditions: montmorillonite, and kaolinite. If drainage is restricted montmorillonite is neoformed by joining of various ions in solution. If drainage is good and various cations are removed kaolinite is neoformed.

SOILS OF VARIOUS CLIMATIC ZONES

Soils in cold and desert climate—Soils of cold and desert climates are often referred as non-evolved soils. These climates are inert for the neoformation of clay minerals; inherited clay minerals from the parent rock are dominant. If soils are derived from crystalline rocks illite and chlorite are dominant minerals, formed from mica + felspar and ferromagnesian minerals respectively. If derived from sedimentary parent rocks, they exhibit the same clay minerals as in the sedimentary rocks.

Soils in temperate climate—In temperate climate evolution of clay minerals does take place. However, as the temperature is low, and the humidity is also low, hydrolysis of silicates is slow and not complete. Clay mineral studies in soils of temperate climate have revealed that following sequence of transformation takes place (MILLOT, 1970)

$$\begin{tikzcd}
\text{Illite} \arrow[r] & \text{Illite-Vermiculite} \arrow[r] & \text{Vermiculite} \arrow[dd, bend left] \\
& \text{Chlorite} \arrow[r] & \text{Chlorite-Vermiculite} \arrow[u] \\
& \text{Montmorillonite} & \text{Vermiculite-Montmorillonite} \arrow[l]
\end{tikzcd}$$

Thus we see that two of the most common inherited clay minerals, chlorite and illite are transformed ultimately to montmorillonite. This change can stop at any stage, whenever equilibrium is attained. Important studies on the genesis of clay minerals in temperate climate are by WALKER (1950), BROWN AND JACKSON (1958), BHATTACHARYA (1962), DROSTE *et al.* (1962), CAMEZ (1962), MITCHELL (1962), MILLOT AND CAMEZ (1963), GJEMS (1967).

Soils in warm climate—Soil processes are active in warm climatic zones. Important are the ferruginous soils in warm climate. Due to increased temperature and availability of water hydrolysis is active. Kaolinite is often the main mineral in areas with strong weathering and good drainage. In areas of poor drainage montmorillonite becomes prominent mineral. Illite is sometimes abundant in little evolved soils (MILLOT, 1970).

Soils in tropical humid climate—Tropical humid climate has relatively high temperature, and abundant water is available for weathering and hydrolysis. Both physical and chemical processes are vigorously active. Soils are commonly referred to as lateritic soils (MILLOT, 1970). Important contributions to the laterite soil processes are by AUBERT (1954), MAIGNIEN (1958) and LAJOINIE AND BONIFAS (1961).

Lateritic soil genesis results into complete hydrolysis of the silicates and the ions, e.g. Si, Al, Mg, Ca, K and Na are released. The Mg, Ca, K, and Na are quickly removed by percolating waters. Fe, Al, and Si are only partially removed, the rest combines to produce iron-oxides (goethite, hematite), hydrated Al-oxide (gibbsite, boehmite), and aluminium silicate (kaolinite). Gibbsite is formed if drainage is excellent, removing the silica, only alumina is left behind. In well-drained environment removal of silica is partial, rest of silica combines with alumina to produce kaolinite. Locally, in rare cases, in poorly-drained environment where cations are present montmorillonite may be formed. Nevertheless, kaolinite is the most characteristic clay mineral of lateritic soils. In tropical climate, because of heavy rains and high temperatures vegetation is dense, and soil development is extensive. In such areas tens of metre thick soil cover may develop with kaolinite as the predominant mineral.

PROCESSES OF CLAY MINERAL GENESIS

Under the physico-chemical conditions of the hydrosphere there are three important processes in the genesis of clay minerals (MILLOT, 1970): (*a*) Detrital inheritance, (*b*) Transformation, (*c*) Neoformation.

Detrital inheritance—The term denotes detrital clay minerals which remain inert in the new environment because of their stability.

During weathering and pedogenesis detrital clays are inherited from parent rocks. If the parent rocks are crystallines or metamorphics, e.g. granite, schist, gneiss illite and chlorite are inherited. On the other hand, if the parent rocks are sedimentaries, e.g. shales, marls a variety of clay minerals are inherited depending upon the clay mineral suite of the parent rocks. Inherited clay minerals from parent rocks, and clay minerals formed during soil genesis are transported to basin of deposition. Important minerals produced during soil genesis are kaolinite, and montmorillonite.

Normally, inherited clay minerals do not undergo any modification in the basin of deposition because of their stability and non-aggressive milieu of the basin. However, if the basin of deposition possesses marked chemical character, e.g. hypersalinity, hyperalkalinity, inherited clays may be transformed under the new set of physico-chemical conditions.

Transformation—The term transformation incorporates changes that modify a clay mineral without affecting its basic crystal structure. Transformation of clay minerals can take place during pedogenesis, sedimentation and diagenesis. Transformation is achieved by degradation or aggradation.

Degradation—It may be regarded as negative transformation causing loss of substance. During degradation stable minerals, i.e. illite and chlorite are attacked and changed to open lattice minerals with variable basal spacings (like mixed layer minerals, montmorillonite) which are more stable in an environment rich in water. A common sequence of transformation in nature is as follows (MILLOT, 1970):

$$\xrightarrow{\text{Degradation}}$$

Illite ⇌ I-V mixed layer ⇌ vermiculite ⇌ V-M mixed layer ⇌ montmorillonite

$$\xleftarrow[\text{Aggradation}]{}$$

The change from stable minerals to open minerals is very gradual, and takes place step by step. For example, if mica is degraded first of all K-ions are replaced by water molecules, the bond between the layers are loosened and the spacing between them increases. Changes also take place within the layers: ferrous ions are oxidized to ferric ions causing breaking of lattice, oxygen ions are replaced by hydroxyl ions. Mg ions migrate between the layers to balance the vacant charges. Gradually, mixed layer minerals are formed, and with increasing degradation montmorillonite is formed. On X-ray diffractograms this change is marked by broadering of 10 Å peak, appearance of diffuse peaks between 10 and 14 Å (mixed layer minerals), 10 Å peak becomes indistinct and a well-formed peak appears at 12-14 Å unaffected upon glycolation (vermiculite), 14 Å peak affected upon glycolation (montmorillonite). JACKSON *et al.* (1954), JACKSON (1959) observed this sequence of changes in the soil profiles:

Mica ——→ Intermediate stage ——→ Vermiculite ——→ Montmorillonite

Aggradation—It is positive transformation causing gain of substance. Open and unstable minerals with variable basal spacings are changed to minerals similar to mica and chlorite. Aggradation causes reconstruction of degraded minerals into normal clay minerals. This process occurs under conditions where solution rich in cations is available. This process is active in the basin of deposition and afterwards during diagenesis.

Aggradation involves fixation of cations and reorganization of the crystal lattice. By this process mixed layer minerals are changed to more regular minerals like illite and chlorite.

Neoformation—The term neoformation denotes genesis of clay minerals by joining of various ions dissolved in water under suitable physico-chemical conditions. Neoformation can take place during soil genesis, sedimentation, and diagenesis.

Presence of water causes leaching of minerals, hydrolysis and dissolution takes place. If ions are removed quickly, the milieu becomes acidic, neoformation by substraction takes place, e.g. kaolinite formation. If water discharge is low, and ions are not removed, milieu becomes alkaline, neoformation by substraction in an alkaline environment takes place, e.g. sericitization of felspars, chloritization of ferromagnesian minerals, three layer minerals are formed. If drainage is restricted, dissolved ions are concentrated. Dissolution and hydrolysis is slowed down, the milieu becomes markedly alkaline and neoformation by addition takes place, e.g. montmorillonite, attapulagite-sepiolite, chamosite.

Neoformation of clay minerals is totally independent of SiO_2/Al_2O_3 ratios of the original silicate minerals providing silica and alumina for the process. During weathering in low-leaching conditions cations and silica are abundant, minerals with high SiO_2/Al_2O_3 ratio, e.g. sericite, montmorillonite are formed ($SiO_2/Al_2O_3 > 2$). Weathering in moderately leached conditions produces kaolinite, due to joining of silica with alumina ($SiO_2/Al_2O_3 = 2$). If leaching is vigorous silica is removed along with cations, alumina is left behind, and crystallizes in situ as residual deposit of gibbsite ($SiO_2 = 0$).

In confined environment products of hydrolysis accumulate, cations and silica are abundant, alumina is rare. The result is formation of clay minerals with low alumina content. Thus, degree of leaching and lessivage is most important factor controlling the neoformation of clay minerals.

Neoformation during weathering and pedogenesis—Following are the more important reactions leading to clay mineral neoformation:

(1) Sericite is formed from felspars in the initial stages of weathering. Alkaline and alkaline earth elements are removed, maintaining an alkaline milieu in which mica is formed. Presence of Ca^{2+} ions favours the formation of mica lattice.

(2) Chlorite is formed from ferromagnesian minerals. During hydrolysis in an alkaline milieu ferromagnesian minerals are broken and organized preferentially into chlorite.

(3) Montmorillonite develops in soils where hydrolysis occurs, but drainage in poor and milieu is alkaline.

(4) Kaolinite develops in soils with increased leaching, and acidic milieu.

Neoformation during sedimentation—During sedimentation kaolinite cannot be formed. It is a typical mineral formed on the continents. Illite and chlorite may be formed in minor amounts. However, important minerals formed in the basin of deposition are glauconite, attapulgite, sepiolite, montmorillonite,

Neoformation during diagenesis—Kaolinite commonly develops during diagenesis of permeable sandy sediments, which are subjected to circulation of acidic pore fluids. Acidic fluids cause hydrolysis of silicates, e.g. felspars, mica ions are released, silica and alumina combine to form kaolinite. In more advanced stages of diagenesis illite and chlorite are formed; kaolinite changes to sericite (DUNOYER DE SEGONZAC, 1970).

To sum up the clay genesis, it can be said that detrital inheritance is the major phenomena to provide clay minerals in the hydrosphere during sedimentation. Detrital inherited clay minerals are brought from provenance into basin of deposition. These clay minerals are transformed or neoformed only if the basin of sedimentation has a clearly marked chemical character, e.g. genesis of attapulgite-palygorskite in alkaline milieu. Mostly, inherited detrital material is left inert in the basin of deposition. Later, during diagenesis, depending upon the physico-chemical conditions, and composition of pore fluids transformation or neoformation of clay minerals may take place.

MAJOR GROUPS OF CLAY MINERALS AND THEIR STRUCTURE

The accepted definition of the clay minerals is that "Crystalline clay minerals are hydrated silicates with layer of chain lattices consisting of sheets of silica tetrahedra arranged in hexagonal form condensed with octahedral layers; they are usually of small particle size."

Clay minerals are thin flakes, their lattice is built up of sheets of cations, e.g. silica, aluminium, magnesium, ion. Two basic structural units are involved in the lattice of clay minerals: octahedral unit, and tetrahedral unit (For details see GRIM, 1968).

In an Octahedral unit each cation, i.e. Al, Fe, Mg is surrunded by 6 oxygen or hydroxyl ions in octahedral coordination. In a tetrahedral unit each silica ion is surrounded by 4 oxygen or hydroxyl ions in tetrahedral coordination. Several octahedral and tetrahedral units join together to produce octahedral and tetrahedral sheets respectively.

The main groups of clay minerals are based on how these tetrahedral and octahedral sheets are stacked together. In the present study following clay mineral groups have been identified: kaolinite, illite, chlorite, montmorillonite, and mixed layer minerals. Other important groups of clay minerals not encountered in this study are vermiculite group, attapulgite-sepiolite group. **Brown** (1961) and **Grim** (1968) give a detailed account of the structure of the clay minerals.

Kaolinite group—The term originates from a Chinese hill where kaolin was quarried for centuries. Kaolinite, like other clay minerals is a phyllosilicate—a sheet silicate. Each layer of kaolinite is made up of a tetrahedral and an octahedral sheet. Such layers are stacked one above the other at a periodicity of 7 Å. In other words, kaolinite group is characterized by 1:1 elementary layers, i.e. a single silica tetrahedral sheet and a single alumina octahedral sheet is combined as an unit. The charges are balanced, and there is very little substitution of other ions for Al.

Illite or mica group—The term illite is used to designate all the clay minerals with crystal structure similar to that of micas. The mica structure is made up of three sheets, i.e. an octahedral sheet is embedded between two tetrahedral sheets (2 : 1 layers). Every fourth tetrahedron of the tetrahedral sheet is occupied in the centre by Al-ion, which results in the deficit of charge for the sheet. This deficit of charge is counter-balanced by large ions, mostly K, which occupies the position between the unit layers. The illites, i.e. mica minerals in clay fraction of the sediments are marked by a deficit of K ions.

The basal spacing is about 10 Å. The term illite is used to denote all the 10 Å minerals in a clay fraction which do not expand upon glycolation. There are several polytypes of mica-dioctahedral, trioctahedral, IM, IMd etc.; however, they are difficult to determine.

Montmorillonite group—Montmorillonites are clay minerals very similar to micas in crystal structure, but bond between the layers are very weak. This group is also designated as smectites. They are trimorphic minerals like mica (2:1 layers). The spacing depends upon the nature of ion and number of water molecules present between the silicate units, usually one or two molecules of waters are present. Mostly basal spacing is 14 Å. The water molecules can be replaced by organic compounds, and the spacing may vary. Upon various treatments spacing may change between 10-17 Å. These minerals therefore are said to possess expandable lattice.

Chlorite group—The chlorite minerals are mostly known from metamorphic rocks. However, they may be common as clay minerals in sedimentary rocks. Chlorites are regarded as tetramorphic minerals (2 : 1 : 1 layers) : Mica layers are bonded by sheets with a brucite structure. The basal spacing is about 14 Å. Chlorites can be both tri-octahedral and dioctahedral. Substitution of ions in tetrahedral and octahedral mica sheets, as well as in brucite sheets are common and varied, giving wide range of chlorites.

Mixed layer mineral group—Mixed layer minerals are clay minerals in which different kinds of layers (e.g. chlorite, illite, montmorillonite) alternate with each other. Mixed layer minerals give broad peaks between 10-14 Å, depending upon the nature of inter-stratified layers. This group is very variable in composition. In broad terms mixed layer minerals are of two types:

(*a*) *Regular*—layers of different types, for example type I and II, alternate according to a specific law, i.e. I II I II—. Such regular mixed layer minerals may be given specific mineral names.

(*b*) *Irregular*—layers of different types are randomly interstratified. In nature all transitions between regular and irregular mixed layer minerals are found. Theoretically all kinds of layers can be interstratified with each other. Most common interstratifications are those between illite, montmorillonite and chlorite. Depending upon the layers interstratified names are given: illite-montmorillonite mixed layer, montmorillonite-chlorite mixed layer.

Mixed layer minerals do not behave as mixture, but as true minerals whose basal spacings are equal to the sum of those of the two interstratified layers.

METHODS OF STUDY

Separation of clay fraction (<2 μ)—A portion of the core samples were crushed gently avoiding breaking of grains. The crushed samples were then treated with a dilute solution of $H_2 O_2$, heated at 60°C. for several hours to destroy organic matter. $H_2 O_2$ solution was added until the reaction was completed.

Disintegrated samples free from organic matter were then subjected to wet sieving through 63 μ sieve, and sand fraction (>63 μ) was separated from silt+clay fraction (<63 μ). The <63 μ fraction was poured into Atterberg cylinder and thoroughly shaken. Na-pyrophosphate was added as dispersing agent. The suspension was allowed to settle, and after ca. 18 hours, depending upon the temperature, all the particles larger than 2 μ had settled down. The exact settling time can be calculated according to Stoke's law. The supernatant clay suspension was taken off after the calculated time.

This process was repeated several times until the supernatant liquid was almost clear after the required settling time.

The clay suspension was then filtered and clay fraction obtained. Clay fraction was gently pulverized in an agate morter.

Preparation of samples for X-ray diffraction analysis—There are several methods which are used for preparation of samples in the study of clay minerals by X-ray diffraction. The choice of method is very important if quantification of clay minerals is attempted. The problem of sample treatment and slide preparation technique is very important. This problem has been discussed at length by GIBBS (1965, 1968). Clay minerals can be studied in basal or randomly oriented samples. Use of preferentially oriented samples (basal oriented) is widespread because the diagnostic basal reflections are emphasized, overall pattern becomes simple, and sensitivity is increased.

At present there are several mounting methods in use for preparation of preferentially oriented samples (BROWN, 1961; KITTRICK, 1961; GIBBS, 1965). The important methods are smear-on glass slide, suction on ceramic tile, pipette on glass slide, beaker on glass slide, centrifuge on glass slide, centrifuge on ceramic tile, powder press technique. GIBBS (1965) recommends the use of smear-on glass slide, suction on ceramic tile, and powder press technique, becuase they give high precision and effect of mineral seggregation is less. However, in the present study pipette on glass slide technique was used. Clay fraction was taken into suspension by ultrasonic treatment, a thick slurry was prepared. Because of high concentration of suspension flocculation takes place, and effect of seggregation is reduced. This method gives preparations where preferred orientation is good, and effect of seggregation is low (less than in centrifuge methods).

For each sample, three slides were prepared by pipetting the dispersed thick slurry onto glass slides (2.8 ×4.8 cm). Slides were dried at room temperature. In quantitative X-ray studies upper surface of the preparations should be very smooth and representative of the total sample, since the X-rays penetrate only a thin uppermost film of the sample.

X-ray technique—Among the various methods used in the study of clay minerals, the X-ray method is now the most widely used method in determination of clay minerals. The advantages of X-ray method over other methods are that it is non-destructive and requires only small amounts of material. X-ray diffraction technique is more rapid than the X-ray photographic methods. In the present study X-ray diffraction technique has been used.

Clay mineral particles are mostly platy in nature and acquire readily a high degree of preferential orientation. The identification of clay minerals is based on the recognition of basal (001) reflections. In X-ray difffractograms of the preferentially oriented samples 001 reflections are greatly enhanced, while the other reflections are diminished and suppressed. For more specific determinations, sometimes, randomly oriented samples are used, e.g. for identification of polymorphs of mica. X-ray studies were made on a Phillips (Müller Mikro II) diffractometer under the following condition:

—CuK α_1 as source, Ni filter, beam slit 1°, detection slit 0.1°, 40 kV, 20 mA, goniometer speed 1/2°/min., paper speed 600 mm/hr. During slow scanning of certain portions, i.e. chlorite-kaolinite peak, the goniometer speed was reduced to 1/4°/min. The scale factor (sensitivity) was varied from sample to sample according to the intensity of the X-ray reflections, mostly 1×10^3 sensitivity was used.

For each sample three diffractograms were prepared. The first one was made by scanning a specimen from 3-30° 2θ at room temperature. The slide was subsequently glycolated by placing it in glycol vapours for at least one hour at ×60°C., then the sample was exposed to X-rays again, and scanned from 3-15° —2θ. In samples containing both chlorite and kaolinite slow scanning was done between the region 24-26° 2θ in glycolated samples. The third scanning was done of the ignited samples. The slide was placed in a muffle furnace at 550°C. for 1 hour. The X-ray diffractogram was prepared immediately after the ignition.

DETERMINATION OF CLAY MINERALS

Clay fraction is made up of predominantly clay minerals, and minor amounts of other minerals, e.g. quartz, felspar, amphiboles etc. are also present. In this study we have been concerned only about the clay minerals. The identification of minerals by X-ray diffraction analysis is based on the reflection of X-rays by the characteristic atomic lattice planes within the mineral crystals. The clay minerals are layer lattice minerals, and it is very difficult to identify the individual clay-mineral species in a mixed clay sample. Thus, recognition of clay mineral groups is emphasized. As identification is based on lattice spacings, and since some of the mineral groups have several identical lattice spacings, special treatment of the samples is necessary to distinguish some of the groups. The following clay mineral groups have been identified:

Montmorillonite—The material of the clay fraction whose 001 peak expands to 17Å upon glycolation is considered as montmorillonite. On diffractograms of unglycolated samples it shows a peak around 14 Å. On ignition the 14 Å peak disappears and comes to 10Å position.

Mixed layer minerals—Interstratification of several clay minerals is called mixed layer minerals. In the present study they are mostly illite-montmorillonite interstratification; in some cases chlorite is also interstratified. The identification is based on the presence of broad and variable peak or peaks between 10-14 Å. On glycolation mineral peak breaks into two components: one moves towards 10 Å, the other between 14-16 Å. On ignition mixed layer mineral peak disappears and moves to 10 Å position.

Illite—The basal spacing (001) at 10 Å is illite. It remains unaffected on glycolation and ignition. Other minor peaks are at 5Å, and 3.3 Å. 3.3 Å peak interferes with a strong quartz peak.

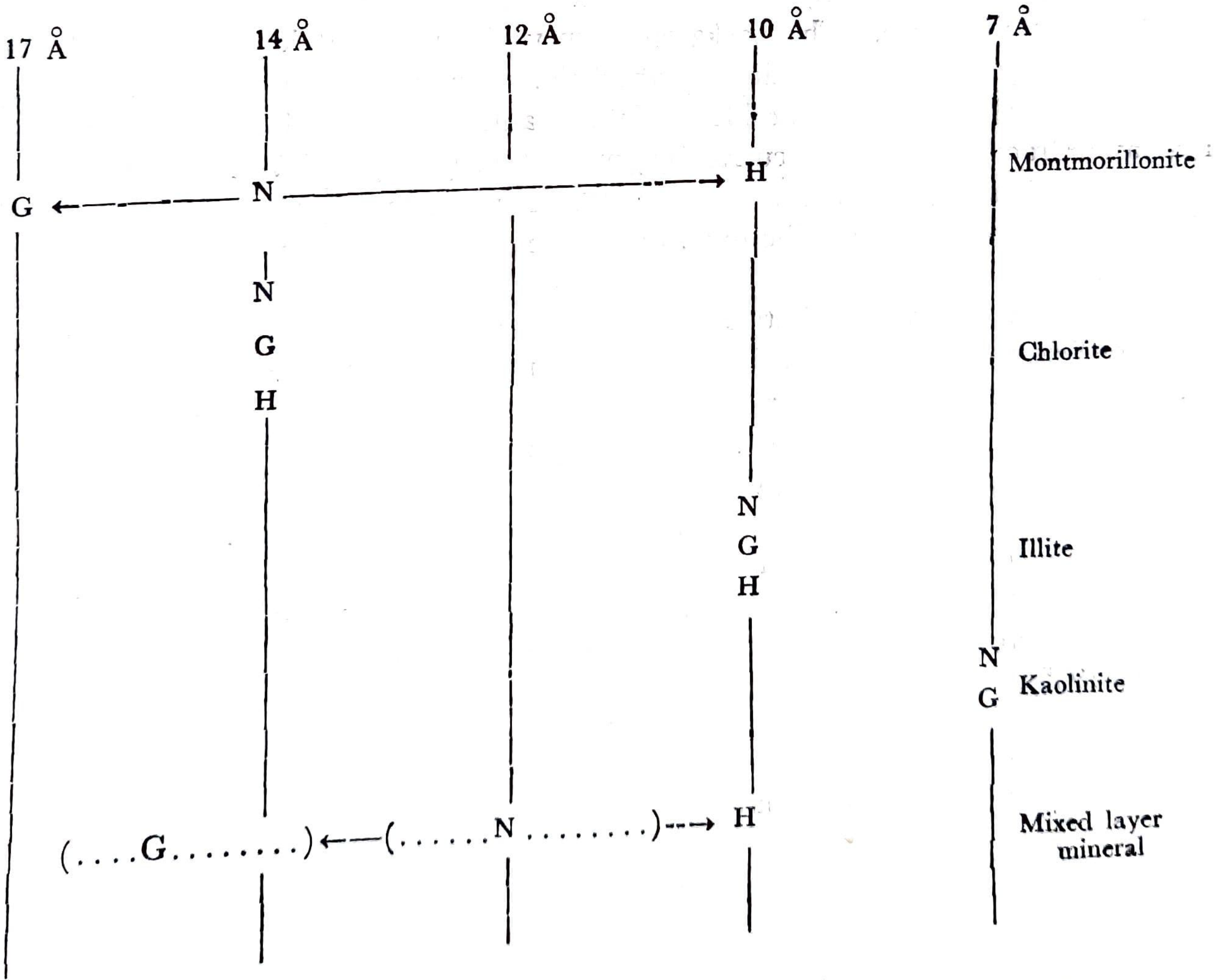
Chlorite—Chlorite is characterized by a series of basal reflections at 14 Å, 7 Å, 4.7 Å, and 3.54 Å. 14 Å peak interferes with the montmorillonite reflection in unglycolated samples. In diffractograms of glycolated samples, 14 Å peak is visible, if content of montmorillonite is low. 7 Å peak interferes with Kaolinite peak. 4.7 Å peak is independent, but intensity is low. 3.54 Å shows good resolution from 3.58 Å peak of Kaolinite. Chlorite reflections remain unaffected upon glycolation. 14 Å peak is stable upon ignition, its intensity usually increases after heat treatment.

Kaolinite—Kaolinite gives strong reflections at 7.2 Å (001) and 3.6 Å (002). These reflections are unaffected upon glycolation. The reflections disappear on heating to 550°C.

The identification of Kaolinite is difficult owing to the fact that (001) and (002) reflections of the kaolinite are overlapped by the (002) and (004) reflections of chlorite respectively. In the presence of chlorite, strong reduction in the peak height of 7.2 Å reflection in ignited samples is taken as an evidence of the presence of kaolinite. Treatment with —HCl dissolves the chlorite. Reduction in the height of 7 Å peak in—HCl-treated samples is an indication of the presence of chlorite, together with kaolinite. These methods were used to check the presence of chlorite together with kaolinite in the samples.

The scheme used in the identification of clay minerals in this study is given in Table-1.

Table 1—Scheme for identification of clay minerals used in this study



N—Natural sample (room temperature) G—Glycolated sample H—Heated sample (550° C)

QUANTITATIVE DETERMINATION

The identification of clay minerals is based on the study of positions of the basal reflections and their possible alterations under various treatments, i.e. glycolation and ignition. There are several methods suggested for the quantitative evaluation of various minerals in a given sample (for details see MITCHELL, 1960; MACKENZIE, 1961; OINUMA and KOBAYASHI, 1961; JOHNS *et al.*, 1964; QUAKERNAAT, 1968). The problems concerning with the quantitative determination in mineral mixtures have been discussed by ALEXANDER AND KLUG (1948); VON ENGELHARDT (1955, 1959). A quantitative estimate of different minerals in a given sample can be obtained from the relative intensities of X-ray reflections. There are many factors which make the quantitative estimation of clay minerals uncertain, e.g. varying degree of crystallinity, varying chemical composition within the individual mineral groups. Thus, intensity of a mineral's characteristic X-ray diffraction peak can not be directly taken as a measure of its abundance in a mixed sample. Moreover, the use of internal standard also has only limited utility in the quantitative determination of clay minerals, because of the obvious reasons that different samples of the same mineral show a range of crystallinity and chemical composition, which control the intensity of reflections.

However, semi-quantitative information about the relative abundance of minerals can be obtained from X-ray diffractograms. Peak height can be taken as a measure of intensity. But the integrated area under a peak is a better measure of intensity in heterogenous samples of clay minerals because it minimizes the effects of chemical composition and crystallinity. The area of the reflections lying above the background on the glycolated diffractograms are measured by means of a planimeter. An important problem is placing of the background curve. The background curve is curved at low-angles, and almost horizontal at higher angles, making a hyperbola. For each sample a best fitting background curve is chosen. Some problems of the quantitative determination of clay minerals on X-ray diffractograms are discussed by JOHNS *et al.* (1954) and WEAVER (1958, 1961).

Quantification of clay minerals is very important while studying a geological succession, as it is of great interest to see the quantitative variation in the clay mineral content of a succession.

In the present study, quantitative calculation of clay minerals have been done after the method of BISCAYE (1965), which is a modified version of the method suggested by JOHNS *et al.* (1954). Weighted peak area percentages were calculated for each clay fraction with the assumption that montmorillonite, mixed layer mineral, illite, kaolinite, and chlorite constitute 100% of the mineralogy. This assumption is reasonable, as non-clay minerals in a clay fraction are present only in traces. For the quantitative calculation, following factors were used: Peak area of montmorillonite (17 Å), peak area of illite (10 Å) ×4, and peak area of kaolinite+chlorite (7Å) ×2. The weighted peak areas were summed up to 100 and percentages for each mineral were calculated—weighted peak area percentages (BISCAYE, 1965).

7 Å peak area is divided between kaolinite and chlorite using a factor obtained from 3.5 Å double peak. The resolution of this peak is mostly good, thus it can be successfully employed in quantitative estimation.

$$\text{The ratio } (\alpha) = \frac{3.58 \text{ Å peak area}}{3.58 \text{ Å} + 3.54 \text{ Å peak areas}}$$

is the kaolinite contribution to the 7 Å peak area. This is a better method to evaluate chlorite than to use 14 Å peak, which interferes with montmorillonite and mixed layer minerals. This problem has been discussed by BISCAYE (1964).

Mixed layer minerals have been considered like montmorillonite. Peak area of mixed layer mineral in diffractograms of glycolated samples has been regarded 1 in relation to ×4 illite and ×2 kaolinite+chlorite.

SYSTEMATIC DESCRIPTION OF THE SAMPLES

In the following survey of the clay mineral composition of the investigated samples is given. The clay mineral composition is discussed zone wise, from bottom towards top of the Gondwana succession. Weighted peak area percentages are given in Table-2.

Table 2—Percentages of clay minerals (weighted peak area) in clay fraction ($<2\mu$) of Gondwana sediments, Korba Coalfield, Madhya Pradesh, India.

Geological unit	Palyno-logical zone	Sample No.	Core Depth in m.	Lithology	Weighted peak area in % Mont-morillo-nite	Mixed layer mineral	Illite	Kaoli-nite	Chlorite
		1	18.28	Coarse-grained Sst.			6	94	..
		4	39.20	Coarse-grained Sst.			6	94	..
		6	44.57	Coarse-grained Sst.			9	91	..
		9	95.30	medium-grained Sst.			3	97	..
		10	97.00	medium-grained Sst.			3	97	..
		12	114.50	sandy shale ..			6	94	..
	III B	14	117.50	coarse-grained Sst...			traces	88	12
		15	129.84	shaly coal. ..			6	94	..
		20	148.84	medium-grained Sst.			6	75	19
		21	150.90	shaly coal ..		73	..	27	..
		28	189.80	shaly coal ..		37		63	..
		35	229.61	medium-grained Sst.				100	..
		40	257.27	medium-grained Sst.			12	81	7
Barakar									
		42	271.32	sandy shale ..		19		81	..
		48	298.50	coarse-grained Sst...			traces	55	45
		52	312.80	sandy shale. ..				100	..
		55	316.00	shaly Sst. ..		62		38	..
	III A	56	316.80	sandy shale ..		56		44	..
		57	318.50	medium-grained Sst.				69	22

Geological unit	Palyno-logical zone	Sample No.	Core Depth in m.	Lithology	Weighted peak area in %				
					Mont-morillol-nite	Mixed layer mineral	Iilite	Kaolinite	Chlorite
		61	337.60	sandy shale ..		56		44	..
		66	360.25	coarse-grained Sst. . .				77	23
		68	360.95	shale ..		43		57	..
		71	365.50	medium-grained Sst.			traces	43	57
		77	413.70	medium-grained Sst.			9	76	15
		83	443.50	coarse-grained Sst. . .			10	45	45
		91	472.30	shaly Sst. ..				100	..
	II B	93	473.00	shaly Sst. ..				42	traces
		95	474.90	medium-grained Sst.				64	27
		100	517.75	medium-grained Sst.				71	29
		103	519.20	shale ..		42		58	..
		107	523.39	gray shale ..	9		15	76	..
	II A	111	525.99	medium-grained Sst.	12		12	76	..
		114	527.10	shale ..	15		25	60	..
Barakar		117	548.50	very coarse-grained Sst	..		..	90	19
Talchir		121	621.15	shale ..	68		13	19	..
	I B	127	649.10	medium-grained Sst.	64		16	10	10
		130	660.50	shale ..	78		6	16	traces
		132	666.15	sandy shale. ..	72		7	19	2
		134	668.90	shaly Sst. ..	32		46	18	4
		136	674.55	shaly Sst. ..	90		traces	10	traces
	I A	142	685.05	pebbly Sst. ..	60		32	6	2
		144	688.00	pebbly Sst. .	65		7	6	2

Diffractograms of each sample are reproduced in Pls. 1-14. For each sample three diffractograms are reproduced: at room temperature, glycolated and ignited up to 550°C.

Zone I A—sample Nos. 144, 142, 136, *and* 134 (Pls. 1 & 2). Clay fraction of the samples of this zone are characterized by the abundance of montmorillonite. The average composition is:

montmorillonite	67%
chlorite	2%
illite	21%
kaolinite	10%

Except for sample 134, montmorillonite is well-crystallized and shows a sharp peak. In glycolated samples 17 Å peak is very prominent and possesses a shoulder on the high angle side, suggesting presence of montmorillonitic mixed layer minerals. Illite (10 Å) is present only in small amounts, its peak is rather broad suggesting low crystallinity. Kaolinite is also present, its peak is broad indicating poor crystallinity. Chlorite is present only in minor amounts, in ignited samples 14 Å peak is mostly destroyed; only in sample 134, 14 Å peak is present in ignited sample. These facts indicate poorly crystalline, unstable chlorite.

Zone I B—Sample Nos. 132, 130, 127, 121 (Pls. 2 & 3). Clay mineral association is identical to that of zone IA, montmorillonite is dominant mineral with minor amounts of illite, kaolinite, and chlorite. The average composition is:

montmorillonite	71%
chlorite	3%
illite	10%
kaolinite	16%

Montmorillonite is well-crystallized as shown by sharp, pointed basal peaks. The basal peak of montmorillonite (17 Å) in glycolated samples is asymmetrical with a gentle slope towards higher angles, an indication of the presence of mixed layer minerals in small amounts. Illite is present, but very poorly crystallized. Kaolinite is moderately crystallized. In other words crystallinity of Kaolinite of this zone is better than that of zone IA. Chlorite is present in minor amount; its crystallinity is poor, and basal peak (14 Å) is destroyed in ignited samples. However, 3.58 Å peak is bifurcated, suggesting presence of both kaolinite and chlorite.

Zone IIA—Sample Nos. 117, 114, 111, 107, 103 (Pls. 3-5). Dominant clay mineral of this zone is kaolinite, montmorillonite is very subordinate. In the topmost sample of this zone mixed layer mineral appears for the first time. The average composition is:

montmorillonite	9%
chlorite	3%
illite	13%
kaolinite	75%

Sample No. 117 is a very coarse-grained sandstone, dominated by kaolinite, and small amounts of chlorite in the clay fraction. Kaolinite is well crystallized, chlorite is poorly crystallized and unstable. It is destroyed during heating up to 550°C.

Sample Nos. 114, 111, and 107 are very similar in clay mineral composition. They contain dominantly well-crystallized kaolinite; montmorillonite and illite are also present. In diffractograms of the untreated samples a broad peak area is present between 10-14 Å region. On glycolation two peaks appear at 10 Å (illite) and 17 Å (montmorillonite) respectively. These peaks are rather broad, suggesting poor state of crystallinity. More over, mixed layer minerals are also present, indicated by poorly developed peaks between 10-17 Å. Ignited clay mineral slides of the samples 114 and 111 show a small, broad peak at 14 Å, indicating presence of chlorite. This suggests that mixed layers are made up of illite-chlorite-montmorillonite. Crystallinity of montmorillonite is very poor.

These samples represent a border line case where it is difficult to decide whether to identity it as separate montmorillonite and separate illite or as mixed layer mineral. Only because in glycolated samples independent peaks appear at 10 Å and 17 Å, it has been suggested that both minerals are mainly present independently. For convenience, content of mixed layer minerals has also been grouped under montmorillonite. Both shales and sandstones contain similar clay minerals.

Sample No. 103 is a shale, and contains well-crystallized kaolinite as main mineral. In glycolated samples a broad peak appears at 12-14 Å. Upon glycolation this peak splits up into a 10 Å peak and another peak at 14 Å. This has been interpreted as mixed layer mineral with montmorillonite as main constituent. In ignited sample a small peak appears at 14 Å suggesting presence of chlorite in mixed layer mineral. Except for sample No. 117, chlorite is present only in mixed layer mineral. If it would have been present in free state 3.56 Å peak of chlorite must have been visible along with kaolinite. However this peak is not visible.

Zone II B—Sample Nos. 100, 95, 93, 91 (Pls. 5 & 6). Clay mineral assemblage of this zone is much different from the lower part of the succession. A marked change is total absence of montmorillonite. In this zone clay mineral assemblage is controlled by the gross lithology.

In sandstones (Sample Nos. 100 and 95) kaolinite is the major mineral, together with illite and chlorite. Average composition of clay fraction in sandstones is:

kaolinite	68%
chlorite	28%
illite	4%

In shale (Sample No. 93) mixed layer mineral, and kaolinite are the main mineral; chlorite is present in traces. Composition of clay fraction in shale is:

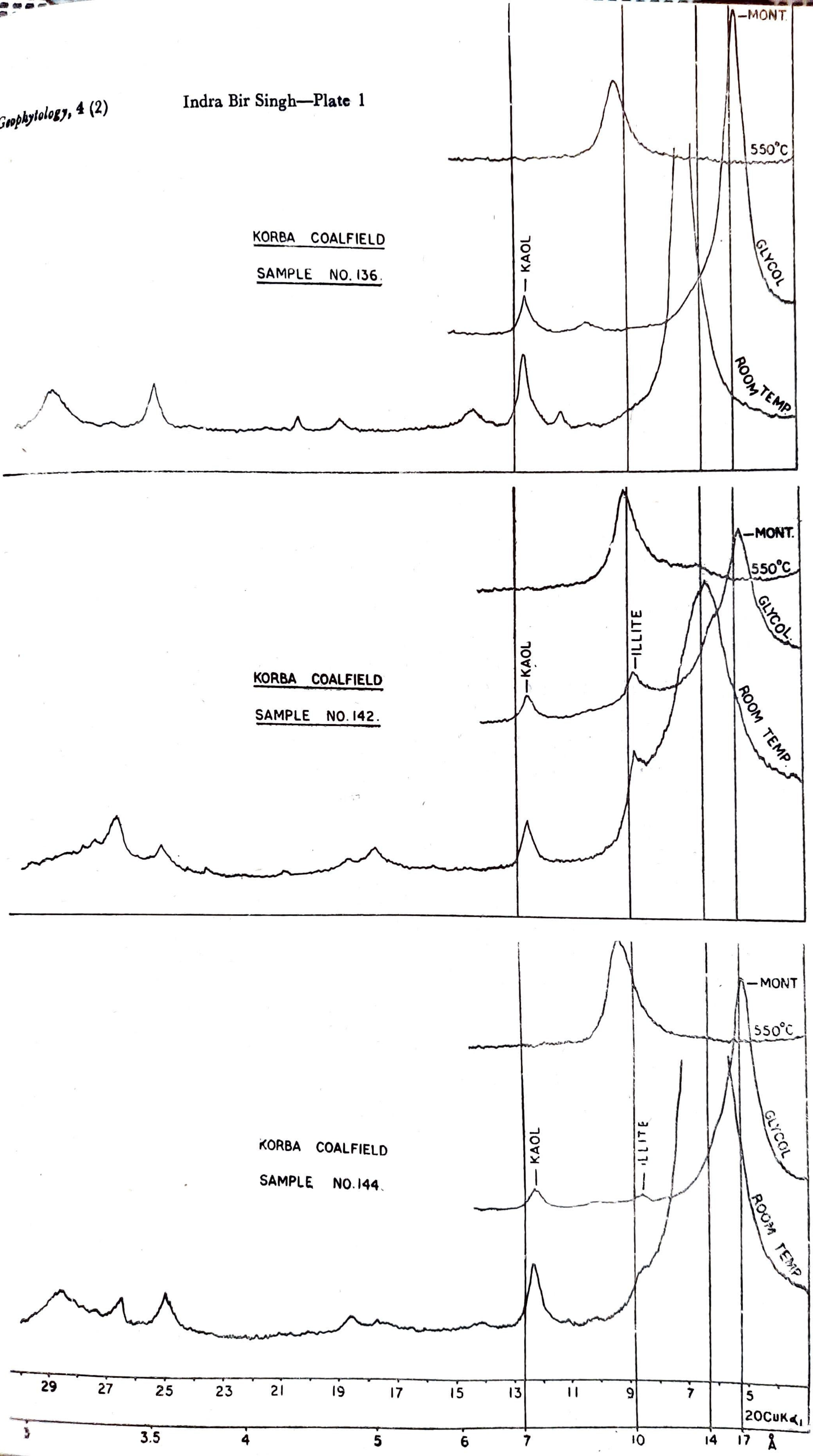
mixed layer mineral	58%
kaolinite	42%

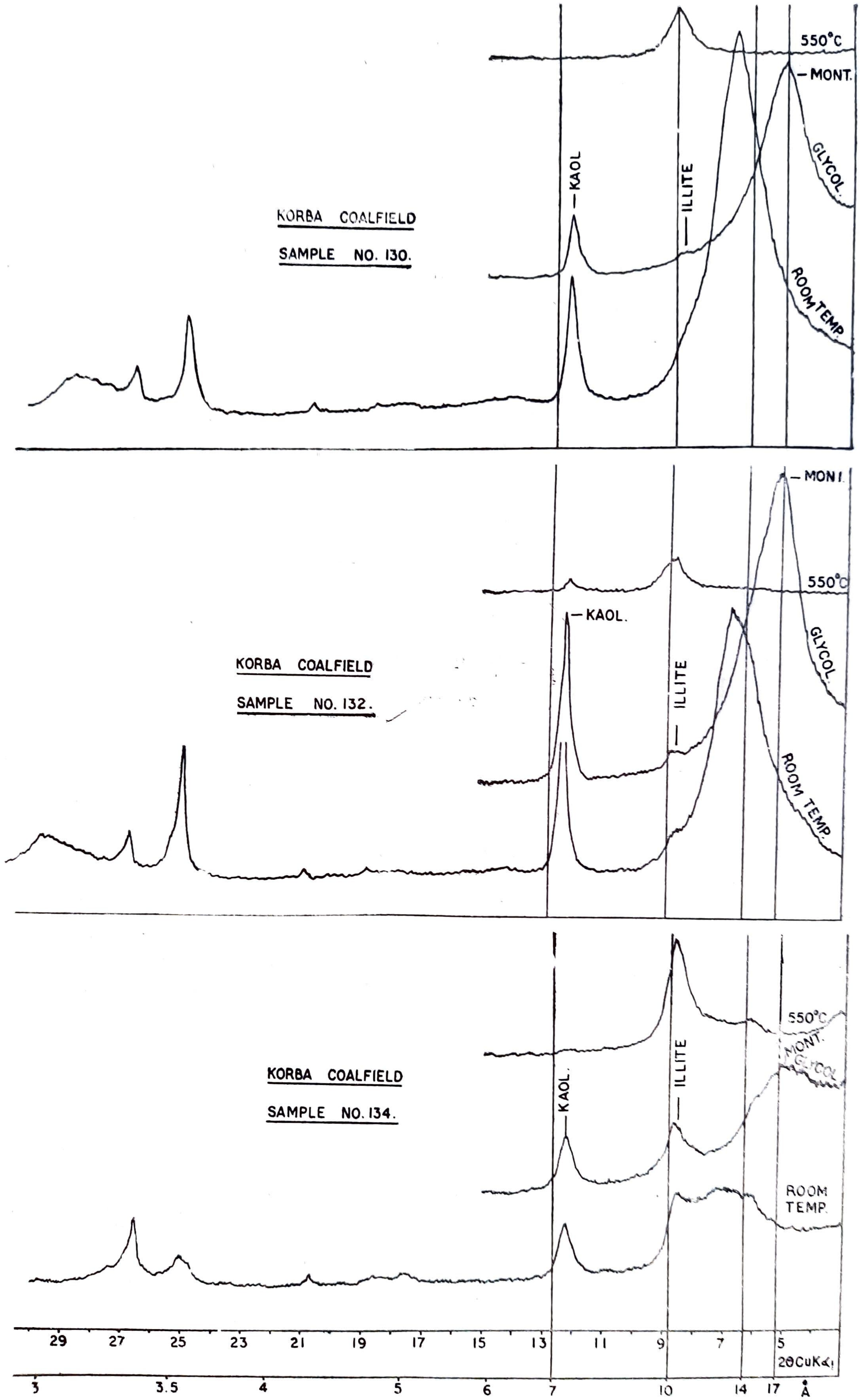
In untreated sample a broad peak is present at 11 Å, which in glycolated sample splits into two parts: One at 10 Å (illite component) and the other at 14 Å (montmorillonite component).

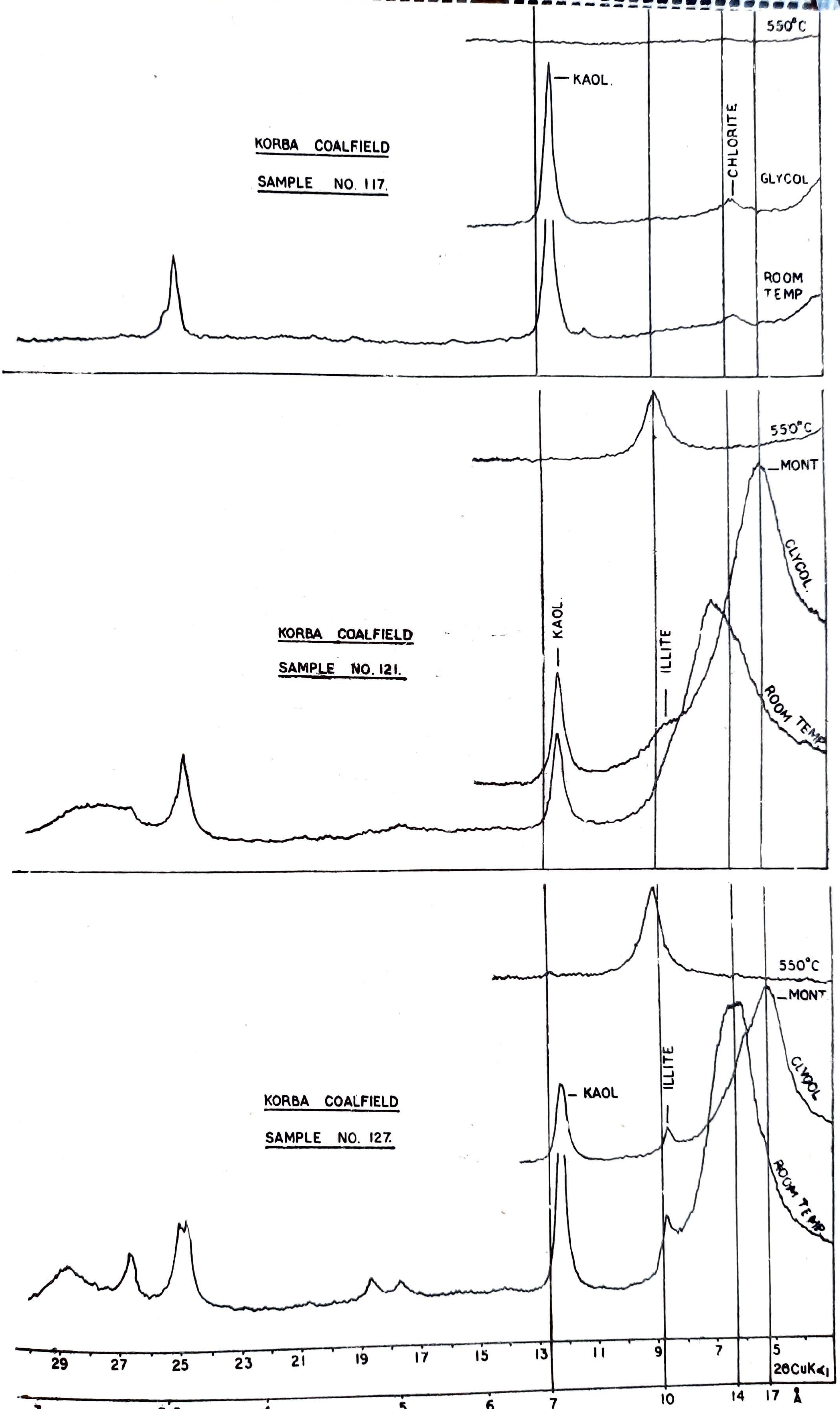
Sample No. 91 is a shaly sandstone from sand-shale intercalation facies. Its clay fraction is monomineralic, made up exclusively of kaolinite.

Zone III A—Sample Nos. 83, 77, 71, 68, 66, 61, 57, 56, 55, 52, 48, 42 (Pls. 6-10). Clay mineral assemblage of this zone is very similar to that of zone IIB. Here also clay mineralogy is controlled by gross lithology, i.e. clay mineral assemblage in sandy and shaly beds is different. In coarse-grained porous sandy sediments (Sample Nos. 83, 77, 71, 66, 57, 48) kaolinite is the most abundant mineral. Chlorite is always present in varying amounts; illite may be present in minor amounts. Average composition in sandstones is:

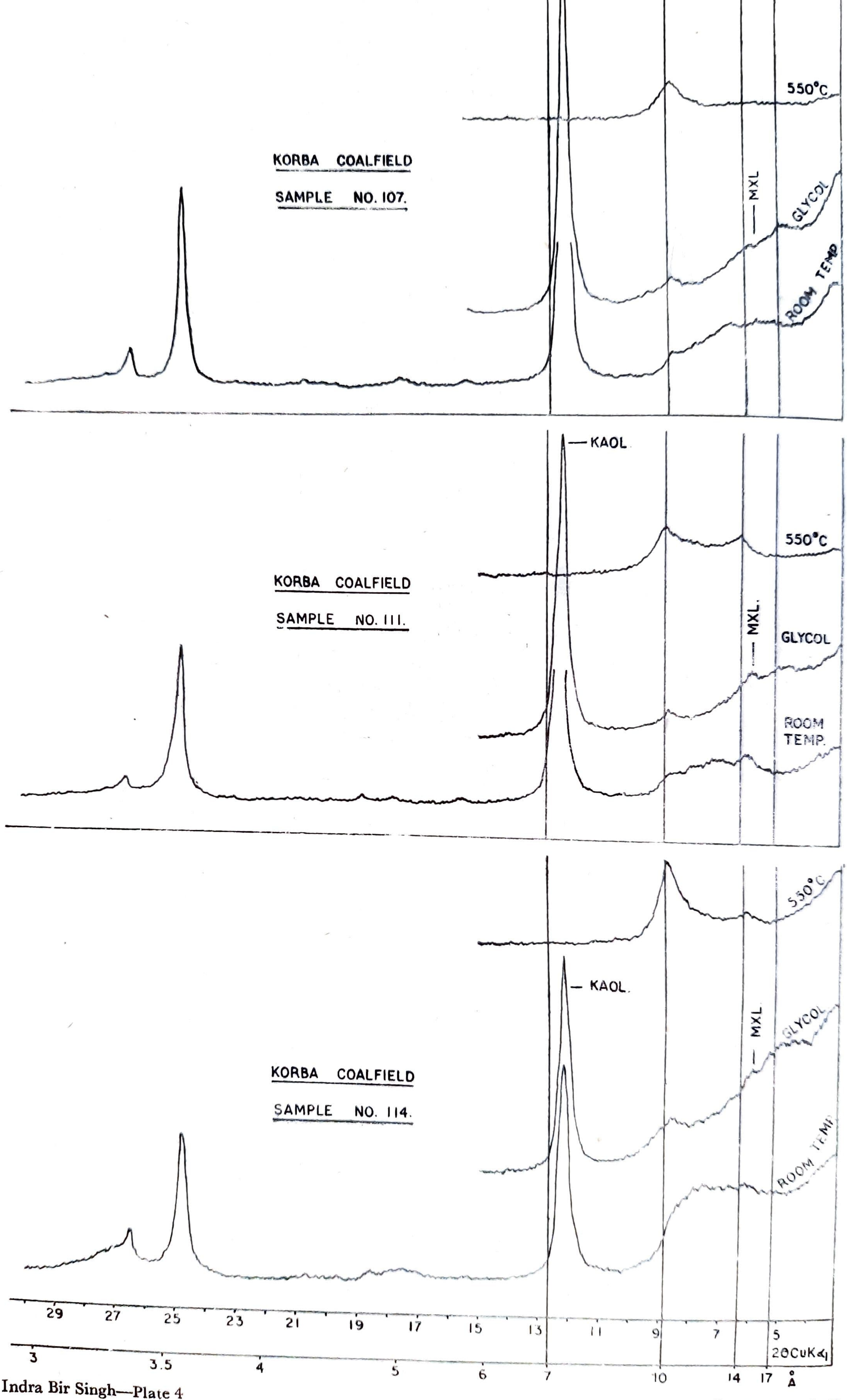
kaolinite	61%
chlorite	34%
illite	5%


KORBA COALFIELD
SAMPLE NO. 136.
—MONT.
550°C
GLYCOL
ROOM TEMP.
KAOL
KORBA COALFIELD
SAMPLE NO. 142.
—MONT.
550°C
GLYCOL.
ROOM TEMP.
KAOL
ILLITE
KORBA COALFIELD
SAMPLE NO. 144.
—MONT
550°C
GLYCOL
ROOM TEMP.
KAOL
ILLITE
29
27
25
23
21
19
17
15
13
11
9
7
5
2θ CuKα1
3
3.5
4
5
6
7
10
14
17
Å

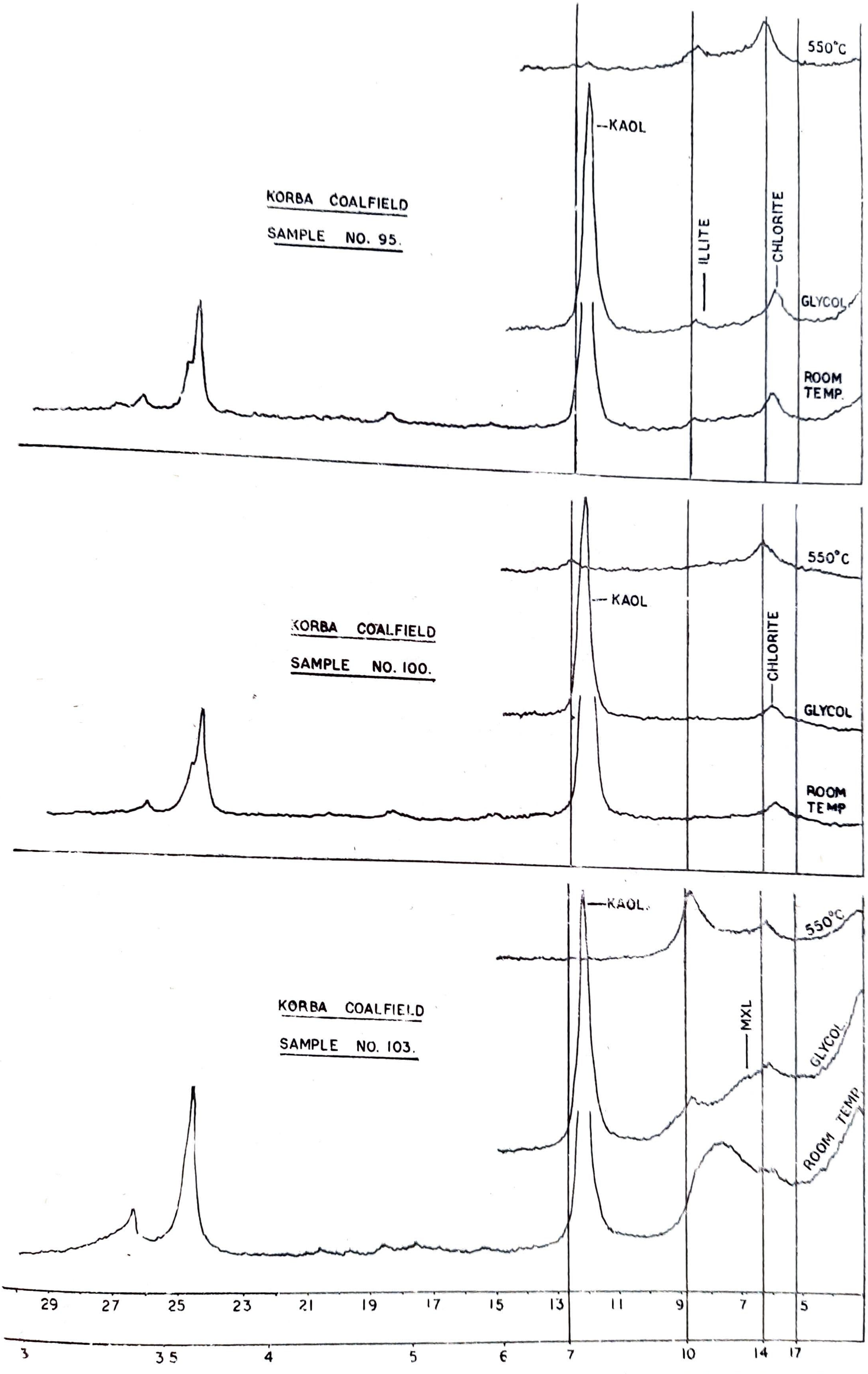

KORBA COALFIELD
SAMPLE NO. 130.
550°C
MONT.
GLYCOL
ROOM TEMP.
KAOL
ILLITE
KORBA COALFIELD
SAMPLE NO. 132.
MONT.
550°C
GLYCOL
ROOM TEMP.
KAOL.
ILLITE
KORBA COALFIELD
SAMPLE NO. 134.
550°C
MONT.
GLYCOL
ROOM TEMP.
KAOL.
ILLITE
29
27
25
23
21
19
17
15
13
11
9
7
5
2θCuKα
3
3.5
4
5
6
7
10
14
17
Å

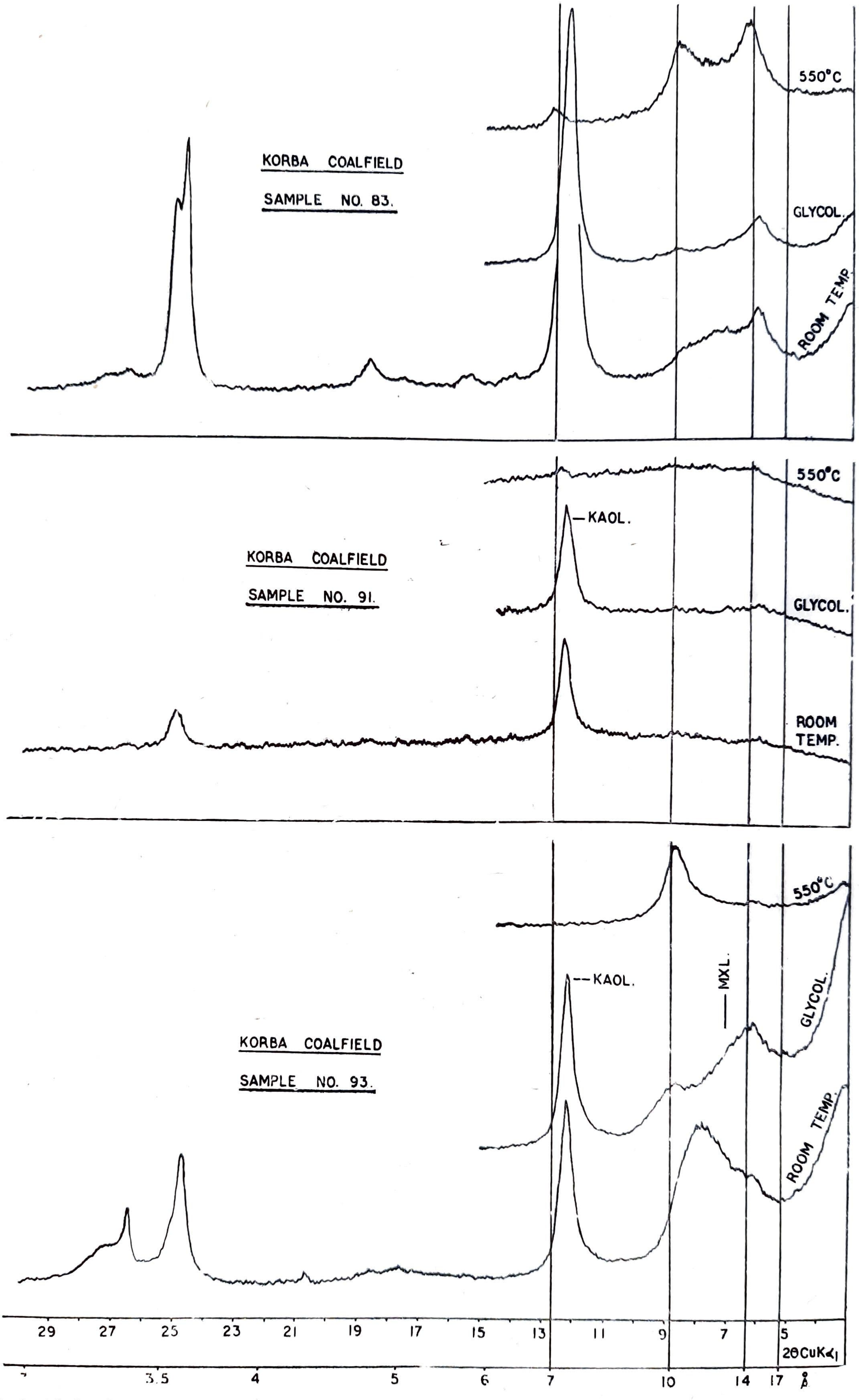


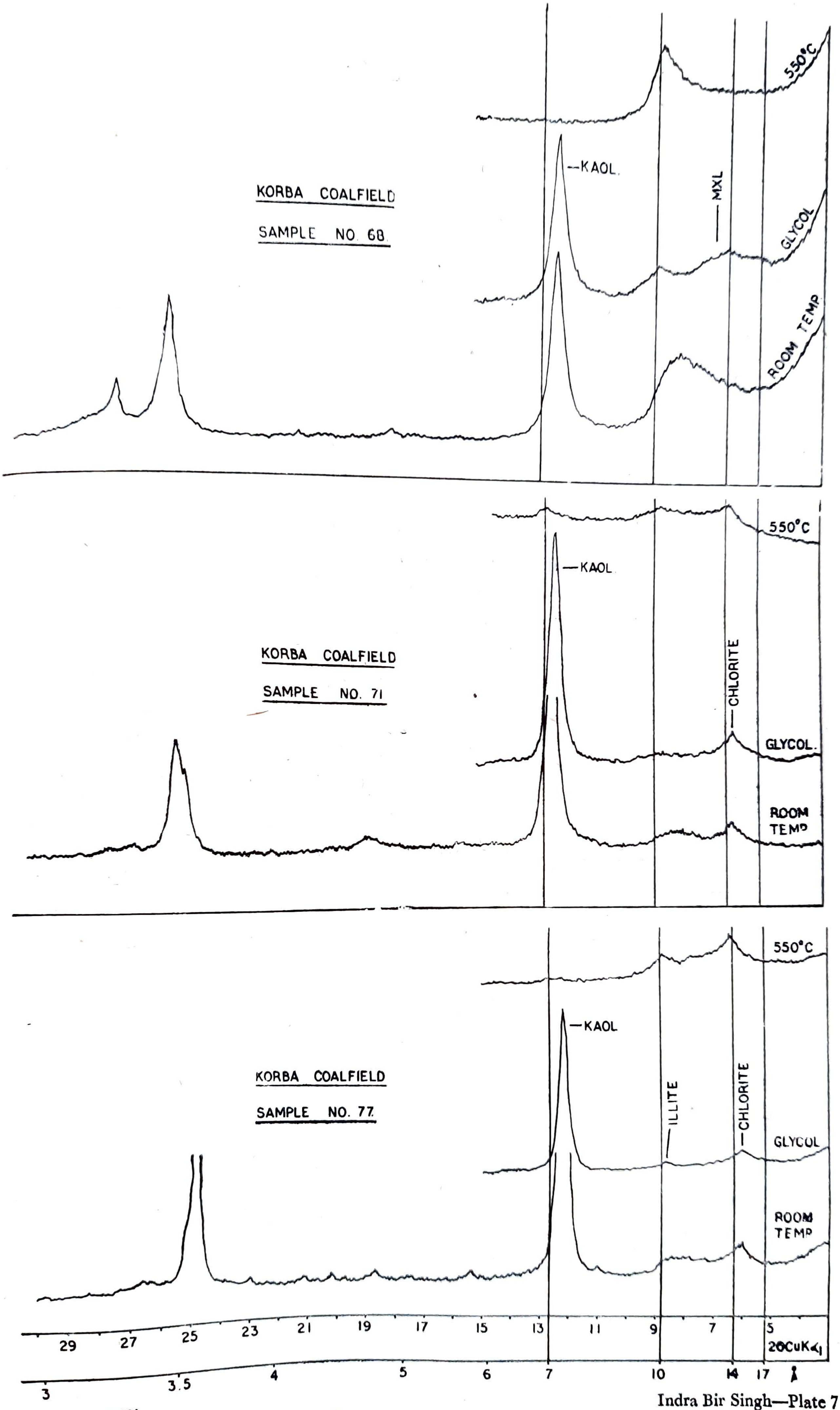
Indra Bir Singh—Plate 3



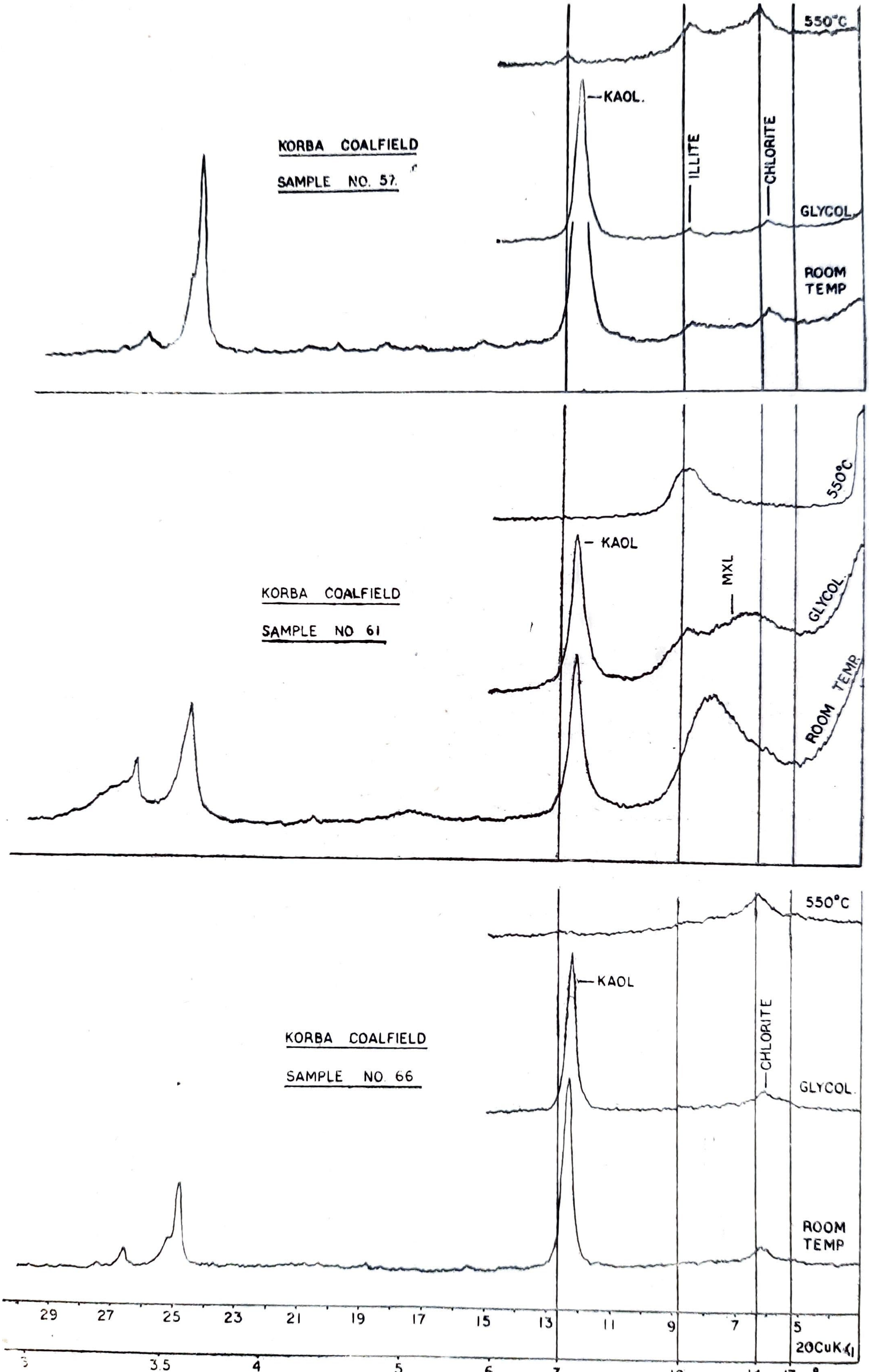
Indra Bir Singh—Plate 4

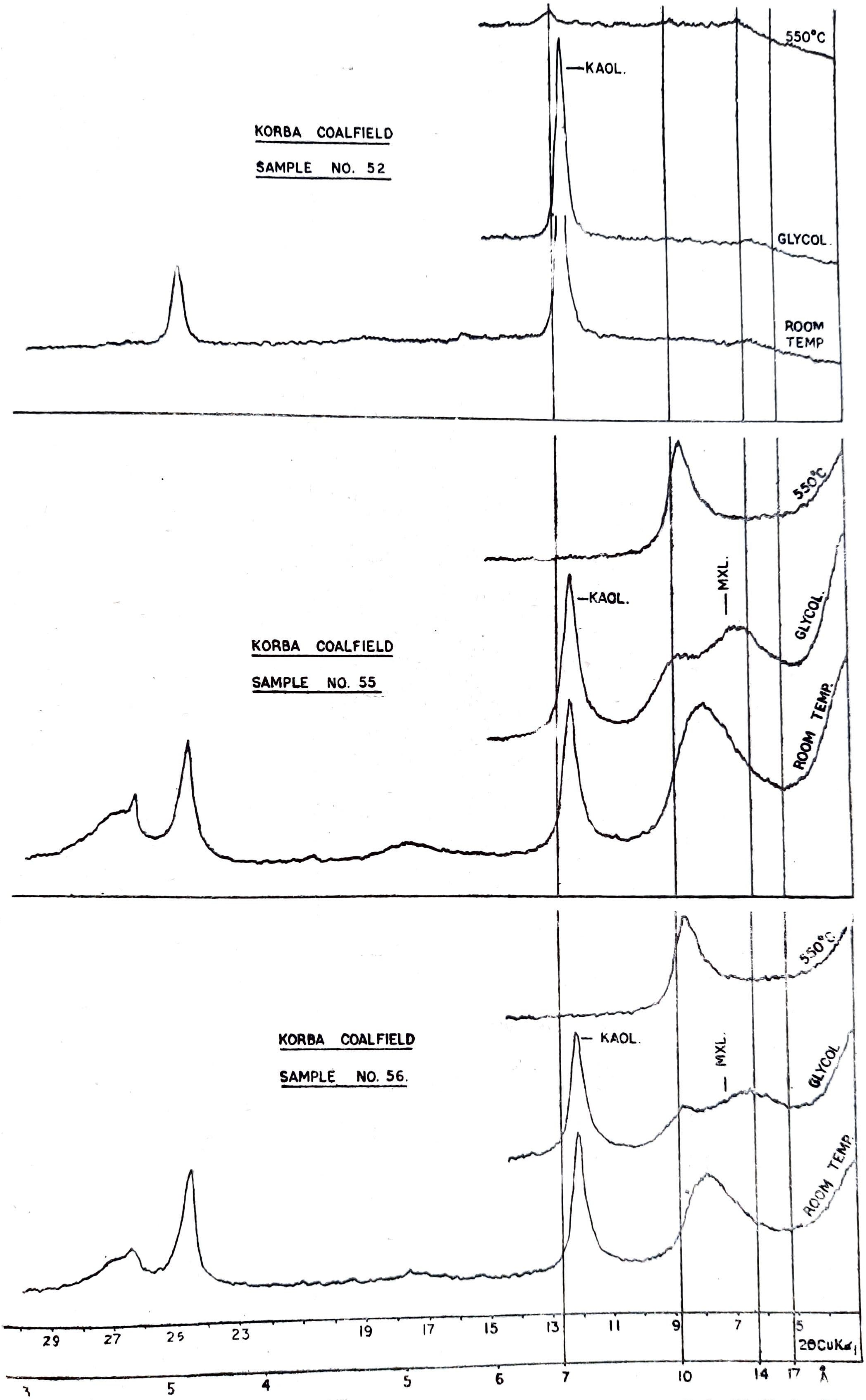

550°C
KAOL
KORBA COALFIELD
SAMPLE NO. 95.
ILLITE
CHLORITE
GLYCOL
ROOM TEMP.
550°C
KAOL
KORBA COALFIELD
SAMPLE NO. 100.
CHLORITE
GLYCOL
ROOM TEMP
KAOL.
550°C
KORBA COALFIELD
SAMPLE NO. 103.
MXL
GLYCOL
ROOM TEMP
29
27
25
23
21
19
17
15
13
11
9
7
5
3
3 5
4
5
6
7
10
14
17


550°C
KORBA COALFIELD
SAMPLE NO. 83.
GLYCOL.
ROOM TEMP.
550°C
—KAOL.
KORBA COALFIELD
SAMPLE NO. 91.
GLYCOL.
ROOM TEMP.
550°C
--KAOL.
MXL.
KORBA COALFIELD
SAMPLE NO. 93.
GLYCOL.
ROOM TEMP.
29
27
25
23
21
19
17
15
13
11
9
7
5
2θ CuKα1
3.5
4
5
6
7
10
14
17
Å

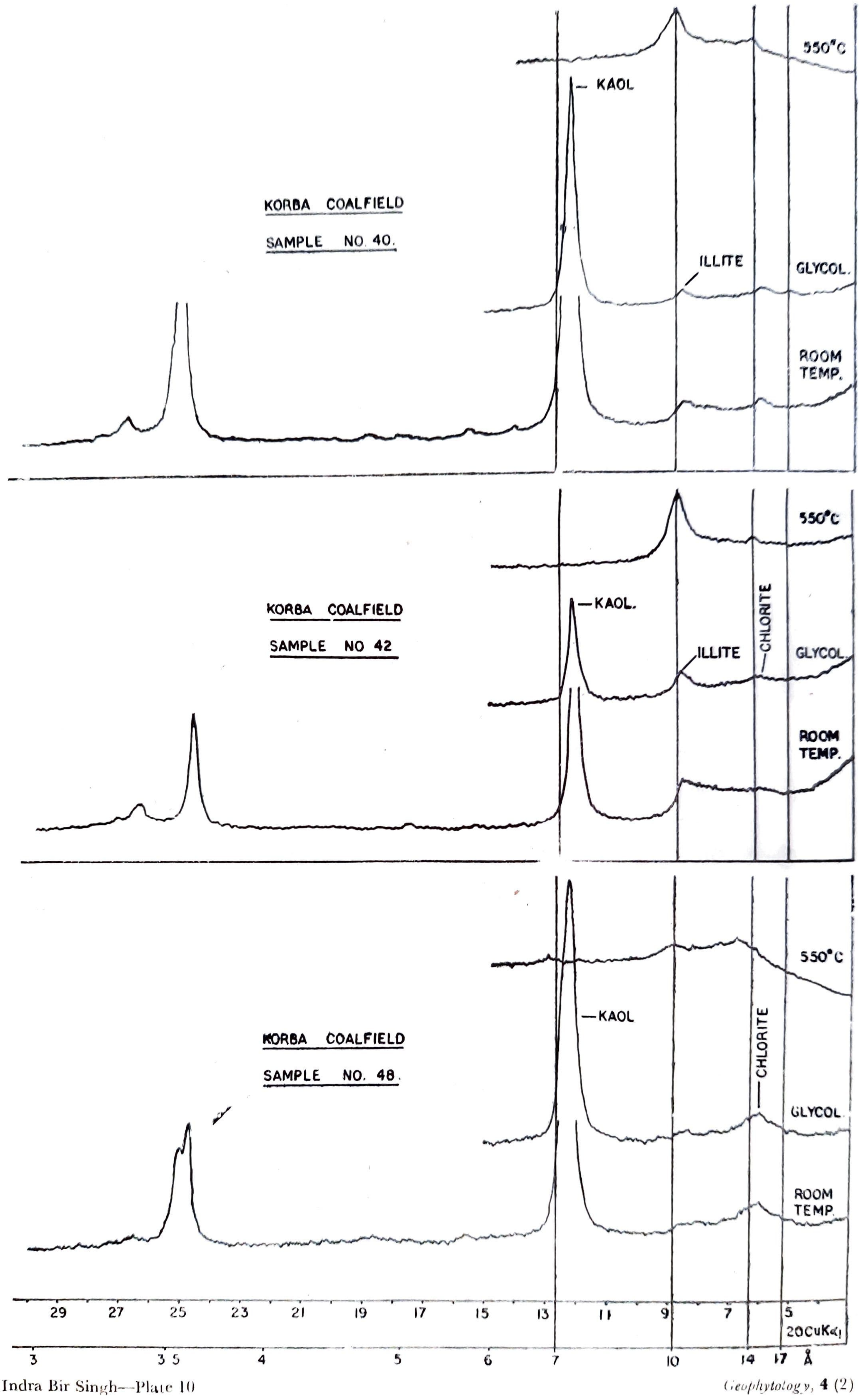


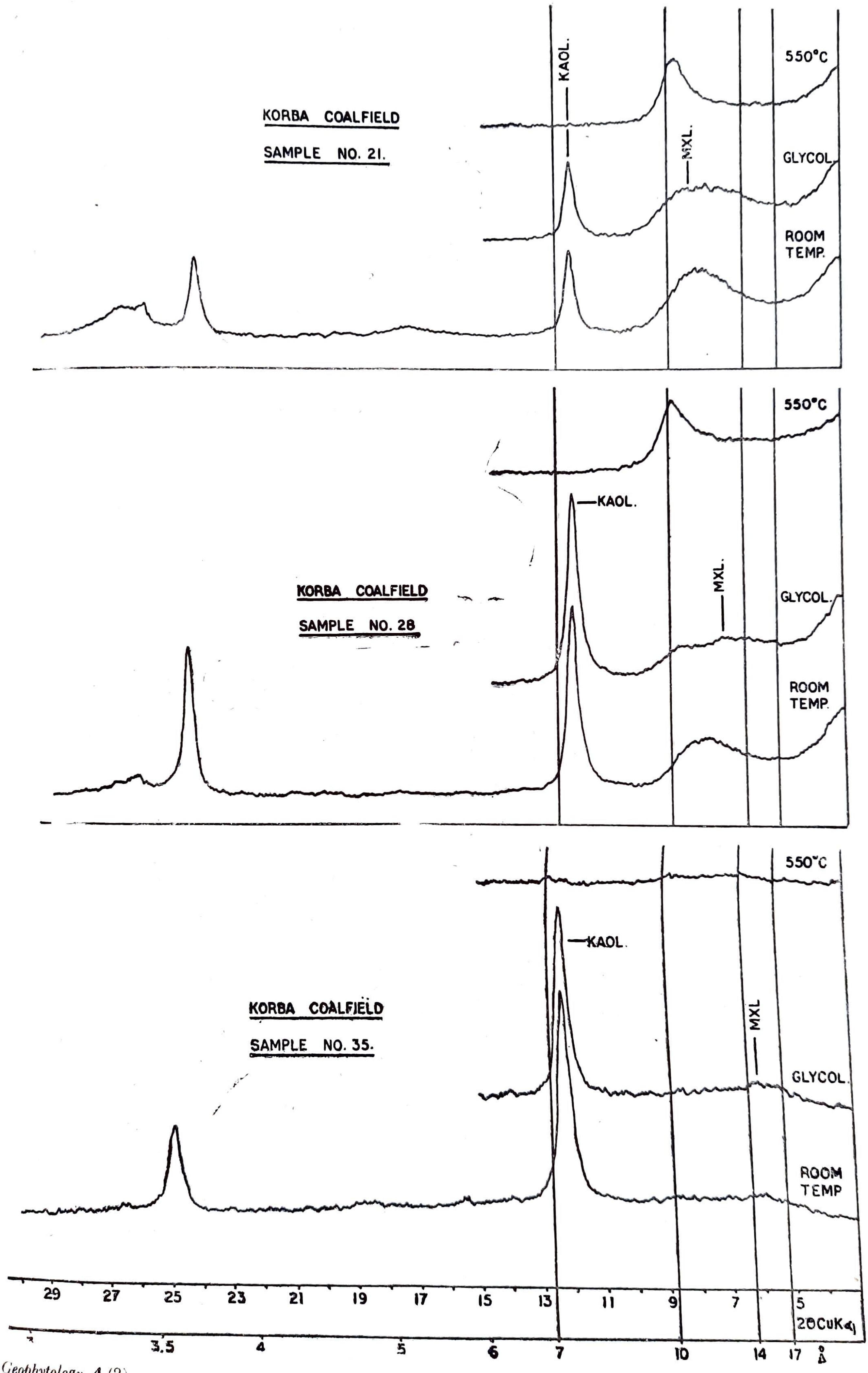
Indra Bir Singh—Plate 7


550°C
KAOL.
KORBA COALFIELD
SAMPLE NO. 57.
ILLITE
CHLORITE
GLYCOL.
ROOM TEMP
550°C
KAOL
MXL
KORBA COALFIELD
SAMPLE NO 61
GLYCOL.
ROOM TEMP.
550°C
KAOL
KORBA COALFIELD
SAMPLE NO 66
CHLORITE
GLYCOL.
ROOM TEMP
29
27
25
23
21
19
17
15
13
11
9
7
5
2θCuKα
3
3.5
4
5
6
7
10
14
17
Å

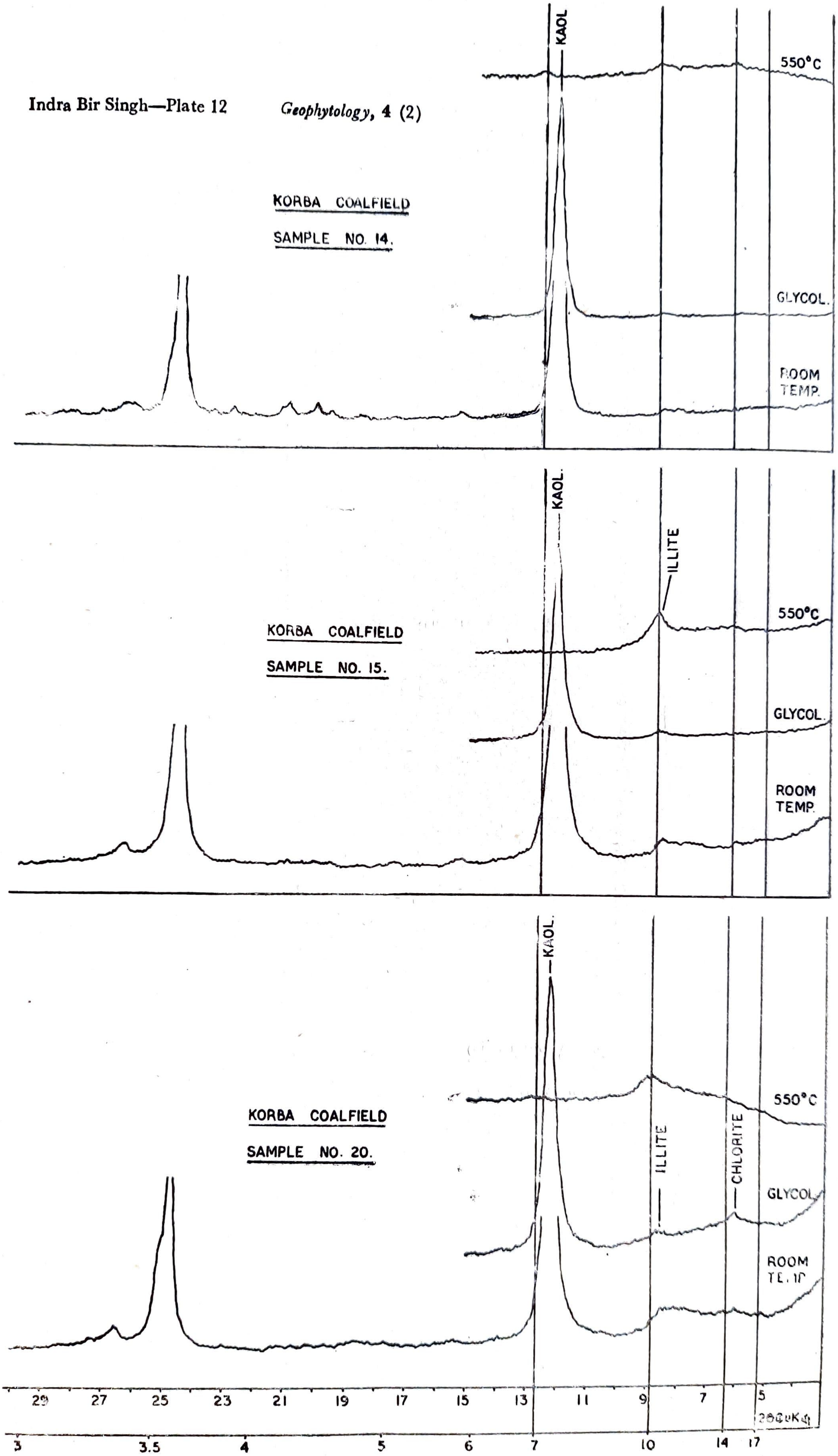


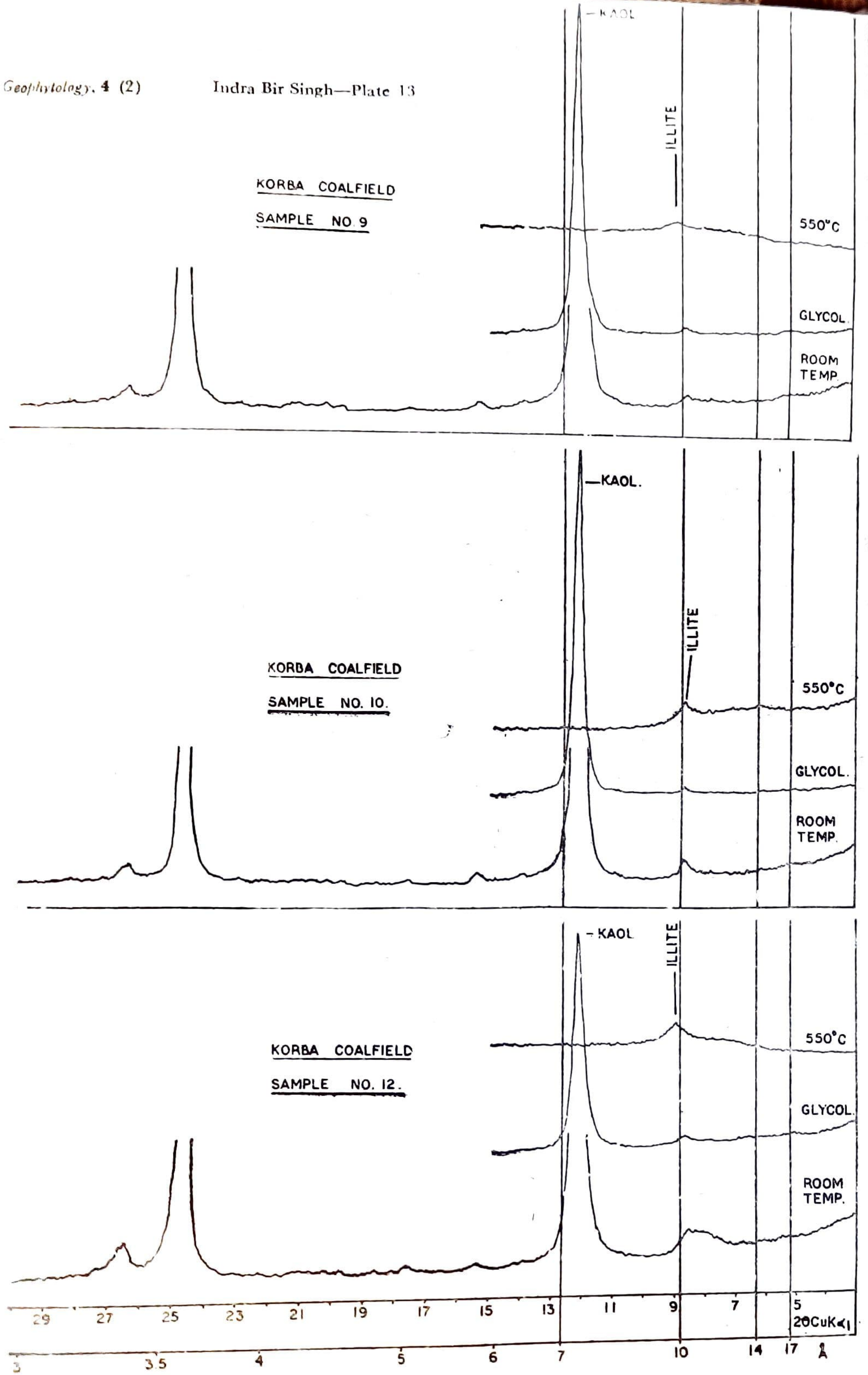
Indra Bir Singh—Plate 9

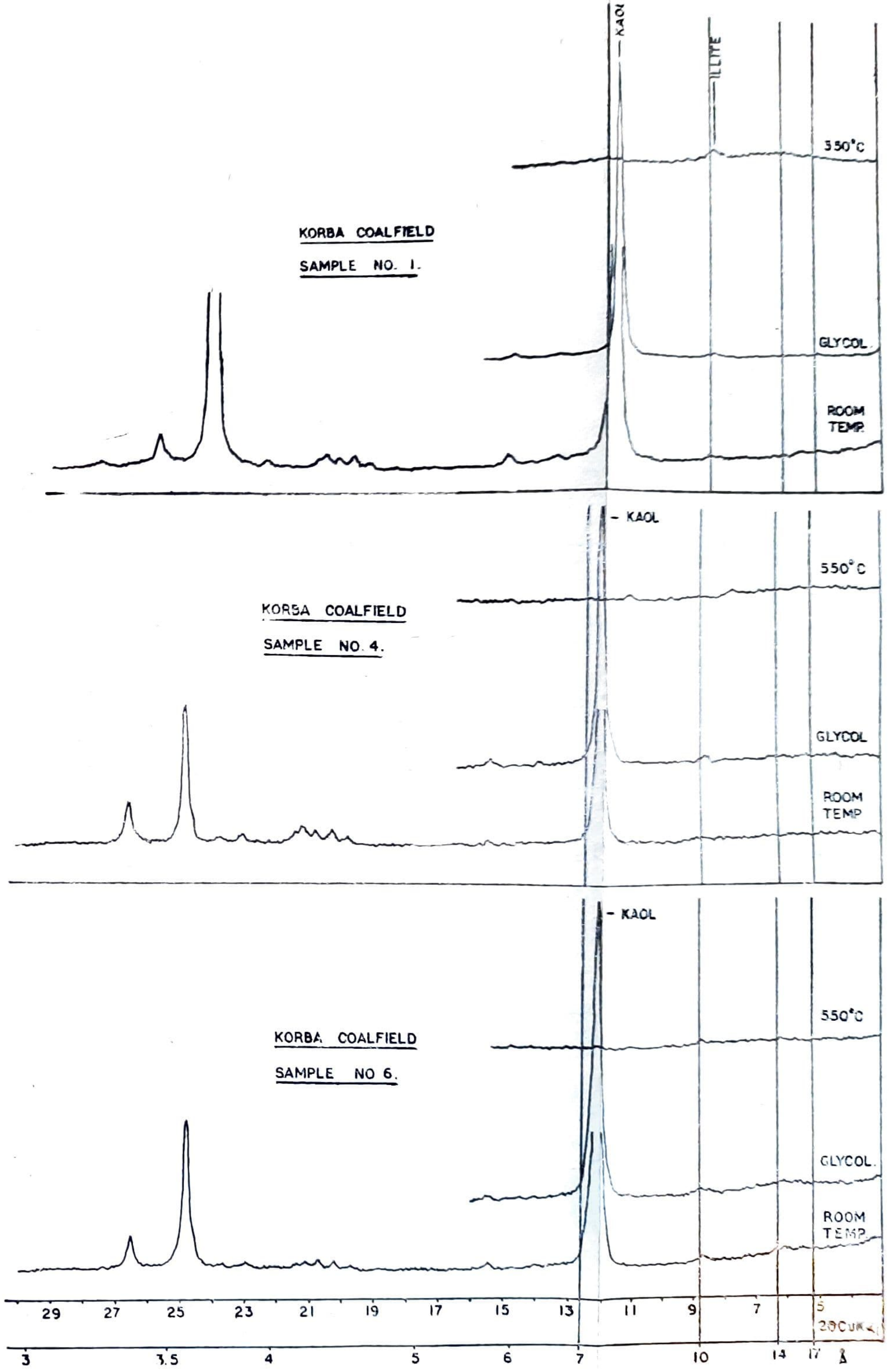

550°C
KAOL
KORBA COALFIELD
SAMPLE NO. 40.
ILLITE
GLYCOL.
ROOM TEMP.
550°C
KAOL.
KORBA COALFIELD
SAMPLE NO 42
ILLITE
CHLORITE
GLYCOL.
ROOM TEMP.
550°C
KAOL
KORBA COALFIELD
SAMPLE NO. 48.
CHLORITE
GLYCOL.
ROOM TEMP.
29 27 25 23 21 19 17 15 13 11 9 7 5
2θCuKα1
3 3.5 4 5 6 7 10 14 17 Å



Indra Bir Singh—Plate 11

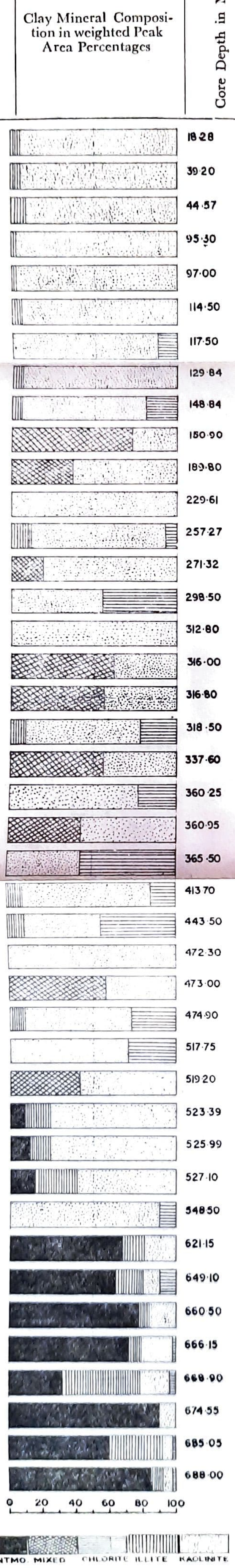

KAOL
550°C
KORBA COALFIELD
SAMPLE NO. 14.
GLYCOL.
ROOM TEMP.
KAOL.
ILLITE
550°C
KORBA COALFIELD
SAMPLE NO. 15.
GLYCOL.
ROOM TEMP.
KAOL.
550°C
KORBA COALFIELD
SAMPLE NO. 20.
ILLITE
CHLORITE
GLYCOL.
ROOM TEMP.
29
27
25
23
21
19
17
15
13
11
9
7
5
2θ CuKα
3
3.5
4
5
6
7
10
14
17


KORBA COALFIELD
SAMPLE NO. 9
KAOL
ILLITE
550°C
GLYCOL.
ROOM TEMP.
KORBA COALFIELD
SAMPLE NO. 10.
KAOL.
ILLITE
550°C
GLYCOL.
ROOM TEMP.
KORBA COALFIELD
SAMPLE NO. 12.
KAOL
ILLITE
550°C
GLYCOL.
ROOM TEMP.
29
27
25
23
21
19
17
15
13
11
9
7
5
2θCuKα1
3
3.5
4
5
6
7
10
14
17
Å


KAOL
ILLITE
550°C
KORBA COALFIELD
SAMPLE NO. 1.
GLYCOL.
ROOM TEMP.
KAOL
550°C
KORBA COALFIELD
SAMPLE NO. 4.
GLYCOL
ROOM TEMP.
KAOL
550°C
KORBA COALFIELD
SAMPLE NO 6.
GLYCOL.
ROOM TEMP.
29
27
25
23
21
19
17
15
13
11
9
7
5
2θCuKα
3
3.5
4
5
6
7
10
14
17
Å

Text fig. 1 Clay Mineral distribution in the clay fraction ($< 2\mu$) of the Lower Gondwana Sediments of the Korba Coalfield, Madhya Pradesh, India.

Stratigraphic Unit	Palynological Zone	Sample No.	Core Depth in Meters	Mineralogical Zone	Remarks
BARAKAR	III B	1	18·28	E	Kaolinite is dominant mineral. Illite and chlorite are very subordinate. No expandable lattice minerals. Subtropical to tropical humid climate, excellent drainage.
		4	39·20		
		6	44·57		
		9	95·30		
		10	97·00		
		12	114·50		
		14	117·50		
		15	129·84		
		20	148·84		
		21	150·90	D	Kaolinite is the main mineral. Clay mineral assemblage is lithology controlled. In sandstones kaolinite is present together with illite and chlorite. In shales kaolinite is present together with mixed layer mineral. In lower part (palynological zone II B) mixed layer mineral is of illite-chlorite—montmorillonite type. In rest of the zone mixed layer mineral is of illite—montmorillonite type; no chlorite. In uppermost part (palynological zone III B-lower part) amount of montmorillonite in mixed layer mineral is low. Warm temperate to substropical climate, modrate to good drainage.
		28	189·80		
		35	229·61		
		40	257·27		
	III A	42	271·32		
		48	298·50		
		52	312·80		
		55	316·00		
		56	316·80		
		57	318·50		
		61	337·60		
		66	360·25		
		68	360·95		
		71	365·50		
		77	413·70		
		83	443·50		
	II B	91	472·30		
		93	473·00		
		95	474·90		
		100	517·75		
	II A	103	519·20	C	Kaolinite is main mineral, minor montmorillonite. In uppermost part independent montmorillonite is replaced by illite-montmorillonite mixed layer mineral. Temperate climate, moderate drainage.
		107	523·39		
		111	525·99		
		114	527·10		
		117	548·50		
Transitional	I B	121	621·15	B	Montmorillonite is the characteristic mineral derived from pre—and/or post-glaciation soils. Minor kaolinite, illite and chlorite. Cold temperate climate, impeded drainage.
		127	649·10		
		130	660·50		
		132	666·15		
Talchir	I A	134	668·90	A	Montmorillonite is the characteristic mineral derived from pre-glacial soils. Minor amounts of kaolinite, illite and chlorite. Glacial climate.
		136	674·55		
		142	685·05		
		144	688·00		


Clay Mineral Composition in weighted Peak Area Percentages

In fine-grained shaly sediments (Sample Nos. 68, 61, 56, 55, 52, 42) mixed layer mineral together with kaolinite is the characteristic association. Mixed layer mineral is illite-montmorillonite, no chlorite is present in mixed layer mineral. Sample No. 52 is monomineralic in composition, only kaolinite is present. Average composition in shales is :

mixed layer mineral	39%
kaolinite	61%

Sample No. 83—Kaolinite and chlorite are the main minerals, illite of poor crystallinity is present in minor amounts. Presence of some mixed layer mineral is suggested by the difference in diffractograms of unglycolated and glycolated samples.

Sample No. 77—Crystallinity of illite is very poor, some mixed layer minerals are present.

Sample No. 71—Illite in traces, some mixed layer minerals present.

Sample No. 68—Mixed layer mineral peak is at 11 Å, which is affected upon glycolation. The slope of mixed-layer mineral peak is rather gentle towards lower angles. A montmorillonite-illite mixed layer.

Sample No. 66—Total absence of illite and mixed layer mineral. Only kaolinite and chlorite are present.

Sample No. 61—Illite-montmorillonite mixed layer; no chlorite in mixed layer.

Sample No. 57—Illite is poorly crystallized, traces of mixed layer mineral are present.

Sample No. 56—An illite—montmorillonite mixed layer mineral with a peak of 11 Å in unglycolated sample.

Sample No. 55—Illite-montmorillonite mixed layer mineral.

Sample No. 52—Monomineralic, only kaolinite is present.

Sample No. 48—Traces of illite.

Sample No. 42—Kaolinite is main mineral. In unglycolated sample 10 Å peak shows a diffused area which in glycolated sample changes to a well-developed 10 Å peak, a diffused area between 10-14 Å, and a small peak at 14 Å. In ignited sample only 10 Å peak is present, no chlorite peak at 14 Å is seen. Thus, the diffused peak area between 10-14 Å is interpreted as poorly crystallized illite-montmorillonite mixed layer mineral.

Zone III B—On the basis of clay mineral assemblage this zone can be divided into two parts: Zone IIIB—Upper part and Zone IIIB—Lower part.

Zone IIIB—Lower part—Sample Nos. 40, 35, 28, 21 (Pls. 10 & 11). Clay mineralogy of this part is very similar to that of Zone III A and II B. In this zone also sandy and shaly beds show different clay mineral associations.

In sandstones (Sample Nos. 40, 35) kaolinite is the most abundant mineral, sample No. 40 contains minor amounts of chlorite and illite. Sample No. 35 is monomineralic and contains only kaolinite, traces of mixed layer minerals seems to be present. The average composition is :

kaolinite	91%
chlorite	3%
illite	6%

In shales (Sample Nos. 28 and 21) clay mineral association is characteristically kaolinite and mixed layer mineral. Kaolinite is well crystallized, mixed layer mineral possesses poor crystallinity. Upon glycolation mixed-layer mineral peak shows only little expansion. It reflects that amount of montmorillonite in mixed layer mineral is low.

Mixed layer mineral is essentially of illite-montmorillonite type, chlorite is not involved in mixed layer. The average composition is:

mixed layer mineral	55%
kaolinite	45%

Zone IIIB—Upper part—Sample Nos. 20, 15, 14, 12, 10, 9, 6, 4, 1 (Pls. 12-14). Clay mineral association of this part is entirely different from the other lower zones. The clay mineralogy is marked by an absolute dominance of kaolinite in all the samples. The average composition is:

kaolinite	92%
chlorite	3%
illite	5%

Bulk lithology has got no control over clay mineral assemblage. In other words, both sandstones and shales show the same clay mineralogy. Kaolinite is abundant, and well crystallized, illite and chlorite are in minor amounts and show poor degree of crystallization. Mixed layer minerals are absent.

Sample Nos. 20 and 15 contain traces of mixed layer mineral. In sample no. 14 chlorite is unstable, it does not exhibit a 14 Å peak (001) in ignited samples.

DISCUSSION

In the last chapter clay mineral assemblage of various zones has been described. It is evident that there are systematic changes in the clay mineralogy of the succession from bottom to top (Text-fig. 1).

We have discussed earlier that clay minerals in a rock can originate in following ways:

1. Inherited clay minerals, derived from the provenance.
2. Transformed and neoformed clay minerals developed in the basin of deposition.
3. Transformed and neoformed clay minerals formed during diagenesis.

Deposition of the succession under study has taken place under glacial, glacio-fluvial, and fluvial environments. These environments do not have any marked chemical character to produce transformation and neoformation of the inherited clay minerals coming into the basin of deposition. Moreover, gondwana sediments represent product of very rapidly deposited detrital sediments. Millot (1970) points out that in rapidly deposited detrital sediments neoformation is negligible. Diagenetic formation of clay minerals in the investigated succession is not of much importance. Important diagenetic changes in the succession are kaolinitization of alumino-silicate minerals, and secondary overgrowth of quartz (Singh, 1974, MS.).

In sedimentary sequences diagenetic changes do take place. Usually, these changes take place if sediments are exposed to concentrated circulating brines, or if sediments are buried to great depths and sufficient time is available. During diagenesis upon burial following sequence of changes is observed:

montmorillonite ⟶ mixed layer mineral ⟶ illite

In thick sedimentary sequences, with increasing depth the content of montmorillonite decreases, and the content of illite increases.

In the Gondwana succession of Korba Coalfield, montmorillonite is abundant in the lower part, but totally absent in the upper part of the succession. The effect of diagenesis upon burial is absent.

Thus, in the present study, except for a part of kaolinite formed during diagenesis, all the clay minerals can be regarded as inherited detrital. Detrital clay minerals brought into the basin of deposition are derived from the weathered rocks and soils in the provenance. Clay minerals of soil and weathering mantle are strongly dependant upon the climate, i.e. supply of water, degree of leaching, pedogenesis, temperature. Thus, in the case of Gondwana succession clay mineral assemblage can be regarded as direct reflection of the clay minerals in the provenance, controlled directly by climate. Reasons for the changes in the clay mineral assemblage with time in the succession of Korba Coalfield are to be sought in the changes in the climatic conditions during Talchir and Barakar periods. Because of the obvious reasons, in the present context chlorite and illite are considered to be derived by degradation of chlorite and mica respectively. Montmorillonite, mixed layer mineral, and kaolinite are neoformed during pedogenesis; thus, these three minerals provide information regarding pedogenesis and climate.

Zone IA—Environment deduced from palynological and geological studies for this zone is characteristically glacial to glacio-fluvial, with a very cold and dry climate (BHARADWAJ & SRIVASTAVA, 1973; SINGH & SHARMA, 1973; SINGH, 1974).

Low temperatures in a glacial environment inhibit chemical weathering, i.e. hydrolysis of silicates. It means cold climate is not suited for the genesis and evolution of clay minerals. On the contrary, clay mineral assemblage of this zone is marked by the abundance of highly evolved clay mineral, i.e. montmorillonite,

The source rocks for this zone are garnet-bearing metamorphic rocks (SINGH & SHARMA, 1973). These rocks under glacial conditions would produce chlorite and illite as main clay minerals by mechanical degradation of chlorite and mica respectively. So, what are the reasons for this discrepancy ? Following explanation has been suggested: Before the onset of the Gondwana glaciation the Indian Peninsula had been exposed to very stable tectonic conditions. During this phase of tectonic quiteness soils could develop extensively. It is believed, that on the metamorphic terrain around the Korba Coalfield, soils developed profusely before the commencement of the glaciation. Pedogenesis favoured the development of montmorillonite—weak lessivage, presence of cations, mildly alkaline milieu. Minor amounts of kaolinite also developed.

At the onset of the glaciation whole landscape became covered with glaciers. In a glacial landscape mechanical erosion dominated, land surface was actively eroded, huge quantities of sediments were eroded and transported by the glaciers. Glaciers not only removed the soil cover, but also eroded the bed rock. The result was deposition of detrital sediments in which rock fragments are embedded in a fine-grained matrix. Chlorite and illite are derived by mechanical degradation of chlorite and mica respectively, may be in pre-glacial or during glacial conditions.

Montmorillonite of this zone shows very good crystallinity, pointing to very favourable conditions of its genesis during pedogenesis in the pre-glacial period. On the contrary, chlorite, illite, and kaolinite show poor crystallinity.

Zone IB—Miofloral studies suggest a cold temperate climate for this zone. In this zone minor coal seams, coaly matter, and organic debris are present, suggesting that climatic conditions were suitable for the development of vegetation. They are deposits of fluvial environment. Clay mineral association of this zone is also marked by the abundance of montmorillonite. Origin of montmorillonite of this zone can be two-fold:

(i) Derived from pre-glacial soils (similar to Zone IA).

(ii) Originated in the cold temperate climate in the post-glacial soils.

It is felt that both processes must have contributed, but it is very difficult to decide which was the main process. In cold temperate climate, montmorillonite can evolve during soil genesis, if sufficient time is given. GJEMS (1967) observed development of montmorillonite during pedogenesis in the Scandinavian soils. Similar results have also been observed by the study of soils developing on moraines in United States and Canada.

It is suggested that during deposition of this zone, under climatic conditions comparable to that of present-day Scandinavia, montmorillonite developed in soils, and ultimately eroded and deposited by fluvial processes.

Zone IIA—Miofloral assemblage of this zone suggests a very cold temperate climate (glacial).

Clay mineral assemblage is marked by an increase in the content of kaolinite, which is well crystallized. Montmorillonite and illite are present in lesser amounts and show poor crystallinity. From bottom towards top of this zone montmorillonite content gradually decreases, ultimately in the topmost sample it is present only as mixed layer mineral.

Development of kaolinite as main mineral would demand rather good drainage. However, locally in restricted conditions with poor drainage cations concentrate, and montmorillonite and mixed layer mineral are developed. Part of the kaolinite has been formed during diagenesis, however, contribution of diagenetically formed kaolinite is rather low in comparison to the contribution of detrital inherited kaolinite.

Development of kaolinite in soils demands rather warm climate with good drainage. Here, there is a discrepancy in the results obtained from palynology and clay mineralogy. Both the studies suggest high humidity but in addition palynology indicates a very cold temperature, whereas clay mineralogy demands moderate to warm temperatures.

Zone II B—Palynological studies suggest a cold and dry climate for this zone.

Clay mineral assemblage in this zone is lithologically controlled: Sandstones contain kaolinite, chlorite, illite; shales contain mixed layer mineral, kaolinite. Kaolinite is ubiquitous and is main mineral in both the assemblages. Kaolinite develops, if drainage is good; mixed layer minerals develop in impeded drainage conditions.

During deposition of this zone, fluvial processes brought material from metamorphic terrain undergoing active weathering. Clay minerals, degraded minerals, amorphous substance, solutions are taken away.

During fluvial sedimentation sands are deposited in river channels. In the low-lying areas—flood basin—vegetation proliferates and swamps develop. In a sedimentary sequence this facies appears as shale and coal beds.

Sandy facies are marked by good porosity and permeability, so that leaching is always good. Under such conditions kaolinite is stable mineral. In shale facies of flood basins drainage is partially impeded, cations are concentrated in waters. These conditions help in the development of three-layer clay minerals, i.e. mixed layer mineral. In other words, in shaly facies where rate of reworking is low, rate of sedimentation is low and drainage is not good, degraded material, illite and chlorite are transformed and neoformed into mixed layer mineral.

An alternative possibility for explaining the lithological control of clay mineralogy is that mixed layer minerals are formed in minor amount along with kaolinite. During sedimentation size fractionation takes place: mixed layer minerals, making smaller grains concentrate in low-energy conditions of flood basins; illite and chlorite, making larger crystal grains accumulate in high-energy sandy facies of river channels. Silt fraction of several sandy samples contain mixed layer minerals in minor amounts, indicating that mixed layer mineral were brought by rivers from the provenance. It is likely that during diagenesis, in sandy facies mixed layer minerals were changed to kaolinite under the influence of acid pore fluids. Nevertheless the first explanation seems to be more appropriate.

Presence of kaolinite as dominant mineral points to a humid, and moderately warm climate. On the contrary, palynological results suggest a cold climate.

Zone III A—Palynological studies indicate a cool and humid (warm temperate) climate. Clay mineral assemblage of this zone is very similar to that of Zone II B. Clay mineralogy is dependent upon gross lithology. Sandstones contain kaolinite, along with chlorite and illite. Shales contain mixed layer mineral and kaolinite.

It is considered that in the provenance conditions were well-suited for the genesis of kaolinite. Kaolinite along with chlorite and illite was transported by rivers and deposited by fluvial processes. It seems that in flood basin deposits (Coal-shale facies) drainage is poor, leading to concentration of cations in the solution. Under such conditions illite and chlorite alter into illite-montmorillomite mixed layer. On the other hand, in sandy facies, high permeability and acidic pore fluids cause development of some more kaolinite during diagenesis at the expense of felspars. Presence of kaolinite as abundant mineral suggest a humid and warm climate for this zone.

Zone III B—lower part—Palynological studies indicate a cool and very humid (warm temperate) climate. Clay mineral assemblage of this zone follows the same pattern as that of zone II B and III A. Sandstones contain kaolinite along with illite and chlorite. Shales contain mixed layer mineral and kaolinite. Crystallinity of kaolinite is very good. However, mixed layer minerals are poorly crystallized, and content of montmorillonite in mixed layer mineral is low. It can be inferred that even in flood basins conditions were not very ideal for the genesis of mixed-layer mineral. It suggests that drainage of the flood basin facies of this zone improved due to increased humidity as compared to those of lower zones.

Zone III B—Upper part—Palynological studies indicate a cool (warm, temperate) climate with increased humidity. Clay mineral assemblage of this zone is marked by the abundance of kaolinite, with minor amounts of illite and chlorite. Both sandstones and shales show the same clay mineralogy. Kaolinite is very well crystallized, crystallinity of illite and chlorite is poor; mixed layer minerals are totally absent. It suggests that even in flood basin environment drainage was good so that mixed layer minerals could not develop. During deposition of this zone all areas of the environment were well-suited for the genesis of kaolinite. Some kaolinite was formed during diagenesis, especially in the sandy facies.

This zone is also marked by the presence of thick and extensive coal seams which point to dense vegetation. Vegetation produces humic acid, and it has been observed that presence of humic acid plays a favourable role in the genesis of kaolinite.

Clay mineralogy of this zone points to a humid and warm climate, almost subtropical to tropical.

Clay minerals of glacial deposits—Glacial environment is marked by low temperatures in which hydrolysis of silicates and neoformation of clay minerals does not take place. Mechanical disintegration is the main process. Illite, and chlorite are supposed to be the most abundant minerals of glacial deposits, formed by mechanical degradation of mica and chlorite respectively (Millot, 1970). In case sedimentary rocks are eroded by glacial activity clay minerals are retained as such without any significant change. Thus, clay minerals of glacial deposits are mechanically inherited, and their composition depends upon the nature of clay minerals in the source rock. Before the commencement of glaciation in any area a thick weathering mantle and soil cover may develop and contribute to the clay minerals of glacial deposits. In such a case, clay minerals of a glacial deposit would give information about the climatic conditions during pre-glacial period.

In the Gondwana sediments of Korba coalfield montmorillonite is the main mineral in the glacial deposits (Talchirs), and it has been derived from the soil cover developed, before the glaciers could become active. In the present-day environments, sediments of North Sea and Baltic Sea possess predominantly montmorillonite and kaolinite as clay mineral. Chlorite and illite are also present in minor amounts (Unpublished data). These sediments have been derived from the glacial material brought from Fennoscandia. Montmorillonite of these sediments must have been derived from pre-glacial or inter glacial soils.

Study of sediment cores from Black Sea by STOFFERS AND MÜLLER (1972) indicates that montmorillonite is abundant is subarctic climate periods. They have explained the dominance of montmorillonite as a result of non-availability of illite from the Northern region. An alternative explanation can be that montmorillonite bearing soils developed during interglacials, which were eroded and deposited during subarctic climate periods.

At the end, it is postulated that before the commencement of an ice-age, a region is exposed to rather varied climatic conditions over long periods which may lead to the evolution of montmorillonite. In such a case, glacial deposits possess montmorillonite as the most characteristic mineral.

Clay minerals of fluvial deposits—Sediments eroded and transported by rivers are accummulated as alluvium. The chemical milieu of the river waters is very inert and do not cause any significant transformation and neoformation of clay minerals, so that clays of the fluvial deposits represent inherited material from parent rock and material from weathering and soil horizons of the drainage basin. In other words, clay minerals of the fluvial deposits are controlled by the provenance. We know that clay genesis on the continents is strongly depended upon the climate. It leads to the fact that clay minerals of fluvia deposits are well-suited for the reconstruction of palaeoclimates.

Fluvial deposits may be differentiated into two main facies: (1) sandy channel deposits and (2) shaly flood basin deposits. Sandy channel deposits possess good permeability and are always moderately to well-drained. Shaly flood basin deposits are weakly to moderately drained, and much vegetation is present. Under certain conditions an entirely different set of clay minerals may develop in flood basin deposits than in channel deposits. During sedimentation of Barakars, rivers flowed from metamorphic provenance and deposited their sediments in a fluvial basin. Bedrocks, weathering mantle, and soil horizons were eroded and the material was brought to the place of sedimentation.

During deposition of lowermost part of Barakar (Zone IB, climate was well-suited for the genesis of montmorillonite. Upwards in the sequence, conditions gradually became less suitable for the development of montmorillonite; but more favourable for the genesis of kaolinite (zone IIA). During deposition of zone IIB, IIIA and IIIB—lower part conditions were favourable for the genesis of kaolinite in the soils and weathering mantle. When brought into the fluvial basin, in flood basin deposits mixed layer minerals evolved under weakly to moderate drainage. Mixed layer minerals did not evolve in channel sand deposits because of good drainage. At the time of deposition of zone IIIB upper part drainage became good even in flood basins. Genesis of mixed layer mineral stopped. Kaolinite was formed extensively all over.

According to MILLOT (1970) Coal-bearing fluvial deposits—coal measures—are mostly characterized by illite as main mineral, along with kaolinite, mixed layer mineral and chlorite.

Lithological control on the clay mineral assemblage has been also observed by Glass (1958) in the Pennsylvanian rocks of U.S.A. He found that sandstones contain more kaolinite than the clays. This difference has been interpreted as a result of development of kaolinite in sandstones during diagenesis. Sandstone with better permeability and more circulating pore fluids were subjected to more kaolinitization than the shales.

Smoot and Narain (1960) report from Palaeozoic oil sands that Kaolinite and mixed layer minerals are more abundant in sandstones than in shales.

INTERPRETATION OF PALAEOCLIMATES BY CLAY MINERALOGY

Based on the clay mineraology and other lithologic characteristics Gondwana succession of the Korba coalfield can be subdivided into following zones:

Zone A—It corresponds to palynological zone I A. Montmorillonite is the characteristic clay mineral derived from pre-glacial soils. Sand fraction contains both alkali—and plagioclase felspars. Glacial climate.

Zone B—It corresponds to palynological zone IB. Montmorillonite, developed during post-glacial period is the main clay mineral. Sand fraction contains only alkali felspar; but silt fraction contains both alkali—and plagioclase felspars. Cold temperate climate, impeded drainage.

Zone C—It corresponds to palynological zone II A. Kaolinite is the main clay mineral, along with small amounts of montmorillonite. Temperate climate, moderate drainage.

Zone D—It corresponds to palynological zones IIB, IIIA, and IIIB- lower part. Kaolinite is the main clay mineral. In sandstones kaolinite is associated with illite and chlorite. In shales kaolinite is associated with mixed layer mineral. Warm temperate to subtropical climate.

Zone E—It corresponds to palynological zone IIIB—upper part. Kaolinite as clay mineral is very prominent. Illite and chlorite are present in minor amounts. No expandable-lattice minerals are present. Subtropical to tropical-humid climate, excellent drainage.

Palynological and clay mineralogical zones have been put together in Table 3. In general terms, palynological results suggest lower temperatures than the clay mineral studies for the same zone. There is usually good agreement as far as the interpretation of humidity is concerned.

Table 3—Showing palaeoclimates inferred from mineralogical and palynological studies

Palynological Zone	Palaeoclimate from palynology (After Bharadwaj & Srivastava, 1973)	Mineralogical Zone	Palaeoclimate from mineralogy (this study)
III B ..	Cool and very humid (Warm temperate with increasedhumidity)	E	Tropical humid, excellent drainage.
III A ..	Cool and humid (Warm temperate)	D	Warm temperate to subtropical, moderate to good drainage.
II B ..	Cold and dry		
II A	Very cold and humid (Very cold temperate)	C	Temperate, moderate drainage
I B ..	Cold and humid (Cold temperate)	B	Cold temperate, impeded drainage
I A ..	Very cold and dry (glacial conditions)	A	Glacial

CONCLUSIONS

Study of clay minerals in clay fraction ($<2\ \mu$) by X-ray diffraction analysis has been undertaken of a lower Gondwana succession in Korba Coalfield to elucidate the climatic history of the sequence. The main results can be summarized as follows:

1. Study of lower Gondwana succession demonstrates that in glacial and fluvial sequences where mechanical inheritance is the main source of the sediments, clay minerals can be successfully used to establish palaeoclimates.

2. Following clay mineral groups have been recorded: montmorillonite, kaolintie, mixed layer mineral, chlorite, illite. The first three minerals are developed during pedogenesis, so that they are potential minerals to interpret palaeoclimates. Chlorite and illite have been formed by mechanical degradation of coarse-grained chlorite and mica respectively. On the basis of clay mineral assemblage five zones have been established.

3. Talchir sediments (Zone IA or Zone A) possess well-crystallized montmorillonite as the main clay mineral, and is considered to be derived from pre-glacial soils of the area.

4. Barakar sediments are products of fluvial deposition. There is a gradual evolution in clay minerals from lower to upper part of Barakars, suggesting that climate became more and more humid and warmer leading to better drainage and increase in kaolinite content.

5. Zone B shows montmorillonite as main mineral. This montmorillonite is believed to be formed during post glaciation period under a cold temperate climate.

6. Zone C is marked by increase in the kaolinite content. Montmorillonite, though present, is only in small amounts ultimately leading to mixed layer mineral. A temperate climate is suggested.

7. Zone D is marked by kaolinite as main mineral and absence of montmorillonite. In this zone clay mineral assemblage is controlled by lithology, which reflect the sub-environments of a fluvial basin. Sandstones, representing channel sands are marked by dominance of kaolinite, along with chlorite and illite as minor constituents. Shales, representing flood basin deposits are marked by mixed layer mineral and kaolinite assemblage. Mixed layer minerals of the lower part of this zone (palynological zone IIB) may contain chlorite, i.e. illite-chlorite-montmorillonite mixed layer mineral. In the higher parts mixed layer minerals are devoid of chlorite, i.e. illite-montmorillonite mixed layer mineral.

It is believed that in the drainage basin mainly kaolinite was formed, together with inherited chlorite and illite. In sandy facies this clay mineral assemblage has been retained as such. In shaly facies of flood basins with abundant vegetation and impeded drainage mixed layer mineral developed, most probably from chlorite, illite, and degraded material, or even neoformed from solution. The climate during this period was warm temperate to subtropical.

8. Zone E is marked by absolute dominance of kaolinite and absence of minerals of expandable lattices. There is no difference in the clay mineral assemblage of shales and sandstones, indictaing that drainage was good even in flood basins. Almost tropical, humid climate, existed during this period.

9. Some of the kaolinite has been produced during diagenesis by hydrolysis of felspars. Silica so released has been used in the generation of quartz overgrowth. It has been confirmed by petrography. Kaolinitization seems to be more prominent in sandstones than in the shales.

10. There is reasonable agreement in the palynological and clay mineralogical results of palaeoclimates. Interpretation in terms of humidity shows good correspondence;

however, there are minor disagreements in the interpretation of palaeotemperatures. The palynological studies give lower temperatures. The palynological zone IIA interpreted to be very cold is indicated here to be cool and zones IIIA & B interpreted palynologically to be cool (i.e. warmer as compared to zones I and II) are indicated here to be warm.

11. The most abundant clay mineral in the arkosic rocks of Gondwana sequence studied here is kaolinite. This finding is contrary to the opinion of WEAVER (1967) that kaolinite as clay mineral is rare in the arkoses.

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