PRELIMINARY STUDY OF THE KAROO PALYNOLOGY IN THE MID-ZAMBEZI BASIN, RHODESIA

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Abstract

For the first time palynological work has been undertaken in order to assess the potential of this branch of science in the Karroo stratigraphic sequences of the Mid-Zambezi Valley, Rhodesia.

The Matabola Flats borehole core was chosen due to its continuous lithological sequence, thick glacial and inter-glacial beds at the base and due to the lack of any metamorphic or tectonic influences within this vicinity. One hundred and five samples were taken, of which 48 were analysed. Fifty-five Genera and one hundred species have been described, four species of which are considered to be new. A number of forms have been retained under alphabetical species categories due to lack of adeqrate comparison in Africa and a reluctance to create new names in a field already over-populated with often ill-defined forms.

The sediments are considered on lithological, palaeobotanical and palaeozoological evidence to range from Permo-Carboniferous (Dwyka) to Upper Permian (Mid Madumabisa Shales). Palynological evidence supports this. Four distinct microfloral assemblages are illustrated with three transitional zones. These compare very closely both quantitatively and qualitatively with the South African Miospore Florizones (Hart 1967), and miospore assemblages in Tanzania, Congo, Zambia and Madagascar. Abroad, these microfloral assemblages compare relatively closely with those in India, Australia and Antarctica, adding one more link to the universal Gondwana flora of Permian times.

The similar and continuous microfloral content of the three glacial and inter-glacial phases in the Dwyka series indicates that they were of relatively short duration, more probably Upper Carboniferous than Lower Carboniferous in age and that no time gap existed during their deposition. The relatively diverse and well-preserved miospore forms in all three inter-glacial phases, (together with an excellently preserved insect wing found 25' above the lowest tillite) indicate a varied macroflora and fauna in the vicinity.

The four miospore assemblages are discussed with regard to their preservation, abundance and possible major plant affinities, and the implications these factors have on the contemporaneous ecological and climatic conditions.

1. INTRODUCTION

The Karroo, The Permo-Carboniferous sequences in the Mid-Zambezi Basin and Limpopo Basin of Rhodesia, have been studied geologically in some detail, more particularly in the former case due to mining exploration in search of coal in the Ecca Series of the Lower Karroo sequences. The Lower Karroo stratigraphic sequences are consequently well known, particularly in the area around Wankie. However, due to the widespread and horizontal continental nature of these deposits, the lithology is very similar laterally and vertically in areas sampled some distance apart within the basin.

Where no fossils of any description are evident, the stratigraphic tools used in local correlation are those of lithology and facies changes. However, the possibility of employing palynology was considered worthy of investigation. If proved feasible, it was hoped that this approach would lead to a means of correlation independent of depositional and lithological factors, and that each stratigraphical unit would be better defined. In turn, it was hoped to assimilate a better understanding of the sequences and facies changes throughout the borehole core, which could be related to the stratigraphic problems in the basin as a whole at a later date.

The material initially selected for palynological study was a borehole core drilled by the Rhodesian Government in 1963 in the course of water exploration. This is sited in the centre of the Matabola Flats in the Central Sebungwe District of the Mid-Zambezi Valley (co-ordinates 18° 13' S; 27° 45' E; 1960 edition of the 1/1 000 000 geological map of Rhodesia). See Charts 1 and 2.

The selection of this borehole core hinges on three main factors:-

1. The lithological successions throughout the core are apparently conformable, thereby presenting a continuous sequence of Lower Karroo sediments.
2. The core passes through a surprisingly thick succession (334') of Dwyka sediments, the thickest found yet in Rhodesia. Bond and Stocklmayer (1967) are of the opinion that this borehole core must have been sited over an ancient Pre-Karroo Valley in which these sediments were accumulated. From lithological data examined in this series, three cycles of glacial and inter-glacial sedimentation have been proposed. The fluctuation of the position of an ice margin in relation to the site of the borehole core would seem more likely to be the cause of these cycles than any tectonic control of sedimentation. Furthermore, the source of sediments and glaciation probably lay to the east of the basin (present-day Lomagundi District) as indicated by the evidence of rock fragments and mineral grains. Flow would therefore have been westwards into the fresh water lakes and valleys of Dwyka times.

3. Another point of considerable importance is the <u>absence of intrusions in the vicinity of this borehole</u>. It is a well known and lamentable fact that a high proportion of the material sampled elsewhere in the Karroo deposits of Southern and Central Africa is in close proximity to dolerite sills and dykes. The resulting metamorphism either completely destroys miospore structures, or renders them totally unsuitable for study.

Balme (personal communication) states that certain South African Permian sediments sent to him were recalcitrant and yielded no plant microfossils. Likewise a borehole core from Kenya penetrating early Triassic and Permian strata, was very disappointing. In Western Australia samples showing evidence of secondary silification seldom yielded worthwhile material. Balme is of the opinion that the silification process itself may not be instrumental in destroying the organic material, but it may be a reflection of tectonic activity. Although dykes and sills ~~(and lava flows)~~ are widespread in Southern Africa from the base of the Karroo System upwards there are no known sills or dykes in the Mid-Zambezi Karroo Basin, apart from the Kariyangwe dyke swarm in the North-Western sector. This is put forward as an explanation to account for the excellent preservation of plant remains in the productive horizons within the borehole core.

Apart from defining each stratigraphical unit in terms of miospore content, with the resultant assemblage patterns and climatological and ecological implications, another problem exists to which it is hoped, palynology will supply the answer. This is the question of age of the glacial and inter-glacial Dwyka sediments in Rhodesia in relation to international time equivalents and the polar wanderings of the late Palaeozoic. It was only comparatively recently that Dwyka sediments were found in Rhodesia and Zambia. (Bond 1952, 1967, Bond and Stocklmayer 1967; Muirhead 1968; Haughton 1969).

From the accumulated evidence, as summarised by Chappell and Humphreys (1970), the existence of localised high to moderate Pre-Karroo relief seems evident, with radiating intermontane valley glaciation. Analysis of the facies fluctuations indicate glaciation marginal to high areas or a small ice cap rather than to a major continental ice sheet.

Palynology in this respect would serve to indicate whether vegetation existed during this glacial period. If little or no vegetation is evident, this would imply that temperatures and conditions were too cold, thereby indicating the presence in the vicinity of a continental ice sheet, with close proximity to polar regions. Conditions similar to this may be seen today in Antarctica. If, however, vegetation was proved to exist by the presence, in rock samples, of miospores and other hardy resistant plant tissue (xylem elements and cuticles), this would support the idea of more localised intermontane glaciers and small ice cap theory, with vegetation growing in the close proximity of glacial and especially inter-glacial sedimentation and deposition.

Present-day conditions in the Alps or Himalayas may well be comparable. Preliminary work conducted on the three glacial phases of the Matabola Flats Dwyka sediments suggests without doubt that these sediments were deposited in the vicinity of a well-established and diverse local flora, thereby substantiating the latter theory. However, it must be borne in mind that earlier glaciation and floral assemblages may not have been found in these sediments due to a number of reasons; earlier sedimentary evidence may have been gouged out during subsequent glacial advances; or this valley may have been blocked to earlier glaciation; also the fact must be considered that this valley may not have existed in earlier times at all - and therefore no apparent trace of older glacial sediments would be expected.

The problem of relative dating of the Dwyka Series in the Zambezi Valley has been tentatively suggested by Chappell and Humphreys to be Lower to Upper Carboniferous. No plant ~~macro~~fossils have been found to date, with the exception of pollens and spores from the base of the Lower Wankie sandstone (Chappell and Humphreys 1970) which Utting believes to be comparable to the lower part of the Gwembe Coal Formation of the Siankondobo Coalfield (which was originally classified as Upper Carboniferous). An assemblage of plant macrofossils from the younger Blac [illegible] les and Coal Group appears to correspond to the [illegible] *Glossopteris* zone which is considered by Plumstead (1967) to be Uppermost Carboniferous and Lower Permian. This would seem to be an upper ceiling on which to base the Dwyka Series' relative age. Interesting work on palaeomagnetic data from the Dwyka Series by McElhinny and Opdyke (1968) would appear to place these sediments in the Lower Carboniferous.

If an early Middle Carboniferous age is accepted for the Rhodesian Dwyka Series, this would infer that an unconformity must have existed somewhere prior to deposition of the Lower Ecca sediments, and that if the palaeomagnetists are to be believed, the time gap would be of some considerable magnitude. Palynology may well prove or disprove the existence of such a break, for during the later Palaeozoic epochs very distinct miospore assemblages and trends existed, with gradual transition zones. Plant evolution in the Upper Carboniferous onwards diversified and exploded so rapidly that the miospore assemblages, reflecting the existing plant populations of the time, would surely indicate any considerable break or gap in the botanical evolutionary chain. It is tentatively suggested that, from the palynological survey on the Matabele Flats Dwyka sediments, miospores are sufficiently diverse and advanced - as indicated by the presence of Disaccate and Monosaccate forms, and in particular Striate genera - to argue strongly for an Upper Carboniferous and even Lower Permian (Sakmarian) dating. No break in miospore sequences was evident, which implies that no significant time gap is apparent. However, Plumstead (pers. com.) does suggest the inevitable possibility that lower sediments with earlier palaeobotanical evidence may not have been recorded due to total earlier glaciation.

1.1 THE IMPORTANCE OF PALYNOLOGY

The palynological investigation of the Matabola Flats borehole core represents the initiation of this branch of research in Rhodesia. Accordingly, it is regarded as necessary to outline briefly the concepts of this discipline.

Palynology is the study of pollen and spores and their dispersal. More specifically, in the case of stratigraphic correlations, it is the study and application of fossil pollen, pre-pollen and sundry plant spores extracted from geological strata. Miospores, (as these small plant reproductive bodies are collectively termed), originate from the fructifications of all plants from the lowest to the highest orders, and are defined as being smaller than 200µ in size.

Necessarily, these plant fossils are grouped as organ genera due to their dispersed nature and very infrequent close association with parent plants. Botanically the study of Upper Palaeozoic palynology is of interest mainly in the evolutionary trends of the miospore structures themselves. Certain emphasis, however, can be placed on their reliability as indicators of indigenous plant groups existing at that time which in turn has phytogeographic implications. Geologically they form a very useful tool for stratigraphic correlation; whilst in general terms they also may reflect the prevailing ecological climatic conditions at the time of deposition.

This potential success and suitability is due to four main factors:-

1. The highly decay-resistant nature of their exines (spore coats) allows them to persist and survive under most conditions bar those of prolonged oxidation, heat (metamorphism from sills and dykes) and excessive transportation (abrasion).
2. Their small size allows them to be dispersed by wind and water over large areas allowing fairly wide lateral distribution (useful in local correlation). Due to size, miospore particles are similar to sedimentary particles in that they may be subjected to the same processes of erosion and deposition. Therefore they may be termed allochthanous or autochthanous, as in sedimentary particles, depending on their distance from source of origin.
3. Miospores exhibit highly variable and often complex structures which facilitates their differentiation into taxonomic groups. This factor is important in the miospore analysis of samples.
4. By virtue of plant reproductory processes, miospores are produced in vast quantities for general and wide dispersal - an important factor in lateral distribution.

The value of palynology has been demonstrated already by significant scientific contributions, e.g. Smith and Butterworth (1968) working on the British Coalfields, and Hart (1965[d], 1967) outlining major stratigraphic sub-divisions of Southern and Central Africa.

The difficulties and limitations in the scientific application of palynology must be borne in mind when embarking upon this field of research.

To be of any inter-regional or international significance, miospore samples should be prepared and extracted in a similar manner, classified in a mutually acceptable system of taxonomy, and statistically counted and analysed in a universally acceptable and comparative way. This unfortunately is not always the case. It is well known that methods of preparation vary in research units and institutions the world over. Due to the differential resistance of miospore exines to certain acid/alkali solutions, various steps in the maceration techniques do affect some miospores and not others (Smith and Butterworth (1968) and others). Some chemical and storage solutions also affect the size of miospores, causing swelling or contraction - this in turn affects one diagnostic characteristic, that of size.

Classification of miospore taxa is an essential part of palynological analysis and correlation, but this unfortunately is a somewhat subjective concept dependent on the individual worker. Major taxonomic schemes may be based on morphological or phylogenetic concepts, e.g. in Africa, Hart's Permian systematic scheme is an example of the former, whilst a scheme proposed by J. Anderson (Ph.D. thesis in press) is an example of the latter. Minor classification problems arise:

a) When generic and sub-generic forms are divided on different physical criteria and the worker's individual interpretation of these e.g. sub-division of the Monosaccites in Southern Africa by Hart (1965) and Bose add Kar (1966) et al.

b) Due to the rule of priority in the Code of Botanical Nomenclature different but synonomous specific and generic names are used.

c) The basic concept of <u>species</u> is a third subjective variable. This may be defined in terms of morphological, chronological or geographical boundaries. (See Approach to Systematics, Chapter 4.1)

In counting and statistical analysis, results once more vary from worker to worker. The reliability of spore counts is also in question. Tomlinson, as quoted by Smith and Butterworth (1967) writing on the statistical limitations and applications of palynology in British Coalfields, maintains that "the errors arising from the preparation and counting of the samples are not too great and that the examination of a few thousand spores on one or two slides provides a satisfactory estimate of the average conditions in the seam section". However, Dimbleby, in the same publication concludes that any species eventually attaining 1 per cent will be represented within the first 250 specimens. Counts are therefore usually conducted on 250 to 500 per sample but may vary according to the worker.

1.2 PREVIOUS PALYNOLOGICAL WORK

No previous palynological work has been published from Rhodesia, although about 100 samples from a borehole in the Mid-Zambezi Valley covering Dwyka to Lower Beaufort (Permian) sediments were sent to Dr. Schaarschmidt in Frankfurt (Bond, 1967). No detailed results have been published as yet.

Regional work in Africa may be summarised as follows:-

Congo:

Dijkstra first studied the megaspores of the Congo in 1955, followed by Pierart (1958) who worked on the spores and pollens of the Glossopteris flora in the Luena Coals, Katanga. Hoeg and Bose (1960) studied the macroplants and some associated miospores of this flora in several areas; whilst later, more detailed taxonomic work was conducted by Bose and Kar (1966), Kar and Bose (1967) and Bose and Maheshwari (1968) on the miospore assemblages of the Kivu, Kindu-Kalima, Walikale and South of Albertville districts. Further stratigraphic sub-division of the Lower Karroo Lukuga Series was suggested on the basis of the macro- and microfloral evidence by these authors.

Tanzania:

Pant (1958) studied the leaves and fructifications and certain associated miospores in the Glossopteris flora (Upper Coal Measures, Songea Series) of Tanzania. This was followed by Hart, who, in 1960 studied the miospore assemblage of the Mchuchuma Coalfield for a Ph.D. thesis.

This information was published (1965, 1965[a]) and based on this Hart suggested the division of the Lower Coal Measures into two major florizones. He later tested the feasibility of this zonal scheme on a sequence in the Songwe-Kiwira Coalfield of Tanzania (1965[d])

Zambia:

Palynological work has been conducted on the Lower Karroo sequences in Zambia (Upper Luangwa Valley) by Utting (pers comm.; Ph.D. thesis, 1970). This information has not been published as yet.

South West Africa:

Leschik (1951) described some specimens from the Norronaub region of South West Africa. The specimens are poorly preserved, and new genera and species have been erected on preservational features.

Malagassy:

Doubinger and Rakotoarivelo (1960) and Jekhowsky, Sah and Lettulier (1960) studied the palynological evidence of the Karroo sequences of the Morondova and Sakoa Basins of Malagassy; Rakotoarivelo (1960, 1970, Ph.D. thesis unpublished 1972) worked in more detail on the coal and shale sequences of the Sakoa Basin. In 1965, Goubin, drawing on material published from 1960 onwards, compiled a detailed stratigraphic scheme dividing the Middle and Upper Karroo sequence into four palynological zones.

South Africa:

In 1954, Rilett described a number of spore genera from a coal sequence near Danhauser in Natal, whilst Plumstead, in 1957 (and again in 1966), illustrated certain spore genera that were typical of the Lower Karroo Coals of South Africa. Hart (1963) published a report and descriptions of a miospore assemblage from a deep borehole core in the Orange Free State. These were considered to be of *Pre-Glossopteris* age. Following this paper, Hart produced a number of taxonomic papers (1964a, 1964b, 1965a, 1966a, 1969a, 1969b) followed by a book on the systematics and distribution of Permian miospores in which the international age of the Karroo system, based on palynology, is discussed. Hart's papers of stratigraphic importance include the division of the Lower Karroo sequence into four major zones, 1966b. (These overlap with the Upper Karroo zones of Goubin in Madagascar to give a rather more complete palynological zonal scheme for these two areas.) Three papers published later (1967a, 1969b, 1970) enlarge on the stratigraphic sub-divisions of Southern and Central Africa and their equivalents, and suggest a computerised system of stratigraphy and palynology.

As may be concluded from this summary, work of comparative interest may be consulted in regard to the following countries in Africa and Malagassy:-

Congo	-	Lower Karroo sequences
Tanzania	-	Lower Karroo sequences
Malagassy	-	Mid to Upper Karroo sequences
South Africa	-	Lower to Upper Karroo sequences

Work in this field in Zambia is as yet unpublished, and no work at all has been conducted in Rhodesia. Information from the Karroo sediments of the latter two countries would indeed serve to consolidate the Karroo palynological knowledge of Africa South of the Sahara, and possibly bring about a closer correlation between the Northern isolated basins of Lower Karroo sediments in the Congo and Tanzania and the large Karroo Basin of South Africa.

2. GEOLOGY

2.1 INTRODUCTION

The Karroo sediments in Rhodesia are contained in two separate basins, the north-western Mid-Zambezi Basin, the northern part of which continues into Zambia, and the south-eastern Limpopo basin, part of which continues into Mocambique and South Africa (see Chart 1). Watson (1960) and Bond (1952, 1967) amongst other authors, describe these basins as being fresh water lakes receiving sediments from surrounding areas of provenance and from the "great elevated tract of Archaean crystalline rocks roughly corresponding in position to the present day Southern Rhodesian watershed" separating them (Watson 1960, p. 5).

The Matabola Flats borehole core is situated in the central (central Sebungwe District) part of the Mid-Zambezi trough, and only this basin will be discussed in any detail. (See Map 2)

As Bond states (1967), the Mid-Zambezi basin is the best known of the Karroo Basins in Central Southern Africa, mainly due to the existence of the vast actual and potential coal resources situated primarily at Wankie, and subsequently found in isolated situations round the perimeter of the basin i.e. the coalfields of Gwembe-Siankondobo, and Kandabwe (both in Zambia); Nebiri, Marowa, Bari, Mafungabusi, Sessami and in the centre of the basin at Lubu, Sebungu and Sengwa (see Walton, 1960 plate 1). As a result of this, extensive drilling and exploration has continued and a fairly detailed picture of the geological history and the stratigraphy of the Lower Karroo sequences has been attained. This has been reviewed by Bond (see 1952 and 1967).

In the region of the Matabola Flats borehole no economic coal seams are encountered. However, the sequences through which the borehole core passes are thick and conformable and represent a continuous stratigraphic column from the oldest Dwyka sediments through the Lower Wankie Sandstone, Black Shales and Coals, Upper Wankie Sandstone and Lower to Mid-Madumabisa Mudstones (k5c of Bond 1952).

Bond and Stocklmayer (1967) describe in detail the lithology of the Dwyka series, and record the younger Lower Karroo sediments found within the 0' – 1 206' limits of the core. The stratigraphic zones are those based on Bond's work in the Central and Eastern parts of the Mid-Zambezi region.

Figure 1 illustrates the succession encountered within the core together with the list of samples that have proved of some palynological interest. Briefly, the lithologies may be summarised as follows:-

(Taken from Bond and Stocklmayer; Geol. Surv. borehole diagram; and personal logging.)

Local Zone	Thickness	
M/K5	200'	Middle Madumabisa Mudstones: The hole was collared in Lower Beaufort Series mudtones, K5c of the local succession. It consists of 200' of pale, silty shales and mudstones grading down to medium grey mudstones and intercalated by one limestone band, one thin carbonaceous band and three bands of sandstones and grits.

Local Zone	Thickness	
L/K5	250'	<u>Lower Madumabisa Mudstones</u>: The succession alternates frequently between carbonaceous shales, thin coal seams, and dark shales. The top spotted (sideritic) coals occur at 225'.
K4	80'	<u>Upper Wankie Sandstone</u>: Sandstones and grits predominate, with one horizon of medium grey mudstone and one of coarse white felspathic sandstone containing bands of coarse pebbles.
K2-3	40'	<u>Black Shale and Coal Group</u>: The fireclays sometimes found above the Black Shales in coal beds appear to be absent. Coal bands are thin, becoming thicker towards the base, and are interbedded with dark shales and carbonaceous shales. The lowest spotted coal bands occur at about 615'.
K1	200'	<u>Lower Wankie Sandstone</u>: Predominantly sandstones and grits, with one quartz-pebble conglomcrate horizon at 290'. Black and white laminated micaceous sandstones grade into a narrow band of medium grey mudstone. Two small series of contorted shales occur between 800' and 840', and near the base another narrow band of medium grey mudstones is evident.

Local Zone	Thickness	
K o	334'	<u>Dwyka Conglomerates and Shales</u>: Three cycles of sedimentation appear, each beginning with conglomerates containing coarse material, and grading up into fine silts and mudstones.
	2'	<u>Sijarira Group</u>: Horizontal beds of pale purplish grey, medium-grained sandstones. The base of the Dwyka sediments lies unconformably upon this, indicating a considerable time gap.

After detailed analysis, Bond and Stocklmayer (1967) have described the depositional cycles in the Dwyka Conglomerates and Shales as three glacial and inter-glacial phases. Features particularly relevant to this are:-

1) The diamictic coarse fractions: the oldest and youngest of these begins with conglomeratic material containing a fresh un-weathered sub-angular to sub-rounded variety of pebbles. These are set in a fine sandy matrix with no evidence of bedding or sorting. They are regarded as tillites of indisputable glacial origin.
2) These tillites are followed by coarse sub-aquecus sandstones and conglomerates showing rough bedding, more uniformity of pebble size and greater degree of pebble rounding. Similar sediments characterise the coarse fraction of the second glacial phase. These beds are interpreted as glacial outwash gravel deposits formed in deltaic conditions.

3) Of the fine silts and mudstones, the oldest sequence is the thickest and most distinctly varved. It is characterised by light and dark-grey pairs of varves which are regularly banded, averaging 0,1 inches (about 2 mm) in thickness and containing fresh, angular, but well-sorted mineral and rock fragments. The second and third inter-glacial phases possess no clear banding or varves, but they are charakterised by very large rafted mineral grains which are considered to be of glacial origin.

2.2 HISTORY OF SEDIMENTATION

Four major sedimentary patterns are seen in the Mid-Zambezi Karroo sequences (Bond, 1967), of which two are evident in the Matabola Flats borehole-core.

1. The first cycle includes the Dwyka glacial succession, and Lower Wankie sandstone, followed by the fine-grained Black Shale and coal beds.
2. The second cycle commences with the coarse Upper Wankie sandstone, which grades into the fine grained Madumabisa Mudstones.

Basically deposition took place in a large lake or trough (following the basin concept of Bond (1952, 1967) and Watson (1958)) after minor sub-basins or depressions of the valley floor had been filled with the earliest Karroo sediments. This trough extends in a north-east, south-westerly direction coincident with the drainage course followed by the Zambezi River today.

Sedimentary and Tectonic Sequence

1. Sedimentation began in glacial times with the deposition of fluvio-glacial sediments (tillites, conglomerates and varves) during the final phases and retreat of the Dwyka ice (whether this was a local mountainous ice cap or a large continental ice centre is yet a moot point). The first depositional phase, therefore, consists of these sediments filling depressions in the form of scattered lakes on the gently uneven Pre-Karroo floor.

Following this, the Zambezi trough is considered to have taken the form of a single large lake which was initially very shallow but which soon developed an asymmetrical form with the steepest slope in the north (Watson, 1960). Transport into the basin appears to have been roughly radial throughout the entire Karroo System (Bond, 1967). In Lower and Middle Ecca times tectonic movement appears to have been localised in the east and south of the basin, resulting in active transport of sandstones into the lake i.e. (Lower Wankie sandstones, as seen in the Matabola Flats borehole core). Simultaneously minor sandstones were deposited in the western and northern portions, but conditions become shallower and relatively more stable here, thereby supporting the flourishing growth of the coal-forming flora at Wankie, Lubimbi and Nkendabwe (the Gwembe coal-formation). Carbonaceous sediments are found throughout the rest of the basin in Middle Ecca times, but for the most part these consist of thin alternating coals, carbonaceous shales and carbonaceous mudstones indicating a continuous "heaving and settling movement of no great magnitude" in lacustrine conditions (Bond, op. cit.) The Black Shales and Coals (K^{2-3}) encountered in the present borehole core are characteristic of the latter conditions.

2. Rela..vely rapid inundation of the now shallow basin resulted in the start of the second depositional and tectonic phase in Upper Ecca times. Here Bond (1967) regards "local uplift probably by faulting, combined with a general tendency for the basin floor to sink" to be the probable cause of well developed sandstones

in the west and south (Upper Wankie sandstones). Relative stability in the north and east resulted in absent or thin sandstone horizons.

A long period of "quiescence and minimum relief" (Bond, 1967 p. 188), with gradual slumping and deposition resulted in the lacustrine to estuarine conditions in which the fine grained Madumabisa sandstones were accumulated.

Lower Madumabisa mudstones (or Upper Carbonaceous mudstones referred to by Watson (1960)), are regarded by the latter author as indicative of rapid, deeper flooding than the Lower Black Coals and Shales (or Lower Carbonaceous mudstones). This is explained by Watson thus - "it is as if likely sites for prolonged coal-flora growth was everywhere too rapidly overwhelmed to allow time for coal formation". During Mid-Madumabisa times "persistent argillaceous sedimentation went hand-in-hand with a sinking of the Pre-Karroo floor", in other words, slow gentle deposition prevailed in a mature environment in which sedimentation occurred at the same rate as the sinking of the basin. All the sediments indicate a continuous subaqueous (but with fluctuating depth) environment; Bond has even suggested that some of "the Upper Madumabisa sandstones might be loess-like wind-blown dusts".

Once sedimentation had filled the Zambezi basin to capacity, and sinking had ceased, a long period of erosion occurred. This was followed by the third phase of sedimentation which was initiated by large scale folding and faulting with deepening of the basin and strong rejuvenation of relief all around it.

No sediments of the third depositional phase are, however, encountered in the Matabola Flats borehole core.

2.3 CLIMATE

The climates envisaged in the Karroo sequence pass through four major phases beginning with the Dwyka glacial epoch and ending in Upper Karroo times with hot, desert to semi-desert conditions just prior to large scale vulcanism. Within the Lower Karroo sediments, and certainly those through which the Matabola Flats borehole core passes, three phases transitional to one another may be seen.

1. A sub-Arctic climate is found in the Dwyka sequence in which the prevailing climate must have been very cold as deduced from the lithological evidence of glaciation. In some views conditions would have been similar to that experienced on the fringe of Antarctica today; however, should the local mountain ice-cap theory prevail, conditions may be expected to be similar to that on the margins of the Himalayan or Swiss mountain glaciers.
2. Sub-Arctic to cold temperate climates are believed to have prevailed in the Lower Wankie sandstone, Black Shales and Coals and Upper Wankie sandstone in (Bond, 1967; Lacey, 1961, 1969; Plumstead, 1966, 1967). i.e. Lower and Middle Ecca times. Plant evidence, indicating the prevailing climate, is lacking in the Matabola Flats borehole core. Thus the climate is deduced here on the basis of
   a) lithological correlation with those areas that are fossiliferous, and
   b) logical reasoning following on the cessation of glaciation.

3. Cool to warm temperate climates are considered to have evolved during Upper Ecca and Lower Beaufort times i.e. Lower and Middle Madumabisa Shales. Once again the borehole core yields no macrofloral evidence, but by stratigraphic and lithological comparison this may be deduced.

After a considerable erosional gap and time interval the climate of Upper Karroo times is regarded as having ameliorate , warm and hot temperate

2.4 PALAEONTOLOGY

Although no macrofloral evidence was found throughout the borehole sequence, it is pertinent to note the plant assemblages usually associated with the relevant stratigraphic units.

1. Dwyka:
   Until the present study no plant or animal fossils had been found in Dwyka sediments, bar a report that miospores were numerous in the inter-glacial sediments (Bond, 1967) quoting initial report from Dr. Schaarschmidt, Frankfurt.. (However, during the present study, a fossil insect wing was recovered from shales just above the oldest tillite, whilst miospores, plant cuticle, woody fragments and the oil algae Botryococus are found well-preserved and in relatively abundant quantities. This indicates a well-developed local flora.)

2. Lower Wankie Sandstone:
   Few macrofossils have been found to date. However, Chappell and Humphreys (1970) report the presence of pollen and spores in one shale sample at the base of the lower Wankie Sandstone. These are quantitatively and qualitatively similar to assemblages recorded from the lower part of the Gwembe Coal Formation of the Siankondobo Coalfield, Kazinze Basin, Zambia (according to Utting, personally communicating with the authors.

Of the macroflora fossils, Lacey (1961) quotes the occurrence of <u>Lycopodiopsis</u>, a <u>Glossopteris</u> scale leaf and <u>Carpolithus</u> sp. Of the Southern African major macroplant zones of Plumstead (1967), the one to which these fossils (and those of the Black Shales and Coals and Upper Wankie Sandstone) belong is the Permo-Carboniferous Mixed floral zone.

3. <u>Black Shales and Coals</u>:

Evidence of the prevailing flora is found in a few sites around Wankie. Amongst the most recent sites was that discovered by Chappell (1969, quoted by Bond 1970) in the central part of the Zambezi Basin. Here the relative abundance of <u>Gangamopteris</u> leaves "indicates Lower Ecca affinities, a hint that the Wankie Coal may be a little older than was previously thought likely" (Bond 1970, p. 469). Lacey (1961, 1969) collected only <u>Lycopod</u> megaspores, <u>Glossopteris</u> leaf fragments, and "stems (indet.)", whilst Walton (1929, quoted by Lacey, 1961) recorded <u>Glossopteris</u> species, fragments of Equisetalean stems and fern-like plants.

4. <u>Upper Wankie Sandstone</u>:

The celebrated Upper Wankie sandstone flora discovered by Lightfoot near Wankie, occurs in one shale horizon. The significance of this flora is in its strange assemblage, that of the normal Ecca <u>Glossopteris/Gangamopteris</u> elements, but with a "northern element" consisting of eight species within the genera <u>Sphenophyllum</u>, <u>Pecopteris</u> and <u>Chansitheca</u> (Lacey, 1961). These floral elements are considered to have existed in Europe at more or less the same time, but in warm conditions, which has led Jongmans (1952, quoted by Lacey, op. cit., p. 44) to suggest "that the climate was only a little more severe (in Rhodesia) than that existing in the Northern Hemisphere at the same time". Lacey does, however, point out that "this incursion of the Northern flora might merely indicate a temporary climatic change". Plumstead (1965), however, does discuss the view that Southern Hemisphere macroplants may have been undergoing parallel evolution to the Northern Hemisphere forms and therefore may not have migrated from the North or necessarily lived in identical conditions.

The fact that <u>Glossopteris</u> and in particular <u>Gangamopteris</u> leaves found in the Upper Wankie sandstone are much smaller than their equivalents in South Africa also leads to the suggestion that the climate had become much warmer. The cold-loving Gangamopterids were probably struggling for survival and slowly dying out during this period, (a factor also noted in the reduction of one of the major miospore taxa, viz. Monosaccate miospores).

5, <u>Lower Madumabisa Mudstones</u>:

The characteristic elements are those of the <u>Glossopteris</u> flora, with <u>Phyllotheca</u>, <u>Taeniopteris</u> and <u>Vertebraria</u>, and wood with seasonal growth rings. The "northern element" in the shale of the Upper Wankie sandstone is not apparently found in this series, and <u>Gangamopteris obovata (cyclopteroides)</u> becomes rare to absent. (This is the beginning of Plumstead's Transition Phase or true <u>Glossopteris</u> flora.)

6. <u>Middle Madumabisa Mudstone:</u>

This is once more characterised by the *Glossopteris* flora, with *Taeniopteris*, *Phyllotheca* and *Sphenopteris* and fossil wood. Plumstead (1967) is of the opinion that highlands supported the *Glossopteris* and other wooded elements, whilst the swamps and low-lands abounded in equisetalean reeds and lower plant orders.

2.5 ENVIRONMENTS AND DEPOSITION

The presence of certain chemical elements (or their deficiency) and the occurrence of certain minerals has recently been studied in order to obtain information on depositional environments.

1. Bond (1967, and pers. comm.) has reported the work on oxygen and carbon isotope ratios by Keith and Weber (1964) in which the marine or fresh water origin of sediments (e.g. limestones) may be determined. Following on from this Professor Keith is reported to have conducted a C^{13} deficiency analysis on eight samples taken stratigraphically through the Madumabisa Mudstones. The abundance of vegetation in and around the lake in the Mid-Zambezi Valley was deduced by climatic and other means to have been at its peak in the middle of the stratigraphic sequence. This proved to be correct in terms of the C^{13} deficiency analysis, as C^{13}, normally very deficient in high vegetation, reached its peak of deficiency in the middle.

   The (O^{16}/O^{18}) oxygen isotope ratios were employed on the same samples as above. Information from this analysis showed that "Southern Africa was closer to the rotational pole in Madumabisa Mudstone times" (Bond, 1967, p. 190).

   None of the above analyses have been conducted on the Matabola Flats borehole core; however, the implications of a non-marine origin of the sediments (particularly limestones), and the confirmation of periods of peak vegetation during Madumabisa Mudstones do have a direct bearing on the subsequent analysis of the palynological content of the present borehole core.

2. The presence of abundant siderite in all but the lowest coals of the Black Shales and Coals series is considered to be of stratigraphic importance as well as enlightening on the origin of deposition. It is a known fact that anaerobic bacteria, in an oxygen-deficient environment, with certain pH and Eh limits will release sulphur and other elements from organic matter in order to obtain energy. The resultant sulphur (and often carbonates) combines with ionised iron to form siderite and pyrite (Tschudy and Scott 1970). Therefore an abundance of these minerals is considered to indicate mildly acid lake water with a good supply of rotting organic matter which is in an anaerobic and stagnant condition well below the aerated surface of the water. This is the depositional environment thus deduced for the accumulation of the Black Shales and Coals siderite-spotted and spherulitic sediments.

2.5 SUMMARY OF STRATIGRAPHIC SEQUENCES

From evidence previously published it may be deduced that the following conditions prevailed during the stratigraphic sequences of the Matabola Flats borehole core.

Dwyka: The depositional environment was one of a shallow lake or valley into which the glacial and inter-glacial sediments were (psited under morainic, deltaic and lacustrine conditions. The climate must have been sub-arctic (to frigid?) and the macroflora unknown but expected to be fairly varied.

Wankie Sandstone: The depositional conditions are deduced to have been shallow water and deltaic, with rapid erosion at source, flooding and rapid deposition. The climate is considered to have been similar to that of the Dwyka with the ice front in the vicinity of the lake. The plants found indicate an early, cold-loving flora assigned by Plumstead to her Zone III, or Permo-Carboniferous Mixed flora which occur in South Africa during and just after the glacial era.

Black Shales and Coals: The lacustrine conditions due to the subsidence of the basin (with minor vertical movements) is thought to have caused the accumulation of the fine sediments characteristic of this stratigraphic unit. The climate is thought to have been cold temperate, supporting a cold-loving Gangamopteris and early Glossopteris flora. The lack of good plant preservation in the carbonaceous beds has been a point of discussion. Watson (1960) is of the view that plant accumulation was slow and exposed to oxidation for long periods prior to burial, resulting in little recognisable organic tissue but a large quantity of oxidised organic detritus (micrinite). The presence of siderite indicates an anaerobic mildly acid lake bed with a large supply of rotting organic material.

Upper Wankie Sandstone: Represents a shallowing of the basin with rapid coarse sedimentation once more. One deep water grey mudstone facies bearing plant remains indicates a minor subsidence. The climate is considered to have been cold-temperate but becoming warmer (reduction in size of leaves of cold-loving Gangamopterid plants).

The flora is typical of the Middle Ecca Mixed Glossopteris flora (Zone III, Plumstead, 1967) with an unusual "Northern element".

Madumabisa Mudstones: A long period of relative stability and minimum relief with gradual sinking of the basin occurred resulting in lacustrine to estuarine depositional conditions. The climate is envisaged as being cool temperate to warm temperate, supporting a woodland (Glossopterid) and mudflats (Equisetalean) flora. The C^{13} deficiency analyses indicate an abundance of vegetation in and around the swamps and lakes during deposition of the Madumabisa Mudstones, with a climax in Mid-Madumabisa times. The limestones characteristic of these sediments are considered to be of fresh water origin, as indicated by oxygen and carbon isotope research.

3. METHODS

3.1 SAMPLES EXAMINED

Sampling was commenced on the full sequence of the 1 206' borehole core. Samples were collected at an average of 6' intervals, but more frequently in specific or promising horizons, and more spaced where large depths of sandstone were encountered. A total of 105 samples were collected, labelled by depth in feet and separately packaged (to guard against contamination). See figure 1 for sampling levels.

Identical sets of the samples, (taken by breaking one small borehole core sample vertically in half), are housed in the Geology Department of the University of Rhodesia, Salisbury, and in the Bernard Price Institute, University of the Witwatersrand, Johannesburg.

3.2 EXTRACTION TECHNIQUES

Introduction:

In 1969, some months were spent on establishing a suitable miospore extraction process, generally applicable to the variety of Karroo lithologies encountered (Falcon, 1969). Many experiments and much control testing eventually led to a maceration schedule based on the combination of various techniques recommended from a number of sources. The schedule is laid out in Table 1, and a detailed discussion follows.

Briefly, the overriding difficulty was that of successful standardization. The samples varied from coarse sandstones and fine siliceous shales or mudstones with little carbonaceous content, to high grade coals and carbonaceous shales. In order to assess the overall palynological picture, one basic method was employed in the initial maceration of the entire suite of samples. (Steps 1, 2A and 3). This simplified method served to show the degree of carbonaceous content of every sample. Where little or no plant matter existed (other than resistant woody elements, cuticle or miospores) this schedule proved sufficient in yielding relatively clean miospores, e.g. as in the case of Dwyka samples and most Madumabisa Shales down to a level of about 260'. However, where carbonaceous content was high (>50 per cent), oxidation techniques (step 2B) had to be employed in order to release the hardy miospore assemblages. Length of oxidation varied with the amount of carbon present: this fell into two groups -
a) that with oxidation of 10 minutes, and
b) that with oxidation of 1 hour. It was not possible to treat the high grade coals satisfactorily in the more specialised manner they require. Thus only carbonaceous shales were studied within the coal-banded sequences.

Results of maceration on this broad basis were such that variations in miospore yield and preservation were made distinguishable. Those samples yielding little or no miospore content after two separate maceration tests were considered to be barren.

TABULATED METHOD OF MIOSPORE EXTRACTION - TABLE 1

STEP 1	CRUSHING	
	SAMPLE DETAILS	EQUIPMENT
SAMPLE WEIGHT	2 - 3 gms	
CRUSHED SIZE	½" FRAGMENTS OR 75" MESH SIEVE B.S.	MORTAR AND PESTLE
	N.B.:- POUNDING NOT CRUSHING MOVEMENT	

STEP 2A	CARBONATE DISPERSAL		DEMINERALISATION		SILICO-FLUORITE DISPERSAL	
	REMOVAL OF CARBONATE COMPOUNDS		REMOVAL OF SILICATE COMPOUNDS		REMOVAL OF SILICO-FLUORITE (SOLUBLE GEL) RESULTING FROM DEMINERALISATION	
	SAMPLE DETAILS	EQUIPMENT	SAMPLE DETAILS	EQUIPMENT	SAMPLE DETAILS	EQUIPMENT
CHEMICAL	50% HCL	POLYTHENE BEAKERS	20 ML. 50% HF	POLYTHENE BEAKER	50% HCL	GLASS TUBE
TIME	1-2 HOURS		2-3 DAYS		WASH THROUGH X 1-2	
CONDITIONS	HEATED	WATER-BATH	HEATED - 12 HRS. ON 12 HRS. OFF	WATER-BATH	WARM ACID; OR HEATED SAMPLE	WATER-BATH
WASHING	1-4 X DISTILLED H_2O	CENTPIFUGE	4 X DISTILLED H_2O	CENTRIFUGE	3 X DISTILLED H_2O	CENTRIFUGE

I

THOSE SAMPLES HIGH IN ORGANIC CONTENT MUST BE FURTHER PROCESSED

II

MOUNTING MAY BE COMMENCED FROM STEP 2:-

a) WHEN INITIALLY SAMPLING ENTIRE CORE (STANDARD PROCESS)

b) WHEN SPECIFIC SAMPLE IS LOW IN CARBONACEOUS CONTENT

ST[illegible]	OXIDATION		HUMIC-ACID DISPERSION	
	REMOVAL OF DETRITAL ORGANIC COMPOUNDS, (CELLULOSE, HEMICELLULOSE AND LIGNIN; AND DERIVATIVES)		REMOVAL OF HUMIC-ACIDS RESULTING FROM OXIDATION	
	SAMPLE DETAILS	EQUIPMENT	SAMPLE DETAILS	EQUIPMENT
CHEMICAL	40 mls. SCHULTZ SOLN.	GLASS TUBE	40 mls. 30% Na_2CO_3	GLASS TUBE
TIME	a) 10 MINS. b) 1 HOUR		(IMMEDIATE)	
CONDITIONS	HEATED	WATER-BATH	HEAT TO BOILING	
WASHING	2 X DISTILLED H_2O	CENTRIFUGE	3 X DISTILLED H_2O	CENTRIFUGE

STEP 3	MOUNTING	
	MIX CELLOSIZE OR CLEARCOL WITH ORGANIC RESIDUE	STORAGE TUBE
	PLACE 1 DROP ON COVERSLIP	DRIED
	MOUNTED IN CANADA BALSAM ON SLIDE	

Detailed Extraction Methods:

The methods employed in miospore extraction may be grouped into physical or chemical categories. Briefly, chemical extraction includes demineralisation by the use of acids, and the dissolving of organic compounds by oxidation and alkali solution. Physical Methods include heavy media separation, centrifugation and other devices using specific gravity differences between organic and inorganic matter. Palaeozoic sediments require the use of the latter methods prior to complex chemical treatment (thereby concentrating the organic matter to be macerated) or subsequent to chemical treatment (in this case removing any remairing mineral grains and residual organic detritus).

The references, upon which the extraction experiments were based, are summarised as follows:-

ANDERSEN, Svend T.	1965	Mounting media and mounting techniques. In Handbook of Palaeontological Techniques. W.H. Freeman and Co.
ANDREWS, H.N.	1961	Studies in Palaeobotany; John Wiley and Sons; New York and London.
BALME, B.E. and HASSELL, C.W.	1962	Upper Devonian Spores from the Canning Basin, Western Australia. Micropalaeontology, vd. 8, p.p. 1-28.

FELIX, Charles J.	1961	An Introduction to Palynology (see ANDREWS).
	1963	Mechanical sample disaggregation Palynology. Micropalaeontology, vol. 9, No. 3, p.p. 337-339.
GRAY, Jane	1965	Palynological Techniques, Handbook of Palaeontological Techniques (p. 471). W.H. Freeman and Co.
HART, G.F.	1962	Palynology - Key to Stratigraphy. S.A. J. of Sc., vol. 58, No. 12, p.p. 365-374.
LEE, Howard W.	1964	A Modified Method of Coal Maceration and a simple Technique for Slide Preparation. Micropalaeontology, vol. 10, No. 4, p.p. 486-490.
NEVES, R.	1958	Upper Carboniferous Plant Spore Assemblages from the Gastrioceras Suborenum Horizon, North Staffs. Geolog. Mag. vol. XCV, No. 1.

Most constructive correspondence was conducted with Dr. B.E. Balme; Dr. M. Muir; Dr. W.S. Lacey; (describing Dr. Kulkarni's Methods); Dr. H. Owens; Dr. J. Utting and J. Anderson.

Physical Methods:

The size of the sample to be macerated from each rock matrix type should vary due to the probable abundance of miospores in a carbonaceous shale, or the sparce distribution as in certain sandstones. However, due to the standardising techniques employed in this research programme a standard weight of 2-3 grams, per sample was treated.

The crushing of the sample was conducted in a pestle and mortar, using a gentle pounding movement, rather than grinding, in order to prevent powdering. This was found to be the most common and adequate method for the compact, hard Palaeozoic sedimentary rocks - as opposed to the ultrasonic vibrations (tank or probe) or domestic liquidisers used frequently in less compact and more recent sediments (Andrews, et al. 1961).

Methods of disaggregating and dispersing sample material (following crushing) by physical means are numerous, but are often limited in usefulness. These methods involve flotation, centrifugation, ultrasonic generation, and the use of pyredine and H_2O_2. In many cases these methods, involving organic and inorganic separation, are used in conjunction with chemical maceration techniques (as in the present system of extraction) or before or after these methods. For example, Neves (1958) and Hart (1963) introduce a bromine flotation step prior to chemical treatment which they maintain appears to increase mechanical disintegration thereby facilitating HF attack on mineral residues with little or no argillacegus matter remaining. Utting (correspondence) incorporates a Zinc-Bromine (2,4 s.g.) flotation step following chemical treatment for the separation of any inorganic detritus still remaining. In experimenting with this method, the removal of the bromine by means of a sintered glass funnel and, subsequently, by boiling proved beyond the scope of the facilities available. Another problem in using the bromine flotation method is that the organic (to the top) and inorganic (to the bottom of the liquid) separation under gravity is very slow and incomplete. Muir (correspondence) also cautions against differential flotation, thereby biasing samples intended for numerical analysis.

Centrifugation was found to be the most adaptable and suitable method of physical separation, and this was employed during the chemical maceration steps. Disadvantages of this method involved basically the difficulty in attaining a speed at which differential centrifugation and separation of the organic and inorganic particles occurred. However, the advantage of relatively short time taken in sample treatment outweighed the disadvantages. This method is further discussed under chemical treatment, cleaning methods.

Use of the ultrasonic generator as a means of dispersal also displays merits and disadvantages.

This mechanical device disperses a) fine-grained sediments, b) precipitates or flocculates, and c) insoluble fine particles adhering to and partially obscuring miospore grains. It may be used also in conjunction with or alternating between chemical steps. The method by which it functions is that of high-frequency vibrations, instigated by a generator, passing through a fluid in the transducer tank. An operational disadvantage is that the machine is unpleasantly noisy at low-frequency ranges, whilst being of little use and more trouble at high frequencies. A further disadvantage, as found in experimentation, is that of the probable higher incidence of spore breakage (Grey reporting on Tschudy, 1965).

The use of H_2O_2 and pyredine for disaggregation was considered too time-consuming and more suited to specialised treatment of high ranking coals (Raistrick, 1934, and Muir, correspondence).

<u>Chemical treatment</u>

Chemical treatment consists of:-

1. Demineralisation, and
2. Oxidation with subsequent solution of oxidised organic matter.

In the case of highly carbonaceous rocks - roughly over 50 per cent carbonaceous matter - oxidation is the primary objective in chemical treatment; whilst in less carbonaceous sediments ($\pm$50 per cent organic matter) a fuller treatment of demineralisation and oxidation is conducted.

Where there is little or no residual carbonaceous matter, only acid treatment for demineralisation is necessary. Therefore, the quantitative mineralogical composition of rock will go in the specific steps to be included in the maceration technique, whilst the qualitative or chemical composition of such constituents controls the length of time taken at each step, e.g. a high ranking coal may take up to 7 days to be sufficiently oxidised, whilst lower grades would take far less time. (In the case of the Rhodesian samples a standard demineralisation scheme was adopted throughout, whilst oxidation steps fell into three categories; a) those samples not requiring oxidation, b) those requiring a short intensive (10 minute) treatment, and c) those requiring longer (a standard time of 1 hour)). Individual samples may well have been perfected in cleanliness but bulk sample treatment precluded this.

1. Demineralisation:- In order to understand the demineralisation process, the inorganic compounds found in sedimentary rocks are considered.

   1. Carbonates

      In sedimentary matrices of interest to palynologists, the anhydrous carbonates of the calcite group are the most commonly found members within the carbonate suite. Removal of carbonates from matrices is commonly achieved by use of HCl. This step is routinely adopted before further demineralisation (using HF) as calcium salts react with HF forming calcium fluoride (CaF_2) which takes the form of a fine precipitate difficult to remove. 10 per cent HCl solution is recommended by Grey, whilst Muir, Utting, Owens and Anderson operate on a 50 per cent solution. This latter strength is used in the present extraction scheme. It is essential to ascertain that the reaction is complete. This is achieved by adding fresh acid from time to time, and often by increasing the concentration. (The acid may cease to function when no longer potent thereby leaving undissolved carbonates present).

HNO_3 in 10 per cent aqueous solution is also used to remove carbonates, as in the case of coal, but this may be closely allied to the function of oxidation. Phosphoric or ortho-phosphoric acid may also be used, and is reported to dissolve some Al, Ti and Fe compounds in addition to the carbonates. However, H_3PO_4 has the disadvantage of requiring large volumes of the acid, and the subsequent formation of further phosphate compounds (apparently soluble in HNO_3 or Schultz solution.) Acetic acid in 10 per cent solution is used by Balme for dissolving carbonates, whilst some workers also work with 6 per cent solution of Formic acid.

2. Sulphates

The most common sulphates to be encountered are Anhydrite ($CaSO_4$) and Gypsum ($CaSO_4.2H_2O$). Once again hot HCl or HNO_3 in 10 per cent aqueous solution will dissolve these minerals, as does Sodium Thiosulphate (hypo).

3. Sulphides

In this group pyrite ($Fe\,S_2$), marcasite (FeS_2) and siderite ($FeCO_3$) are most common, and are frequent constituents of sedimentary rocks and in particular are found in the Black Shales and Coals and Lower Madumabisa Mudstones. As stated by Gray (p. 549) "the blue and bluish-grey colour of shales and mudstones sometimes associated with coal seams is usually due to finely disseminated iron sulphides". In carbonaceous residues sulphide compounds are notably opaque and may be angular particles or carbon-like globules aggregating with microfossils. Sulphides are usually soluble in cold conc. HNO_3 (10 minutes to 24 hours) but the presence of marcasite may require that the sample be boiled in HNO_3. The latter treatment does usually dissolve pyrite as well. Gray noted that as HNO_3 is a strong oxident, it may cause damage through over oxidation. Therefore the removal of metallic sulphides by heavy media separation may prove a better alternative. Despite experimentation with HNO_3 abundant sulphides and Fe carbonates could not be entirely eradicated in some samples, particularly those in Lower Black Shales and Coals horizons.

4. Silicates

Silicates and more especially quartz comprise a great proportion of clastic rocks and sediments, and their removal has proved to be most effective by the use of hydrous hydrofluoric acid (HF). This step follows the HCl treatment for the elimination of carbonates due to the formation of the flocculate calcium fluoride (CaF_2) with HF. Special precautions were taken in the handling of HF, with the necessary equipment and body protection.

The concentration of HF is usually that of commercial HF, i.e. 48-52 per cent acid, although Gray reports that 72 per cent, and up to 90 per cent HF, have been used by some palynologists who claim to have better results than at lower concentrations. An ethyl-alcohol solution is reportedly even more reactive than the water solution. In correspondence (Balme, Owens and Utting) 40 per cent and 50 per cent HF are recommended, and these subsequently proved as effective as the other strengths. It is of interest to note that in one particular case - that of Mr. Kulkarni (working on Karroo material in England) - hard shaley samples taken from Lower Karroo sediments, although immersed in commercial HF for several hours, gave no better results and disintegrated no further than those samples which were merely treated in Schultz solution.

The time required for dissolving silicates varies from minutes to several hours, depending on their abundance and on the continuing strength of the acid solution. For most samples 8-12 hours is sufficient, with additional gentle boiling to speed the reaction. Heat causes the additional volatilization of silicon fluoride (SiF_4) as well as TiF_4 (thereby extracting Titanium). Gray mentions that from personal experience, boiling in HF up to 20 minutes does not appear to damage the spores. In recommendations (received) all but one of the workers used water baths at 40°C and 60°C; three allowed the samples to stand for 2-3 days, five up to 20 hours and one (Balme) for only 10 minutes. The method found to give optimum clarity and greater consistency through the borehole samples was that of 2-3 days heating periods (40°C water bath).

Balme, Muir and Gray mention the necessity of following the HF treatment by an HCl "wash"; this is even more essential in Palaeozoic rocks (Muir - correspondence). Gray enlarges on this by explaining that in a water environment, certain compounds and salts (colloidal silica, silica fluorides of Al, CaF_2, MgF_2, KF, NaF) may precipitate following the use of HF. Therefore, in order to maintain these siliceous compounds in solution, warm HCl (10-25 per cent) must be used as a rinse - most of these compounds are soluble in this reagent. Balme (1960) confirms this and states that the reaction of HF on minerals of the clay-mica suite produces complex insoluble fluorides which appear in slides, with related inorganic substances, as finely dispersed gel-like masses obscuring microfossils and diluting the concentrate of spores. Fine-grained marine sediments, especially rich in chloritic minerals, he maintains, yield the highest proportion of insoluble reaction products. It is interesting to note that only those methods of Balme and Muir include this stage. It was personally noted that the addition of this step did indeed clear much of the gel-like dirt otherwise encountered on slides.

2. Oxidation of organic matter

Organic material found within sediments and derived originally from plants is deposited primarily into the soil and secondarily by means of erosion and deposition into the normally accruing sediments in lakes, bogs, deltas and other natural catchment areas.

This organic material may be in the form of light, air-borne or water-suspended matter, or whole plants or parts thereof - including leaves, twigs, reproductive structures (flowers and fruits) and remains of all these in the form of cuticular structures.

Chemically the composition of the numerous structural elements in vascular plants is taken as being virtually the same throughout the several hundred million years since their origin. This is assumed to be as waxes, polysaccharides and lignin h always been present despite the countless varieties of flora through geological history that have contributed to their existence (Gray, p. 539).

Accumulating organic matter will include resins, waxes, proteins, carbohydrates (sugars, starches), derived carbohydrates (the Polysaccharides, hemicelluloses, cellulose) and lignin - and the very resistant, inert pollen and spore exines. Of these, cellulose, hemicellulose and lignin are most worthy of note as they form the largest part, by weight, of plants and, therefore, are the most common of the contributed compounds. They also form the "parent material" to most of the unwanted organic detritus of interest to palynologists.

The methods for eradicating organic detritus include alkali treatment, acetylation and oxidation. These steps refer mainly to those sediments containing complex organic compounds possibly still in heterogeneous states of decomposition. In the case of Pre-Cenozoic rocks where organic matter is almost, if not completely altered (as in coals and carbonaceous shales) the combined oxidation and subsequent solubilization of the maceration compounds (in the form of humic acids) in alkali solution appears to be the most suitable technique and is, therefore, the most widely used method.

Oxidants used by palynologists vary to some extent due to the chemical and physical variations and maturity of the organic fractions, and the reactions of different types of matrices. It was, therefore, advisable to experiment with the oxidant most suited to the rock samples to be studied and with its length of time in use. Also, great care had to be taken in optically checking the oxidising sample for destruction of the actual pollen spores. It is known that pollen and spores are oxidisable and may be destroyed in certain circumstances. They exhibit variable resistance to different oxidants – as seen when one reagent is harmless to some spores but highly destructive to others, e.g. <u>Crassispora</u> (Gray reporting on Kosanke) is more resistant to fuming nitric acid than Schultz solution; whilst in other samples, small microspores (20-25 μ range) appear to have been destroyed to a greater extent in fuming HNO_3 than Schultz solution. The state of microspore preservation may also influence their recovery with one reagent and not another. Schultz solution followed by KOH has also been reported as producing spores consistently larger than the same species from samples treated by fuming HNO_3.

Several possibilities exist when the oxidation step is commenced (Muir, pers. comm.) - the following is a condensed version of the numerous variations offered by Gray.

1. The spores will be light yellow, transparent and require no further attention.
2. The spores will be dark, and need oxidation by:-
   a. H_2O_2 (Gray, p. 565) - an aqueous solution of 10-30 per cent concentration. In the case of high-grade coals (88-90 per cent carbon range) this method is reported to produce the best quality spores and largest variety.
   b. HNO_3 - Nitric acid reagents are vigorous and must be carefully watched. They are used in the maceration

of coal, carbonaceous shales, etc. Spores are reportedly lost in boiling conc. HNO_3, but cold HNO_3 is considered milder than Schultz solution. Maceration time varies from 1 hour up to 14 days in conc. HNO_3.

c. Fuming HNO_3 This may be used on coals either independently or in conjunction with conc. HNO_3 as used by Utting (see table). Some authors report that dispersal of humic acid using alkali solution is unnecessary following this method of oxidation. (Utting follows this step with the optional use of Zn-Br heavy media separation.) Lack of equipment precluded trial of this method.

d. HNO_3-Br_2 - Zetsche's method. Both HNO_3 and Br_2 are strong oxidants, with the latter beginning the oxidation process and the former completing the task. Muir explains the theory of this: "Since spore exines are composed of unsaturated hydrocarbons, they can be saturated and then oxidised as much as necessary". In her opinion, this is no more effective than Schultz solution.

e. HNO_3 + NaCl - Muir (correspondence) recommends this as an excellent reagent on very high grade coals, the reaction being as yet unexplained. As coal maceration was not attempted, this oxidant was not tried.

f. <u>Schultz Solution</u> - $HNO_3 + KClO_3$ - This is the most popular for coal and other carbonaceous sedimentary rocks and has been adopted in this maceration schedule. It action is also more easily controlled than that of conc. HNO_3. Theoretically, although both reagents are strong oxidants, it is thought that Chloric acid ($HClO_3$), resulting in their chemical reaction, is the active oxidant and bleach. This is probably more powerful than either of the original oxidants above. Schultz Solution is pre_ red in two ways, one suitable for high grade coals and the other for general matrices (see Gray, p. 558). Only the latter was used in the present series of samples.

Following on oxidation comes the removal of the oxidised humic products by solubilization and dispersion by water (Owens and Anderson) or alkali solution - hydroxides and alkali-carbonate solutions. Hydroxide solutions are those commonly chosen, some being more vigorous than others, even when dilute. It must be noted that in strong alkali solution, destruction of the miospores may occur possibly due to swelling, and this occurs more especially in overmacerated residues. 5-10 per cent KOH or NaOH is used by several workers (Balme, Kulkarni, Neves and Hart), but Muir recommends an alkali-carbonate, Na_2CO_3 (10 per cent). Her reason is that cessation of the reaction can be seen more easily (effervescence stops) and thus there is less danger of the solution becoming too alkaline. For this reason the latter (Na_2CO_3) alkali solution was used.

Cleaning of the various reagents between steps is of the utmost importance. This may be carried out by repeated washing and careful decanting, or by centrifugation with careful stirring in between. The latter was found to be preferable due to time and equipment available, although, possibly the former method is more thorough (although slower). In normal centrifugation, all matter in suspension is compacted at the base of the tube which is ideal in washing. But where certain organic and inorganic debris must be selectively removed, centrifugation must be controlled.

(Microfossils and debris must be of different sizes and density for this to be feasible.) Short-time, fast centrifugation will remove fine detritus, whilst drastic decrease in speed and duration will merely concentrate large and heavy particles allowing microfossils still in suspension to be decanted off and concentrated, (Gray, Muir, and Andrews).

In most cases following oxidation, a somewhat more sophisticated method of washing is deemed necessary to separate the organic residues (spores, pollen and cuticle) from the alkali soluble oxidised humic compounds. It is for this purpose that sinter glass funnels with various modifications have been introduced. When one of these is attached to a pressure flask with a rubber blow ball (Owens, correspondence), agitation of the organic residue is achieved by forcing air up into the system and thereby escaping through the filter. A mild suction pump may be added to this to give an alternate blowing and sucking movement. In personal experience it was found that attaching a rubber tube from the tap and allowing water in place of air to