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PHOSPHORUS BEARING MINERALS IN
IRON ORES AND THEIR POSSIBLE
BENEFICIATION

by

El-Sayed Ibrahim Elgayar (B.Sc., M.Sc.)

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SUMMARY

Modern electron optical techniques together with X-ray and mineralogical examination have been used to study the occurrence and form of phosphorus bearing minerals in iron ores.

Three ores have been studied - Bahariya and Aswan from Egypt and Frodingham ironstone from U.K. The iron in the Bahariya iron ore is mainly as hematite and goethite. The gangue minerals are halite, gypsum, barite, quartz and calcite. Iron content is between 49.8 to 63.2% and phosphorus 0.14 to 0.34%. The phosphorus occurs as very fine particles of apatite which are distributed throughout the ore. Removal of the phosphorus would require very fine grinding followed by acid leaching. Aswan iron ore is an oolitic iron ore; the iron content between 41-57% and phosphorus content 0.1 to 2.9%. It is mainly hematitic with variable quantities of quartz, apatite and small amount of clay minerals. In the oolitic iron ore beds, apatite occurs in the hematite matrix; filling in the pores of the oolithic surfaces, or as matrix cementing the ooliths with the hematite grains. In sandstone claybeds the distribution of the apatite is mainly in the matrix. It is suggested that the liberation size for the apatite would be ~80 µm and flotation concentration could be applied for the removal of apatite from Aswan ore.

Frodingham ironstone occurs in the lower Jurassic bed of the South Humberside area. The average iron content is 25% and the phosphorus is 0.32%. Seven mineral phases were identified by X-ray; calcite, quartz, chamosite, hematite, siderite, apatite, and chlorite. Apatite occurs as very fine grains in the hematite and chamosite ooliths; as matrix of fine grains intergrown with chamosite and calcite grains; and as anhedral and surrounded grains in the ooliths (8-28 µm).

It is suggested that two processes are possible for the dephosphorisation; the Flox process or a reduction roast followed by fine grinding, magnetic separation, and acid leaching.

KEY WORDS - IRON ORE, PHOSPHORUS, MINERALOGY, ELECTRON-PROBE ANALYSIS, DEPHOSPHORISATION
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7.1 Superimposed iron and phosphorus electron microprobe line diagrams for the same oolith
A century ago iron ore production was only 2 percent of what it is at present \(^{(1)}\). This grew at a constant rate up to 1900; then slowed down in the period 1900-1940 but has been expanding rapidly since, as is shown in Figure 1.1. The latest estimation of world iron ore reserves is $500 \times 10^9$ tonnes and therefore even at present day usage there is approximately 500 years of iron ore availability \(^{(1)}\).

Perhaps more important than the increasing consumption of the world's iron ore reserves is the change over from the use of lower grade ore of the U.K., and France-Belgium-Luxembourg-Saar (Minette type) to the higher grade ores of the present day discoveries in Australia, Brazil, Canada and Venezuela. Thus, the former countries have changed from being self sufficient iron ore producers (and even exporters) to large importers. Similarly, the United States has become a major importer although there are large deposits in that country of relatively high grade but more difficult to smelt ores (the Taconites of Mesabi). Further, Japan has become one of the top three iron and steel producers of the world despite having no significant home ore production. The chief reason for the switch from lower grade iron ore (<30% Fe) to the 55+% Fe contents is the need to produce the lowest cost products.

Always steelmakers are striving to get maximum production from each production unit to offset the high labour cost and inadequate depreciation allowances. This need to maximise the output from each production unit has resulted in the
FIG. 1.1

PRODUCTION OF IRON ORE,
(1855 - 1980)
growth of oxygen steel making processes. Molten iron is converted to steel by direct oxidation of impurities with gaseous oxygen. This is putting further emphasis on the blast furnaces to increase output in order to satisfy the prodigious appetite of these oxygen steelmaking plants for hot metal.

The demands on blast furnace performance have resulted in a number of important innovations in its operation. Such refinements as increased use of prepared burdens, high-top pressure, blast-moisture control, and injection of hydrocarbon fuels into the tuyere zone are all directed towards improving and increasing the output of existing blast furnaces and at the same time aiming to decrease costs. The most important of these innovations is the use of a prepared iron ore burden which, inevitably, contains impurities, e.g. silica, alumina, lime, magnesia and others which are usually at a lower content but very harmful, e.g. phosphorus, copper, zinc, lead, arsenic, sulphur, chloride, etc. These elements influence greatly the practical value of a deposit and can even make it useless under the present state of technology and economics.

Of these impurities, phosphorus has been a decisive factor in determining the appropriate process for the steel refining of the hot metal from the blast furnace. Unfortunately, almost all the phosphorus of the charge materials is absorbed into the molten iron. It may be recalled that when the Bessemer process was developed using converters with acid
lining in 1856, it was possible to use only ores with a very low phosphorus content, generally under 0.03% due to the inability of the acid slags to absorb phosphate minerals. This limited the use of the Bessemer process in numerous countries, chief among these were the U.K. (Midland ores contained high P) and Sweden (Kiruna iron ores contained up to 2% P).

The Thomas or Basic Bessemer process using a converter with a basic lining made it possible to handle hot metal with a high phosphorus (‡ 1.8% P) and enabled the use of ores containing a high percentage of phosphorus to be charged to the blast furnace. However, there were also many ores in the world with an intermediate phosphorus content, which were suitable neither for the Bessemer nor the Thomas process, and the only process which was suitable was the basic Siemens-Martin process (Open-Hearth).

This process treated hot metal of any phosphorus content mixed with scrap in a very wide proportion, and was the most flexible method available to the iron and steel industry at that time for the intermediate phosphorus containing ores.

The developments in steelmaking during the last 30 years which have included the top and bottom oxygen blown converters (BO F and OBM or Q-DOP) have again imposed limitations on the amount of phosphorus that is permitted in the hot metal to facilitate high production and
economic working. This has re-opened the possible limitation of phosphorus content in the blast furnace charge. If the process metallurgist requires a hot metal to contain less than 0.3% P then the blast furnace charge must contain the appropriate phosphorus content in relation to the iron content. For instance, ores with 50% Fe must not exceed 0.15% phosphorus, and if the iron content is 65%, the phosphorus content in the ores could reach 0.2% without exceeding 0.3% in the hot metal.

From Table 1.1 \(^{(2)}\) it is clear that oxygen steelmaking will be predominant in many countries and the proportion will reach 65% of the steel produced in 1985. Thus, it would appear that there will be increasing pressure from the iron and steelmakers of the world to require the ore manufacturers to provide materials of as low a phosphorus content as possible which may require beneficition to be practised on an ever increasing scale.

Another approach to iron and steelmaking has been the growth of direct reduction processes within the past twenty years and is more applicable to developing countries of the third world. These processes aim to produce iron units from ore without the use of metallurgical coke which is a fast decreasing commodity although it may be replaced by formed-coke in the future. In the same paper Miller (loc cit) forecast an eightfold increase in the production of direct-reduced iron product (Table 1.2). However, it must be said that the quality of the iron oxide feed
directly effects the quality of reduced product and also controls the overall economics of the reduction and steelmaking process. It is suggested that the phosphorus in the oxide raw material must be between 0.01 to 0.05% and thus, no alleviation of the phosphorus problem for the ore producer is apparent.

Therefore, it is important at the present time to examine the origin of phosphorus in some of the world's iron ore deposits and suggest possible beneficication methods to remove this unwanted element.
2. World Iron Ore Resources

Iron is one of the most abundant and widely distributed elements in the earth's crust, constituting approximately 5% of the total. Its occurrence in acceptable quantities for economic extraction is extensive in very many parts of the world.

As for all ores, the principal difficulty lies in the definition of the economic and technical viability of a deposit. Not only does the iron content of the rocks determine the value but also the associated gangue minerals and, in particular, the 'tramp' elements, e.g. phosphorus and sulphur. With present and possible future techniques of mining and benefication ore reserves are difficult to estimate but Table 2.1 gives a guide to total world resources (1). As mentioned earlier, the iron ores are very widespread and in many cases at high iron contents, representing very many years of useful reserves. The following notes are in amplification of the data in table 2.1:

(a) The ore resources of Europe, N. America and Russia contain many of the lower grade deposits (e.g. Taconites, Minette type) which do not appear in most of the other regions. These latter are generally higher grade ores or ill defined low grade material.

(b) The current total for world resources, although very large, must be considered a conservative
<table>
<thead>
<tr>
<th>Region</th>
<th>Resources Quantity</th>
<th>Resources %</th>
<th>Potential ore Quantity</th>
<th>Potential ore %</th>
<th>Total resources Quantity</th>
<th>Total resources %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Africa</td>
<td>6,800</td>
<td>3</td>
<td>24,500</td>
<td>5</td>
<td>31,300</td>
<td>4</td>
</tr>
<tr>
<td>Middle East, Asia and the Far East</td>
<td>17,300</td>
<td>7</td>
<td>54,200</td>
<td>10</td>
<td>71,500</td>
<td>9</td>
</tr>
<tr>
<td>Australia, New Zealand and New Caledonia</td>
<td>16,800</td>
<td>7</td>
<td>Vast</td>
<td></td>
<td>16,800 Plus</td>
<td>2</td>
</tr>
<tr>
<td>Canada and West Indies</td>
<td>36,300</td>
<td>14</td>
<td>89,400</td>
<td>17</td>
<td>125,700</td>
<td>16</td>
</tr>
<tr>
<td>Europe</td>
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<td>8</td>
<td>12,800</td>
<td>2</td>
<td>34,100</td>
<td>4</td>
</tr>
<tr>
<td>South America</td>
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<td>14</td>
<td>58,400</td>
<td>11</td>
<td>92,500</td>
<td>12</td>
</tr>
<tr>
<td>USSR</td>
<td>110,500</td>
<td>44</td>
<td>193,800</td>
<td>36</td>
<td>304,300</td>
<td>39</td>
</tr>
<tr>
<td>United States of America, Puerto Rico, Mexico and Central America</td>
<td>8,200</td>
<td>3</td>
<td>98,100</td>
<td>19</td>
<td>106,300</td>
<td>14</td>
</tr>
<tr>
<td>TOTAL</td>
<td>251,300</td>
<td>100</td>
<td>531,200</td>
<td>100</td>
<td>782,500</td>
<td>100</td>
</tr>
</tbody>
</table>
estimate because potential ore in many parts of the world, such as Africa, Asia, Australia and South America, is difficult to assess on the basis of available information.

In fact, no total has been given for Australia in the potential ore category.

(c) While there are very large iron ore resources for each continent (certainly satisfactory supply for the future expansion of steel industries) it should be noted that there are decided irregularities in distribution. Many countries within well endowed continents have no resources, and local scarcities exist even within regions containing vast iron ore deposits. It is obvious that in the future the problem will be to select the best quality of material for economic development and to minimise ore transportation costs for local situations.

Resolving these two factors will frequently point towards the importation of high-grade iron ores rather than the exploitation of indigenous deposits which have physical chemical and mining disadvantages.

(d) Quantitatively, the greatest resources of iron in the world are found in low-grade deposits, particularly in siliceous iron formations.
It should be noted that in the totals for the United States of America, relatively stringent criteria have been applied in regard to classifying reserves, potential ore being twelve times as great as reserves. This is caused by the fact that reserves in the United States are commonly determined on the sole basis of economic factors.
2.1 Classification of Iron Deposits by Type

Iron ore is classified in many different ways to satisfy the needs of engineers, metallurgists, chemists or economists, or the demands of commerce. The system adopted here for classifying iron deposits is to be found in the United Nations survey of world iron ore resources, 1970 (1). In it descriptive geology and empirical data, rather than genetic processes are stressed.

Descriptive data is used and iron deposits are grouped with reference to a few well-known types and examples. The mineralogy, texture, physical features, composition, kinds of associated rocks, mineral alteration and geological setting are described objectively; and familiar examples are chosen to illustrate the nature and type of deposits. In this thesis four types only of the iron deposits classification are presented, and they are given below.

2.1.1 Lake Superior type

Characteristically, this is a thin banded cherty rock with iron-rich layers that are found in many sedimentary facies. Granules and oolites composed of both chert and iron minerals have a typical textural feature, and the rocks are practically free of clastic material, except in the transitional border zones or in distinct, well defined members within the formation. Continuous layers of the Lake Superior type of iron formations often extend for hundreds of miles along the margin of a geosynclinal basin. The formations may thicken and thin from a few metres to
several hundred metres and occasionally may increase to over 300 metres. There is a close association of these minerals with dolomite, quartzite, black carbon-bearing shales and slate, chert breccias and volcanic rocks. There are Lake Superior types present in late pre-Cambrian rock groups in nearly all parts of the world. Two major types may be distinguished:

a - the low-grade ore (quartzite formations with hematite) including Itabirite; Taconite, Metacoonite; Jaspilite and Iron-Quartzite.

b - the high-grade type (almost pure hematite).
   Examples:-
   - Mesabi Range;
   - Brazil - Minas Gerais;
   - Venezuela - Cerro Bolivar deposit;
   - Krivoi Rog, Ukraine U.S.S.R.

2.1.2 Minette type

The oolitic-texture rocks of this type of iron formation are composed mainly of siderite and iron silicates, such as chamosite or iron chlorides, and of goethite-siderite and chamosite. Many beds containing a high proportion of fine grained clastic material are transitional to sideritic or chamositic mudstones or sandstones. Sand grains may form the nuclei of siderite oolites, as in the Salzgitter and Peace River beds. Occasionally, fossil fragments are present in the oolites. Silica (which is usually more than 20 percent) is present generally in chamosite or iron-silicate minerals or in lesser amounts in clastic grains, rather than chert. The phosphorus content is higher
than that in the cherty Lake Superior types. These iron formations are closely associated with black carbonaceous shale, mudstones and sandy shales, which apparently formed in marine or brackish water in shallow basins. Minette ores are practically important and widespread in Europe, e.g. the sideritic or chamositic ironstones of northern England, the Minette ores of the Lorraine district of France and the Federal Republic of Germany, the Salzgitter ores of lower Saxony, and many other Jurassic beds in northern Europe. This type is also found in the iron formations of the Peace River area in Alberta, and in the enriched Aswan iron ore deposits in Egypt.

2.1.3 Bilbao type

This occurs in massive oxide deposits derived from the weathering of underlying iron carbonate, and in iron deposits of irregular shape occurring as iron carbonate below the water-table and as goethite and hematite above the water-table. The principal deposits of this type are the following:

- Bilbao, Spain
- Erzberg, Austria
- Rudabanya, Hungary
- Khenifra, French Morocco
- Ouenza, Boukadra and other deposits in Algeria
- Djerissa, Douaria and Tamera in Tunisia
- Bahariya, Egypt
- Cumberland, England
2.1.4 Kiruna type

This is characterised by massive magnetite intrusions, usually associated with granitoid of syenite type, accompanied by a relatively higher apatite content.

The principal deposits are:
- Dover, New Jersey in the U.S.A.
- Lyon Mountain, New York, in the Adirondacks in the U.S.A.
- Iron Mountain, in Missouri, U.S.A.
- Pilot Knob, Missouri
- Cerro de Mercado (Durango State), Mexico
- Algarrabo, El Tofo, Romeral in Chile

Further details about classification of iron ore deposits can be obtained from references (1) and (3).
3.1 The Origin of Marine Phosphorite

The greater part of the phosphorites of the world are of marine origin. There are three principal hypotheses of their formation; biolithic, chemical and volcanic-sedimentary.

3.1.1 Biolithic hypothesis

This is based on the suggestions by Backwalker (4) and Bushinskii (5) that marine originating phosphate deposits are based upon sudden changes in the marine environment such as major temperature shifts or volcanic submarine and earth movements. It is suggested that these resulted in catastrophic slaughter of the fish population with subsequent decay of the bodies on the sea floor producing phosphate rich deposits.

3.1.2 Chemical hypothesis

About 200 minerals are known that contain one percent or more \( P_2O_5 \). Most of the phosphorus in the earth's crust, however, belongs to the apatite mineral group (6).

The earth's crust contains about 0.27% \( P_2O_5 \). Most phosphorus is carried to the sea as phosphate minerals or adsorbed on iron or aluminium hydroxides or clay, but some is carried in particulate or dissolved organic compounds, and about one-sixth of the total is carried by this method into solution. The phosphorus content of most rivers and lake waters ranges from about 0.01 to 0.5 parts per million (6). The solution of phosphorus in the water may be raised by
local conditions, e.g. high dissolved organic phosphate content, high Ca/Mg ratio, high pH, high temperature and suitable nucleation sites. It has been established that the solubility of apatite in sea water decreases with increasing pH (7) and also is precipitated in low oxygen potential environments (8). These conditions are conducive to the precipitation of phosphate on parts of the sea bed forming sedimentary deposits (7).

3.1.3 Volcanic sedimentary hypothesis

It is known that only poor phosphorites are found directly in volcanic-sedimentary and volcanic siliceous-schistose deposits. Bushinskii (5) expected that the greatest precipitation of phosphates should occur near the point of appearance of submarine thermal springs, in places where the highest concentrations of phosphate solutions are found. With increasing distance from the points of appearance, the phosphates to an ever increasing degree would be mixed with sea water and diluted by it. Accordingly, the possibilities for their chemical precipitation into sediments should decrease sharply.

3.2 The Origin of Phosphates in Iron Ores

There are two principal sources of iron at the earth's surface; volcanic activity and continental erosion.

Borchert (9) suggested that the iron in the trivalent state is insoluble in the presence of oxygen, when its maximum solubility is of the order of 1 mg.m$^{-3}$. Any
significant solubility of iron in the bivalent state occurs only in acid solutions with a pH value below 7 and then only under reducing conditions and in the absence of free oxygen. In these circumstances its solubility may reach a magnitude of $5 \times 10^4 \text{ mg} \cdot \text{m}^{-3}$. In the presence of free oxygen iron is chiefly precipitated in trivalent state. He also noticed that corresponding to the normal conditions of sea current the dissolved iron moves predominantly towards the oxygen rich shallow sea where it is deposited as limonitic ooliths. However, a considerable proportion of the dissolved iron is precipitated in the CO$_2$ zone to form sideritic clay ironstone. The CO$_2$ zone is between the bottom zone where no oxidation occurs and the shallow water zone where there is oxygen (See Fig. 3.1).

Cook and McElhinny (10) noticed that most of the phosphorus is associated with the iron in an adsorbed form or perhaps in a poorly crystalline iron phosphate phase. But in some deposits, apatite occurs as discrete laminae and thin beds.

Kaplan and Rittenberg (11) pointed out that in reducing environments the phosphate ions tend to stay in solution due to bacterial production of H$_2$S and CO$_2$ which bind Fe$^{2+}$ and Ca$^{2+}$ respectively as sulphides and carbonates; the removal of these cations from solution would keep the phosphate ion in solution.

Mackereth (12) pointed out that the phosphate was released in anoxic pore waters by decomposition of phosphorus.
O₂ - Zone: pH: 7.2 - 8.5
\[ E_h = 0.05 \text{ to } 0.4 \]
Most important constituents: O₂
\[ \text{CO}_2, \text{Cl}^-, \text{SO}_4^{2-}, \text{CO}_3^{2-}, \text{HCO}_3^-, \text{NO}_3^- \]

CO₂ - Zone pH: 6 - 7.5
\[ E_h = 0.05 \text{ to } 0.2 \]
Most important constituents: HCO₃⁻, Cl⁻, SO₄²⁻, PO₄³⁻, NO₂⁻

H₂S - Zone pH: 7.2 - 9
\[ E_h = -0.2 \text{ to } -0.5 \]
Most important constituents:
\[ \text{H}_2\text{S}, \text{C}_x\text{H}_y\text{O}_2\text{C}_x\text{H}_y, \text{NH}_4\text{OH} \]
Pyrite - sapropel - marl
Without Benthos abundant bacteria

FIG. 3.1
SCHEMATIC SECTION OF IRON ORE FACIES AND PHYSICO-CHEMICAL CONDITIONS
containing organic matter and, by the reduction of hydrated ferric oxides which, under oxidising conditions (CO$_2$ zone), bind phosphate onto their surfaces.

Nriagu and Dell (13) suggested that in many instances the ferric oxyhydroxides precipitated from the overlying water was stabilised in the sediments by their subsequent interaction with the phosphorus in the interstitial fluid's as follows:

\[ n \text{Fe(OH)}_3 + m \text{HPO}_4^{2-} \rightarrow \text{Fe}_n \text{(HPO}_4\text{)}_m \text{(OH)}_{3n-2m} + 2m\text{OH}^{-} \]

Ferrosoferric hydroxyphosphates are most stable under oxidising conditions.

Borchert (loc cit.) also noticed that smaller amounts of Fe(HCO$_3$)$_2$ migrated into zones of increasing reduction potential (phosphorus precipitated) to combine with silica originating in the alkaline H$_2$S zone so as to form chamosite.

Nriagu (14) suggested that the Fe$^{2+}$ can be used as an effective precipitant for removing orthophosphates, particularly from anaerobic sludge digesters where the efficiency of other precipitation mechanisms may be low. They show that the hydrated ferric oxides bind phosphate onto their surfaces under oxidising conditions and thus cause the presence of phosphate in iron ores. It is also clear that Fe occurs in the phosphate deposits as a Fe(HCO$_3$)$_3$, chamosite and iron phosphate phases.
4. IRON ORE IN EGYPT

4.0. Introduction

The Ancient Egyptians occasionally used iron and iron ores. This is proved by the finding of iron beads in some of the tombs and by the well known piece of iron found in the inner joints of the Great Pyramid at Giza (15).

The iron ores in Egypt have thus been known from as early as the Eighteenth Dynasty (about 1350 B.C. to 158 B.C.) but have not received much attention until recently (15). This is probably due to the absence of coal in Egypt and to the occurrence of favourably situated rich iron ores in other countries.

The establishment of an iron and steel plant at the end of the last decade was a national requirement to meet the additional steel capacity necessary for reconstruction and development.

There are two main deposits of iron ore in Egypt:- at Aswan and at Bahariya Oasis (Fig. 4.1), these deposits are briefly described below.

Aswan

The ore is in oolitic sedimentary deposits (Minette type) occurring as bands in the middle group of the Nubian Series, interbedded with clays and ferruginous sandstones. The beds are horizontal and the ore bands are either at the surface
or close thereto. Hence mining is by the opencast method. Reserve estimates vary from 158 million to 300 million tonnes (16).

The average grade is given by Gheith (16) as 48% Fe and 12% SiO₂ and 1.0% P.

Now that electricity is being produced from the Aswan Dam generators it is possible to mine the iron ores especially those near Aswan.

Bahariya

The iron ore deposits of the Bahariya Oasis occur at four localities:
Nasser area; Gebel Ghorabi; El Harra; El Gedida.
The deposits are rather irregular forming a succession of lenticular beds of the lower middle Eocene age. The beds vary in thickness from less than 1 m. to more than 12 m. It is suggested (17) that the ore has been formed by slow but intense metasomatic replacement of the lower Middle Eocene limestone with partial replacement of the uppermost section of the Bahariya formation sandstones and the lower member of the Radwan formation. The overburden is variable. In parts, it forms a thin cover of less than a metre, consisting of alluvium, mainly sands and gravels derived from iron formations and/or quartzite. In other parts, the overburden is formed of thick beds of clastics mainly clays, Quartzites and sandstone. In these parts the overburden may reach a thickness of up to 40 metres.
The ore at Bahariya is of a dark brown to dark red colour and consists mainly of hematite and goethite with pockets of ochre and manganese oxides. The gangue minerals are Halite, Gypsum, Baryte, Quartz and Calcite.

The ore reserves are given as 195 million tonnes with an average grade of 52.5% Fe and 6% SiO₂ and 0.12% P.

The iron ore mines of the Bahariya Oasis in the Eastern desert represent the main potential iron ore resources for the new iron and steelmaking complex because they contain a high percentage of Fe and a low percentage of P.

The present thesis comprises, for the first time, a combined detailed geological, mineralogical and geochemical investigation on the Aswan and the Bahariya (El Gedida) iron ores which represent two different types, the Minette and the Bilbao types.

The aim is to achieve a better understanding of the origin of phosphates and their minerals together with their amount and distribution in the ores. It is believed that the realisation of such can be a help in planning the economic use of the ores and in exploration for similar ores in other areas all over the world.
4.1 Bahariya Iron Ore

4.1.1 Nasser area (18)

The iron beds of the Nasser area outcrop along a strip of about 5.0 Km in an east-west direction. The northern part of the Nasser area is a Grabenal wadi and the iron bed is covered by different thicknesses of overburden. Topographically the Nasser area is longitudinally divided into eastern and western parts. In the "eastern" section the ore is mainly found in the form of a single bed whose average thickness is approximately 8.2 m. Its colour is dark brown to dark reddish, partly hard to friable, chiefly hematite with pockets and bands of hard goethite especially at the top where there is always a hard cavernous goethite cap.

Occasionally, the ore is black with a low percentage of manganese minerals. In the far western part of the Nasser area there are two beds separated by a clay layer whose thickness varies from 0.5 m to 5.9 m. The ore is generally hard, cavernous, mainly goethite, brown to dark brown at the top, whereas at the lower parts it is dark brown to dark reddish and black. Mostly it is hard, friable hematite but turns to black colour especially when manganiferrous. The thickness of the iron ore beds varies from 1.0 to 25.7 m. Halite and gypsum form the main impurities in the ore throughout the Nasser area.

The thickness of overburden varies from a few centimetres to 21.6 m. In the south, the overburden is mainly formed
of unconsolidated sand alluvium and iron gravels with salt pockets. Over the rest of the area there are beds of clay and quartz.

4.1.2 Gebel Ghorabi (18)

Ghorabi is topographically divided into 5 parts, separated by either fault or erosional wadis. (North, East, South, West and Central Ghorabi.)

The ore beds are generally covered by 0.50 m thick of alluvium cap. The upper part of the ore is mainly formed of massive hard, brown, cavernous goethite. The lower part is separated from the upper by a clay bed of an average thickness 0.70 m. This lower part is mainly reddish brown to blue-black, friable to slightly hard manganiferrous hematite ore or mixed hematite and goethite. The average thickness of the iron beds in the Ghorabi area is 8.5 m. Quartz, gypsum, calcite and halite are the main gangue minerals associated with the ore.

4.1.3 El-Harra area (18)

The iron deposit of El-Harra is represented by one bed which splits (in the central part of the area) into a number of clastic intercalations of several bands of iron which may reach 5 in number.

The ore bed is covered by a thin cap of wadi deposits with an average thickness 1.2 m. This cover is mainly formed
of unconsolidated sand and gravels of iron and/or quartzite. Occasionally, remnants of a thin quartzite bed are observed to cover the iron bed. The ore is formed generally of hard, dark brown, goethite with yellow ochrous pockets. The goethite is cellular (especially at the southern parts of the area) generally massive and blocky and sometimes fragmental. Hematite intercalations are observed at the lower parts of the ore bed.

In the central part of the area, the ore is covered by a thick overburden, varying from 0.7 m to 14.8 m with an average thickness of 6.1 m. Clay bands form 47% of the total overburden thickness, the alluvium 20%, the sandstone 15%, the quartzite 12% and the limestone 6%. The hematite ore, in the central part, is mainly reddish brown to dark brown to bluish black, friable and porous. Generally hard fragmental goethite pockets and highly manganiferous bands interpenetrate the ore body. Bands of yellow clayey ochre, also alternate with the friable hematitic ore. The average thickness of the iron bed at El-Harra area is 6.2 m.

The main gangue minerals associated with the ore are gypsum, calcite and halite. These minerals are irregularly distributed in the ore body. However, gypsum is mainly observed filling cracks and calcite is seen in appreciable amounts in the peripheral parts of the area.

4.1.4 El-Gedida area
4.1.4.1 Topography
El-Gedida, the biggest and richest iron ore deposit of Bahariya, is located on the northern plateau about 15 km
south-east of the northern extremity of the Bahariya Depression. The area is topographically distinguished into three parts.

1 - the high area in the centre,
2 - the low surrounding valleys, and
3 - the lower plateau limestone surrounding the whole Gedida area.

The central high area is a levelled plateau intersected by numerous valleys, upon which appear five conspicuous peaks. The highest of these peaks is Lyons Hill which is 255 m above sea level (Fig. 4.2a). The average relief difference between the high hillocks and the plateau level is about 40 m, whereas that between the plateau surface and the surrounding valleys ranges from 20-50 m. The scarp face of this high area displays an irregular shape to form several indentations.

4.1.4.2 Geology
A geological map of El-Gedida area, scale 1:5000 covering an area of 17.4 km² (Fig. 4-2b) was prepared by Amer (19). It aimed basically to clarify the relationship between the iron ore deposit and the bounding limestone plateau. The outcrops on the map were measured by a grid survey for the area; the structural features were determined partly from field observations and partly from subsurface data.
The geological ages for the outcrops of the different formations recorded in this area are given below (19):

Recent
1. Superficial deposits and sand-dunes

Oligocene
2. The Radwan Formation, sandstone and quartzites.

Eocene
4. Gazzum Formation, white limestone (upper middle Eocene to upper Eocene).
5. Nagb Formation, pink limestone (lower middle Eocene).

Cretaceous
6. Bahariya Formation, sandstone and clays (Cenomanian).

Besides appearing in pits of the surrounding valleys, iron ore beds in El-Gedida form the central high area and take the same stratigraphic position as the limestones. They are underlaid by Bahariya Formation and overlaid by the Radwan Formation.

The maximum thickness recorded for the ore in the area is 27.1 m as a continuous formation without any interbeds. The clay interbeds are generally absent in the central body but are frequently observed at the peripheries of the ore body; the thickness of the interbeds ranges from 0.6 to
7.7 m. On the other hand, the Eocene deposits equivalent to the iron ore beds have a thickness of about 200 m and are followed upwards by the Radwan Formation.

The following section has been measured (19) in the central high area, at Lyons Hill, (data is partly from outcrops and partly from subsurface).

Top: (The Radwan Formation, quartzite and sandstone) 22.5 m
11 - Banded cavernous iron ore (goethite, brown, yellow hematitic and ochrous). 10.5 m
10 - Banded cavernous iron ore (hematite, dark brown with hard pockets of goethite) 10.2 m
9 - Friable iron ore (hematite, blackish or bluish brown) in the lower part conglomeratic 2.8 m
(9, 10, 11 constitute the iron ores; 23.5 m)

8 - Barite Zone, loose yellow sands with idiomorphic small barite crystals 0.3 m
7 - Sandstone; yellow, medium-grained, indurated, cross-bedded, with thin ferruginous bands 6.0 m
6 - Sandstone, clayey, fine-grained, light-grey with green plastic clay underbeds, yellow ochrous violet or blue shades of colours are observed 3.5 m
5 - Sandstone, light yellow to dark brown, cross-bedded with white quartz grains 10.5 m
4 - Sandstone - white to greyish white, friable to indurated, fine to very coarse-grained. Thin clayey interbeds with oblique laminations become common downwards 24.6 m

3 - Siltstone, white with bluish shade, clayey and massive in parts, with rare cross bedding planes 18.0 m

2 - Sandstone, fine-grained with white quartz grains, yellow with white interbeds at some places, rather compact and ferruginous 6.0 m

1 - Clay, grey, sticky, blocky with very thin sand streaks in parts 1.1 m

(1 to 8 constitute the Bahariya Formations; and continue further downwards) 70 m

The following is the subsurface data obtained during drilling of El-Gedida water well No. 1 (Andrawis (20)) that lies in the eastern valley 2.5 km east of Lyons Hill.

Top
1 - Wadifills and sandy clay 9.3 m

2 - Hematite and hydrogoethite, with clay and sandy clay interbeds 32.1 m

3 - Clay and sandstone alternating bands: the sandstone is colourless, milky white, pink, ferruginous, fine to coarse-grained mostly medium grained, angular to subrounded, compact, hard, siliceous with traces of pyrite, gypsum,
anhydrite, carbonaceous matter and rare white to whitish yellow limestone. Clays are yellow to yellowish brown, pale grey medium hard, sticky

4 - Sandstone and clay: sandstone is colourless, yellowish, white, medium to coarse-grained, subangular to subrounded, partly calcareous, with traces of pyrite and gypsum. Clay is brownish red to yellowish brown, grey, medium hard and sticky

(3 and 4 are very poorly fossilised and the alternation of sandstone and clay represents shallow oscillatory sea deposits. Post-Cenomanian age is assigned to it and probably Oligocene)

5 - Sandstone with alternating shale interbeds: sandstone is fine to medium grained, angular to subangular, calcareous, partly glauconitic with traces of carbonaceous matter. Shale is dark grey, greenish grey and brownish red, medium to hard, flaky with traces of pyrite. This interval includes microfossils of the Cenomanian age

6 - Sandstone with streaks of shale: sandstone is white to pale yellow and pink, coarse to very coarse-grained, rounded to subrounded, partly glauconitic, loosely cemented. The shale is grey, greenish grey to brownish red, medium hard, flaky with traces of pyrite
Andrawis (20) (op. cit.), recorded in sections 3 and 4 only one species of *Truncorotaloides rohri* Bronnimann and Bermudez which he claimed to indicate Middle Eocene age. Also rare species of Cibicides and Ammodiscus were recorded; but they have no significance for age determination. On lithological basis, he considered this formation to be of Oligocene age "for its resemblance with El-Hamra Formation of Said and Issawi (21)". This conclusion was erroneous, since El-Hamra Formation was assigned to upper Eocene by Said and Issawi and not to Oligocene.

4.1.4.3 Classification of El-Gedida iron ores

1. Hard massive ore
2. Banded cavernous ore
3. Friable ore
4. Black manganiferrous ore
5. Oolitic-pisolotic ore
6. Ferruginous sandstone

These six ore types differ from each other in mineralogical composition, and in properties of the hand specimen. They also have a fixed field relation with respect to the geological setting of the ore body (Basta and Amer (17)).

4.1.4.4 Mineralogy of El-Gedida iron ores

4.1.4.4.1 Hard massive ore

This ore type consists mainly of crystalline hematite or goethite. It is chiefly hematitic, hard, crystalline, with a deep reddish-brown colour and a submetallic lustre. It is usually massive but may be occasionally cavernous, in the latter case crystalline barite is observed filling the
cavities. The grade and density of this type is higher than those of the others. Its density is about 2.72 tonne/m³ and its iron content averages about 60%. This ore type occurs in the central high area of El-Gedida south of the line F (see geological map, Fig. 4.2b), with a thickness ranging from 0.5 to 8.0 m. Barite which is the chief gangue mineral in this ore type, is mainly present in the lower parts of the ore body in contact with the sandstone of the Bahariya Formation, where it occurs as small euhedral and brecciated crystals.

Halite and gypsum are two other gangue minerals but are much less common than barite, they usually occur in the upper parts of the ore body particularly filling cracks.

4.1.4.4.2 Banded Cavernous ore

This is formed as an intimate association of goethite and hematite and has a brown or yellowish brown colour. It is usually cavernous, and these are filled with red or yellow ochre powder and sometimes with black manganiferous powder. In El-Gedida area, it is found in the central high area in the north of line F (Fig. 4.2b) and partially underlies the previous ore type.

Density varies from 2.60 to 2.67 tonne/m³, and the iron content is about 57%. Halite, gypsum, barites are the main gangue minerals in the ore. These occur generally as surface cracks fillings or in the form of a salty crust. They are sometimes found intercalated with the ore or in the form of small limited pockets.
4.1.4.4.3 Friable ore

This is formed mainly from fine crystalline goethite and hematite as well as much amorphous limonite and ochrous clays and has various colours grading from bright yellow to dull dark brown with occasional blue or violet tints. The ore is soft and friable. Its density is approximately 2.0 tonne/m$^3$ and iron content about 55%. The most common gangue is the clayey material.

4.1.4.4.4. Black manganiferous ore

This ore type is usually friable. It is constituted from hematite and goethite together with an amount of manganese minerals enough to give it its characteristic black colour.

In El-Gedida, this ore type is present underlying the banded Cavernous ore in the central high area and also at the peripheries of the ore body. Its density is 2.3 tonne/m$^3$ and average iron content 53% with manganese 3.5%. The common gangue minerals consist of fine dispersed quartz grains which increase towards the peripheries.

4.1.4.4.5 Oolitic-Pisolotic ore

This ore type varies from a purely oolitic ore to an almost pisolitic ore or pisolitic-oolitic ore.

In El-Gedida, the oolitic ore is a brown hematitic ore of moderate to high grade (iron content ranges from 55 to 60%) and density 2.25 tonne/m$^3$. The ore is made up of small
oolites (0.5 mm average diameter) with small rhombohedral crystals in the interstices. The main gangue minerals are dispersed quartz grains with halite sometimes filling cavities. This ore type is found in the central high area associating with the banded cavernous ore in the form of pockets or interbeds of small extension.

4.1.4.4.6 Ferruginous sandstone

This low grade ore consists of angular to subangular quartz grains embedded in a matrix of iron oxides and hydroxide (mainly goethite).

4.1.4.5.1 The contact of the iron ore deposit with the underlying Bahariya Formation

1. The contact between the ore body and the underlying sandstone of the Bahariya Formation is sharp but uneven, it shows signs of gradation in places.

2. At the central high area, where the underlying bed is sandstone, a continuous barite zone is always observed just below the iron ore. It consists of small barite crystals and yellow or greenish yellow loose sands. The thickness of this zone varies from 10 to 50 cm in places.

3. A conglomerate band of silicified limestone boulders is sometimes observed either at the base of the ore body or above the base by one or two metres.
4. The last one or two metres of the ore body are always of soft, dark brown ore, except in the case where the conglomerate band or clay marks the actual lower contact. This soft ore has a slightly higher iron content than the main ore that is usually reddish or yellowish brown, much harder and compact. Although the contact between this soft ore and the sandstone is sharp, its irregular nature and the fact that the sandstone below has a similar lithological appearance as the soft ore, may indicate that these few metres of the ore represent the top of the Bahariya Formation that has undergone intense replacement.

4.1.4.5.2 The contact of the iron ore deposit with the overlying Radwan Formation

No clear relationship was observed at this contact but there is an apparent change in facies and the following signs of mineralization.

1 - The lower part of the Radwan Formation is yellow and ochrous.

2 - Round and flattened oolites were observed at the contact and show a high degree of Ferrugination.

3 - In the valleys, the overlying green, glauconitic, gypsum clays show brown stained patches and hematite pockets.
4.1.4.5.3 The contact of the iron ore deposit with the surrounding Eocene deposits

Two types of contacts were observed. The first is inter-fingered contact between the iron ore deposit and the lower middle Eocene deposit, (El-Nagb Formation), and the second is the overlapping contact between it and the other deposits (e.g. Gazzun Formation) with clear unconformity. The first relation is typically represented in the surrounding valleys to the central high area, and is revealed in numerous vertical pits. The second is observed at the northern entrance to the industrial site of El-Gedida mines on both sides of the asphaltic road; here a Ferruginous bed of reworked origin overlaps the white limestone and is separated from it by a flinty conglomerate.

4.1.4.6 Discussion on the origin of the iron ore

The origin of the iron ores of Bahariya has been the subject of discussion by a number of authors. Hum (22) and Attia (15) considered the ore as a shallow water lacustrine sediment possibly of Oligocene age. El-Shazly (23) believed that the iron ores were formed during a lagoonal period (late Eocene-early Oligocene) by slow weathering and leaching of the ferruginous sandstones in the central and southern parts of Bahariya oases. El Akkad and Issawi (18) stated that the ore was a result of shallow marine deposition partly with surface replacement of the carbonate rocks. Said and Issawi (21) assigned to the iron ores the age of lower middle Eocene and advocated the idea of a diagenetic replacement origin. In contrast to the above mentioned
views Nakhlal (24) considered the ore as of Eocene age and
or metasomatic (24a) hydrothermal, replacement origin, and also added the
possibility of partial precipitation from colloidal solutions and by cavity filling.

Amer (19) suggested iron is most probably derived from
volcanic sources. Some of his evidence in favour of such
an assumption are:

1. The presence of extensive bodies of volcanic rocks
in the Bahariya area and on the same structural
lines as the iron ores of El-Gedida, El-Harra and
of Ghorabi-Nasser.

2. The intensive replacement of the topmost part of
the Bahariya Formation sandstones into a soft iron
ore that is richer in iron than the overlying main
ore, a fact that indicates the iron solutions came
from below, concentrated in the sandstone layer
and then migrated upwards into the limestone. In
view of this evidence the assumption made by
El-Shazly (23) that the weathering iron solutions
may have infiltrated from above into the underlying
Cenomanian rocks cannot be accepted (19).

3. In the main iron ores of Bahariya, the average
contents of Ti, Cr, Co, Ni, V, P and Al were all
distinctly lower than those of sedimentary iron
ores and correspond very well with the values
observed in iron ores of volcanic origin.
The average and range of phosphorus contents have frequently been used for the distinction between genetically different iron ores, El-Hinnawi (25). In Figure 4.3 the phosphorus content in the ores of Bahariya and in other famous iron ores from various localities are given. From this Figure it is evident that the average P content in the main iron ores of Bahariya is rather low and shows limited variations. In this respect, the present ores are similar to the ores from Algeria and Tunis related to volcanic processes but are distinctly different from the Aswan, Clinton ores which are subgenetic sedimentary ores derived from weathering solutions.
Fig. 4.3
Comparison between the phosphorus content of iron ore in different locations.
4.2 Experimental Work on Bahariya Ore

Even today the mineral content of iron ores is still mainly obtained by chemical analysis. Discrimination is often made only between magnetite and hematite ores, and with less attention paid to the waste associated gangue minerals.

The evaluation of iron ores according to mineral content and in particular, according to the proportion of various gangue minerals, is not often reported on a routine basis from metallurgical plants. However, the gangue minerals occurring in nature could markedly influence the metallurgical reactions, such as softening temperatures, slag formation, and sintering behaviour. Although the above properties would be useful in determining the required additives and preliminary treatment to obtain a useful product, a more detailed knowledge of the mineralogy of the iron ores nevertheless would make it possible to evaluate the ores better.

It is desirable that information about mineralogy, textural relationships, particle sizes, elemental distributions and mineral associations of a deposit should be known as early as possible in mineral exploration. This data helps the exploration geologist to evaluate the deposit. It can often be obtained by microscopic study of the ore, but sometimes this data may be difficult or impossible to obtain. This will be clarified in the following work.

In addition to chemical analyses, the mineralogical investigation of ores was made by microscopic examination, x-ray mineralogy, electron microprobe analysis, and Stereoscan microscopy.
The material used in this study was taken from two cores which were collected from El-Gedida area (see Figure 4.2a). In the author's opinion the samples taken were representative of the Bahariya ore in the El-Gedida

4.2.1 Lithology

This iron ore bed which has a brown or yellowish brown colour consists mainly of an intergrowth of Goethite and Hematite in almost equal proportions.

The cavities and the cracks are filled with black mangani-ferrous powder. The main gangue minerals are Halite and Barite, and are present as fillings of surface cracks.

The lithology of the iron ore bed has slight variations which is shown in Table 4.1.

4.2.2 Geochemical variation in the vertical section of El-Gedida iron ore deposit (See Table 4.1)

Iron

The chemical analyses of samples collected from the core No. 1 show the maximum Fe content is 63.2% with the minimum at 49.8%. The average iron content in all sections is 59.6%. Also Figure 4.4 shows slight variations in the section.

Manganese Oxides

The maximum Mn oxide content is 1.61% in the first metre from the top, (the minimum Mn oxide content is 0.16%). The vertical distribution of Mn oxide is irregular and often exhibits sharp variations.
<table>
<thead>
<tr>
<th>No</th>
<th>Thick/cm</th>
<th>Reddish brown manganeseous hematite ores</th>
<th>Fe</th>
<th>CaO</th>
<th>MgO</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>P₂O₅</th>
<th>K₂O</th>
<th>TiO₂</th>
<th>SiO₂</th>
<th>NaCl</th>
</tr>
</thead>
<tbody>
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<td>1</td>
<td>10</td>
<td></td>
<td>40.8</td>
<td>0.7</td>
<td>1.17</td>
<td>0.50</td>
<td>0.46</td>
<td>0.57</td>
<td>0.11</td>
<td>0.06</td>
<td>1.7</td>
<td>0.55</td>
</tr>
<tr>
<td>2</td>
<td>20</td>
<td></td>
<td>54.8</td>
<td>0.4</td>
<td>1.23</td>
<td>0.38</td>
<td>0.33</td>
<td>0.34</td>
<td>0.05</td>
<td>0.03</td>
<td>1.2</td>
<td>0.19</td>
</tr>
<tr>
<td>3</td>
<td>20</td>
<td>Reddish brown manganeseous hematite ores with Barite present as yellowish white, occasional shale partings laterally discontinuous</td>
<td>59.0</td>
<td>0.5</td>
<td>1.20</td>
<td>0.35</td>
<td>0.29</td>
<td>0.36</td>
<td>0.07</td>
<td>0.06</td>
<td>1.7</td>
<td>0.26</td>
</tr>
<tr>
<td>4</td>
<td>25</td>
<td>Brown hematite ore with Barite present as yellowish white, occasional shale</td>
<td>59.2</td>
<td>0.7</td>
<td>1.49</td>
<td>0.33</td>
<td>0.15</td>
<td>0.37</td>
<td>0.04</td>
<td>0.03</td>
<td>2.9</td>
<td>2.14</td>
</tr>
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<td>10</td>
<td></td>
<td>62.8</td>
<td>0.2</td>
<td>1.61</td>
<td>0.20</td>
<td>0.17</td>
<td>0.35</td>
<td>0.03</td>
<td>0.01</td>
<td>1.0</td>
<td>4.44</td>
</tr>
<tr>
<td>6</td>
<td>25</td>
<td>Brown hematite and goethite</td>
<td>62.4</td>
<td>0.1</td>
<td>1.36</td>
<td>0.20</td>
<td>0.16</td>
<td>0.33</td>
<td>0.03</td>
<td>0.01</td>
<td>0.8</td>
<td>1.15</td>
</tr>
<tr>
<td>7</td>
<td>20</td>
<td>Brown hematite and goethite ores</td>
<td>58.6</td>
<td>0.1</td>
<td>1.44</td>
<td>&lt;0.20</td>
<td>0.15</td>
<td>0.32</td>
<td>0.02</td>
<td>0.01</td>
<td>0.7</td>
<td>0.0</td>
</tr>
<tr>
<td>8</td>
<td>20</td>
<td>Reddish brown hematite and goethite with Barite and Halite filling the cracks</td>
<td>62.6</td>
<td>0.2</td>
<td>0.92</td>
<td>0.20</td>
<td>0.12</td>
<td>0.50</td>
<td>0.03</td>
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<td>1.0</td>
<td>0.41</td>
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<tr>
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<td>Yellowish brown hematite and goethite ores</td>
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<td>0.16</td>
<td>0.45</td>
<td>0.16</td>
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<td>1.50</td>
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<td>61.0</td>
<td>0.1</td>
<td>1.06</td>
<td>0.20</td>
<td>0.08</td>
<td>0.38</td>
<td>0.14</td>
<td>0.01</td>
<td>0.7</td>
<td>6.00</td>
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<tr>
<td>11</td>
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<td>0.1</td>
<td>0.77</td>
<td>0.25</td>
<td>0.12</td>
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<td>0.47</td>
<td>0.39</td>
<td>0.14</td>
<td>0.47</td>
<td>0.02</td>
<td>0.03</td>
<td>1.2</td>
<td>2.96</td>
</tr>
<tr>
<td>13</td>
<td>25</td>
<td>Friaile yellowish brown hematite and goethite</td>
<td>92.2</td>
<td>0.4</td>
<td>0.81</td>
<td>0.41</td>
<td>0.16</td>
<td>0.48</td>
<td>0.02</td>
<td>0.03</td>
<td>1.5</td>
<td>Tra</td>
</tr>
<tr>
<td>14</td>
<td>25</td>
<td>Friaile yellowish brown hematite and goethite</td>
<td>61.4</td>
<td>0.8</td>
<td>0.79</td>
<td>0.51</td>
<td>0.19</td>
<td>0.41</td>
<td>0.02</td>
<td>0.03</td>
<td>1.6</td>
<td>Tra</td>
</tr>
<tr>
<td>15</td>
<td>20</td>
<td>Friaile yellowish brown hematite and goethite</td>
<td>58.6</td>
<td>0.2</td>
<td>1.18</td>
<td>0.30</td>
<td>0.16</td>
<td>0.57</td>
<td>0.01</td>
<td>0.04</td>
<td>1.3</td>
<td>Tra</td>
</tr>
</tbody>
</table>

PETROGRAPHY STUDIES FOR SAMPLE NO. B SHOW BANDED PISOLITIC HEMATITE-GOETHITE APPROXIMATELY (80/20) AND LITTLE HALITE (SOLUBLE GRAIN).

* THE CHEMICAL ANALYSIS WAS PERFORMED IN THE DEPT. OF METALLURGY LABS BY THE AUTHOR.
<table>
<thead>
<tr>
<th>X-RAY</th>
<th>SCANNING ELECTRON MICROSCOPY</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hematite and Goethite and little Halite</td>
<td></td>
</tr>
<tr>
<td>Hematite and Goethite</td>
<td></td>
</tr>
<tr>
<td>Hematite and Goethite and small amount of Halite</td>
<td></td>
</tr>
<tr>
<td>Hematite and Goethite</td>
<td></td>
</tr>
<tr>
<td>The ooliths show a concentric banded structure. There are anhedral grains within the concentric layers in some ooliths. A few angular grains of Fe (broken ooliths) in some areas of the matrix. Fe x-ray distribution map shows a high concentration of Fe in the ooliths. The Si x-ray shows a high concentration in two anhedral grains. The Al, P and Ca x-ray distribution are below detection limits. S x-ray distribution shows a high concentration of anhedral grains, some of which are in the ooliths and the rest in the matrix. The Cl x-ray distribution shows areas of Cl not associated with other elements.</td>
<td></td>
</tr>
</tbody>
</table>
FIG. 4.4
DISTRIBUTION OF IRON & PHOSPHORUS IN BAHARIYA IRON ORE
Sodium Chloride
The maximum NaCl content is 6.61%. Its occurrence in the iron ore is due to Halite filling surface cracks.

Silica
The silica content does not show a remarkable vertical variation in the core, where it is always low. The maximum content is 2.1% and the minimum content is 0.7, and the average content is about 1.3%.

Phosphorus
This element shows a vertical variation in the ore deposit (Figure 4.4). The maximum phosphorus content is 0.34% and the minimum is 0.14%.

Minor and trace elements
Other elements were detected in the analyses of the iron ore samples of El-Gedida but were generally present in very minor amounts, e.g. MgO (about 0.18%), CaO (about 0.3%), K₂O (about 0.04%), TiO₂ (about 0.03%).

4.2.3 Results of Microscopic Examination of Bahariya Ore
The iron ore was studied in polished thin sections. These showed pisolitic Hematite-Goethite (the ratio of Hematite to Goethite was approximately 60:40). Their average diameter was 310 μm. The section consisted of alternating thin concentric bands of Goethite with broad bands of Hematite. There were also a few angular grains of Hematite and Goethite; the length of the major axis varied from 20 μm to 120 μm.
The main gangue minerals seen were Halite which occurred as filling in the cavities.

4.2.4 X-ray Mineralogy

This was achieved by the use of mineralogical analysis on the same samples used for chemical analyses. Dry sample powders were ground using a pestle and mortar. Uniform grinding times and pressures ensured that effects due to variable grain size and surface features would be minimal. Samples were examined on a diffractometer with Fe-filtered Co radiation, or Ni-filtered Cu radiation using operating potentials of 30 kV with a current of 30 mA, and 40 kV with 20 mA respectively. Goniometer traverses from 3° to at least 60° were run at 1° 20 per minute.

Diffractograms were studied visually to identify as many mineral phases as possible. All identifications reported are based on the appearance of at least two characteristic peaks of the minerals as given in the ASTM powder diffraction index file.

Results of X-ray diffraction mineralogical analyses of six iron ore samples from El-Gedida are presented in Table 4.1. A typical X-ray diffractogram for two of the iron ore samples is shown in Figures 4.5 and 4.6. Three mineral phases were identified by X-ray techniques within the samples. Typically, two mineral phases were observed within any one sample.

Hematite was the major phase, with varying amounts of Goethite and small quantities of Halite were also identified.
Figure 4.5  Typical X-ray

Diffractogram of one of Bahariya Iron Ore Samples (Sample No. B5) (Co Kα)
Figure 4.6  Typical X-ray Diffractogram of one of Bahariya Iron Ore Samples (Sample No. B14) (CoKα)
4.2.5 Scanning Electron Microscopy (Operating Conditions p. 204)

Examination of iron ore with an ordinary petrographic microscope does not allow the study of individual apatite crystals because of their extremely fine-grained texture. However, the micron-sized crystals of sedimentary apatite are well within the range of a modern scanning electron microscope.

Burnett (7) reported that apatite crystals were present in a size range from 0.2 to 1 micron. Scanning Electron Microscopy (Stereoscan) makes possible the mapping of elemental distributions by employing electron-beam scan techniques.

From the two samples examined, one is presented here

In the scan photographs (Figure 4.7) the ooliths show a concentric banded structure. In several ooliths there are anhedral grains within the concentric layers. The average length of the major axis for the ooliths is about 310 μm. A few angular grains of Fe (broken ooliths) are seen in some areas of the matrix; the length of the major axis varies from 20 μm to 120 μm. The Fe X-ray distribution map shows a high concentration of Fe in the ooliths.

The Si distribution shows a high concentration in two anhedral grains not associated with other elements; the length of the major axis is about 30 μm. Si is also seen disseminated throughout the micrographs in very low concentration. The Al, P and Ca X-ray distribution are below detection limits.
Figure 4.7  Scanning Electron
Micrograph of Bahariya Iron
Ore Sample No. 8 and X-ray
Distribution Maps x100

a - Scanning electron micrograph
b - Iron X-ray distribution map
c - Silicon X-ray distribution map
d - Aluminium X-ray distribution map
e - Phosphorus X-ray distribution map
f - Calcium X-ray distribution map
g - Sulphur X-ray distribution map
h - Chlorine X-ray distribution map
Fig. 4.7
Fig. 4.7
S distribution shows a high concentration of anhedral grains, some of which are in the ooliths within the concentric layers and the rest in the matrix (see Figure 4.7); the length of the major axis varies from 20 μm to 40 μm.

The Cl distribution shows a moderately high concentration area (bottom left of micrograph) not associated with other elements.

Fe in the ooliths is shown from petrographic studies to be as iron oxides. Si appears in two anhedral grains and is shown from petrographic studies to be quartz.

S is shown as anhedral grains not associated with the other elements it is shown from hand specimen identification to be Barite. Cl occurs as a high concentration area which was shown from X-ray studies and petrographic studies to be dissolved grain of Halite.

4.2.6 Summary of Results
The iron ore deposits considered in this thesis occur at four localities: Nasser area, Gebel Ghorabi, El-Harra and El-Gedida. The ore at Bahariya is dark brown to dark red colour and consists mainly of hematite and goethite with pockets of ochre and manganese oxides. The gangue minerals are Halite, Gypsum, Barite, Quartz and Calcite.

El-Gedida area contains the biggest and richest iron ore deposit of Bahariya. The ore is classified into six types according to the mineralogical composition and the properties of the hand specimen: Hard massive ore, Banded cavernous ore, Friable ore, Black manganiferrous ore, Oolitic-Pisolitic ore.
and Ferruginous sandstone. From chemical analysis (Table 4.1) the Fe content is between 49.8-63.2% and P is between 0.14-0.34%. From X-ray examination Hematite was the major phase with varying amounts of Goethite and small quantities of Halite.

Electron microprobe analysis and scanning electron microscope studies proved that there are small apatite fine grains disseminated throughout the ooliths.
4.3 Aswan Iron Ore

4.3.1 Topography

The area in which the iron ore deposits occur lies between latitudes 24° 03' and 24° 14' North and longitudes 32° 52' and 33° 22' East.

Its western boundary is the Nile Valley; to the north it is bounded partly by Wadi Subeira and partly by Wadi Allawi, to the east by Wadi Allawi; and to the south partly by Wadi Abu Agag. It extends approximately 50 kilometres from east to west and 20 kilometres from north to south; the area is, therefore, roughly 1,000 square kilometres. The iron ore deposits do not cover the entire region but are found scattered within it.

The area is a plateau varying in level from about 150 metres above sea level at the Nile Valley side to about 350 metres above sea level on the eastern side; the highest peak in the area is Gebel Dheisa, in the south-eastern portion, which is 418 metres above sea level. The Nile valley level in the Aswan district is roughly 100 metres above sea level. The depths of the major wadis of the geographical boundaries vary from 50 to 100 metres.

4.3.2 The Nubian sandstone series

The Nubian sandstone covers more than two-thirds of the district. The strata forming this series includes layers of conglomerates, pebble bands, grits, quartzites, sandstones, clayey sandstones, sandy clays, clays or shales and in places valuable beds of oolitic iron ores. These
layers overlie ancient (Precambrian) igneous (granite, etc.) and metamorphic (gneisses but mainly schists) rocks (25).

In the district, the total maximum thickness of the groups forming the Nubian sandstone series probably exceeds 150 metres. The complete succession of the strata is to be found in the cliffs bordering the Nile Valley and there it attains a thickness of about 100 metres (see next section).

4.3.2.1 Measured Geological sections of the entire Nubian Sandstone series

Vertical sections have been measured by Attia (27). (His section was taken at a point below the word "Inscription" (see map, Fig. 4.8) on the cliff bordering the Nile Valley).

It gave the following succession of strata from top to bottom.

<table>
<thead>
<tr>
<th>Top</th>
<th>Thickness in metres</th>
</tr>
</thead>
<tbody>
<tr>
<td>Upper</td>
<td></td>
</tr>
<tr>
<td>10 Medium to coarse-grained sandstones getting hard and quartzitic near top</td>
<td>35.0</td>
</tr>
<tr>
<td>9 Pinkish sandstone</td>
<td>8.0</td>
</tr>
<tr>
<td>8 Refractory clays</td>
<td>4.0</td>
</tr>
<tr>
<td>7 Ferruginous Sandstone</td>
<td>1.5</td>
</tr>
<tr>
<td>6 Red, oolitic iron ore (lower Horizon)</td>
<td>2.5</td>
</tr>
<tr>
<td>5 Ferruginous sandstone</td>
<td>6.0</td>
</tr>
<tr>
<td>4 Hard, grey sandstone</td>
<td>7.0</td>
</tr>
<tr>
<td>3 Variegated, sandy clays</td>
<td>10.5</td>
</tr>
<tr>
<td>Lower</td>
<td></td>
</tr>
<tr>
<td>2 Medium to coarse grained sandstone, clayey towards the middle</td>
<td>13.5</td>
</tr>
<tr>
<td>1 Kaolinaceous sandstone, peddly at base</td>
<td>4.0</td>
</tr>
<tr>
<td>- Kaolinised, pink granite</td>
<td>-</td>
</tr>
</tbody>
</table>

Bottom

Total thickness of Nubian series: 92.0
FIG. 4.8
GEOLOGICAL MAP SHOWING DESERT EAST OF ASWAN

LEGEND:
- Recent & Pleistocene
- Upper Group
- Middle Group (with Oolitic Iron-Ore Bands)
- Lower Group
- Granite
- Metamorphic

The Nubian Sandstone Series

Scale: 1:180,000
4.3.3 Geological Classification of iron ore deposits in the District

The different forms of iron ore deposits in the district are as follows:

1 - The Ferruginous sandstones
2 - The ferruginous concretions
3 - The Oolitic iron ore

4.3.3.1 The Ferruginous sandstone

The Ferruginous sandstones are reddish, brownish or dark-grey, fine to medium grained rocks; they may be hard and compact or soft and friable. In some localities, the Ferruginous sandstones are clayey or intercalated by thin bands of Ferruginous clay.

Ferruginous sandstones are associated with the oolitic iron ore; they are generally encountered underlying or overlying the oolitic iron ore but sometimes they are seen intervening two bands of the oolitic ore. In many localities, the Ferruginous sandstones cap the oolitic iron ore and these act as a protective cover and save the iron ore from being eroded.

Ferruginous sandstone may contain a little amorphous or even oolitic hematite and in several occurrences, transitions from Ferruginous sandstones to oolitic iron ore are found.

Several analyses have been made of different samples of these Ferruginous sandstones reported by Attia (27). The range of iron, silica, phosphorus and sulphur is tabulated as follows:
The table shows the wide range of iron, silica, and phosphorus contents and the presence of sulphur sometimes. As a whole, the iron content of the Ferruginous sandstone is low and the silica content high.

4.3.3.2 The Ferruginous Concretions

Ferruginous concretions are mainly found in sandstones of the Middle Group of the Nubian sandstone series. The concretions are dark-brown or almost black in colour and occur either in the form of spherical nodules ranging from half a centimetre to five centimetres in diameter or as elongated rods or various sizes. They are hard and have a dense usually radiating structure.

Due to the denudation of the sandstone containing these concretions and owing to their resistance to weathering, they are often seen accumulating in places in large quantities, at ground level giving it a black appearance.

Several analyses have been made of different samples of the Ferruginous concretions by Attia (27). The average percent of the iron, silica, phosphorus are given as follows:

\[
\begin{align*}
\text{Fe}_2\text{O}_3 & \quad 55\% \\
\text{SiO}_2 & \quad 32\% \\
\text{P}_2\text{O}_5 & \quad 0.15\%
\end{align*}
\]
4.3.3.3 The Oolitic iron ore

The oolitic iron ore is the principal and most valuable ore in the district.

It is a compact, dark red, oolitic hematite. In hand specimens, it is made up of spheroidal or ellipsoidal grains of red hematite. The grains are easily seen by the naked eye, they vary in size in different specimens and even in the same specimen. In some, they range between 0.3 and 1.2 millimetre in diameter but mainly 0.7 millimetre diameter. In others, they range between 0.4 and 1.6 millimetre in diameter but chiefly 1.2 millimetre diameter. The material cementing the oolitic grains is a fine, hematitic substance. Attia's (27) work showed that in the Nubian sandstone series, the oolitic iron ore deposits were confined to the Middle Group of the series and that this Group began at the base with the appearance of an oolitic iron ore band of a lower horizon (A) followed by a layer of sandstone with ripple-marks. Then came the strata of refractory clays or their equivalents; these were followed by one or more oolitic iron ore bands of the upper horizon (B) generally separated by Ferruginous sandstone. The band ended with a few layers of various sandstones.

Thickness of the oolitic hematite band (A) varies from 15 to 80 centimetres and the average thickness ranged between 32 and 40 centimetres.
The upper Horizon (B) is the most important one; it usually contains two and sometimes three bands of oolitic hematite separated by sandstone or clay. The oolitic bands are generally accompanied by Ferruginous sandstones.

The extent of the oolitic hematite bands of the Upper Horizon (B) is much more than that of the lower Horizon (A). The total thickness of the oolitic hematite of the Upper Horizon varies from 10 to 350 centimetres; its average thickness in the different areas ranges between 28 and 148 centimetres. Where the oolitic iron ores of both Horizons are present, they are separated from each other by layers, usually of sandstones and clays. In some localities these layers are mainly of sandstones alone with a total thickness varying from 4 to 17 metres (averaging 7.8 metres).

The chemical composition of the ore varies from place to place but the main constituents are Fe₂O₃ and SiO₂. Analyses of a number of samples from the different localities indicate that the content of iron oxide in the ore varies between 54.8 and 88.1 percent, with an average 71.5 percent (27).

A large number of sections of the oolitic iron ore has been taken at several exposures in various localities of the area. Below is given an example for a section from Gebel Timarch area (33° 0'5" 24° 1'0") (27).
Overburden - mainly sandstone 0.2 to 8.00
Ferruginous sandstone with oolites 0.70
Oolitic iron ore (B₂) 1.20
Ferruginous sandstone 0.30
Oolitic iron ore (B₁) 0.80
Clayey sandstone 0.90
Multi-coloured clays with Ferruginous sandstone band 6.50
Sandstone with ripple-marks 1.50
Whitish sandstone 0.90
Greenish clays 0.40
Ferruginous sandstone with oolites 0.20
Oolitic iron ore (A) 0.50

Overall thickness 14.10 to 21.90

4.3.4 Experimental work
Investigations were carried out on samples collected from two areas in the Aswan Field between longitude 33° 6' and 32° 58' and latitude 24° 6' and 24° 9'. The lithology and chemical analyses throughout the two vertical sections were studied and are presented in Tables 4.2 and 4.3. It is the author's opinion that the samples were representative of two types of Aswan ore. In addition to the chemical analysis and lithology a mineralogical investigation of the ores was made by microscopic examination, X-ray mineralogy, microprobe analysis, and stereoscan microscopy.
<table>
<thead>
<tr>
<th>No.</th>
<th>Depth from</th>
<th>Lithology</th>
<th>SiO₂ %</th>
<th>CaO %</th>
<th>Fe %</th>
<th>Al₂O₃ %</th>
<th>FL</th>
<th>Petrography</th>
<th>X-ray</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>300</td>
<td>Overgrown clayy</td>
<td>53.30</td>
<td>2.13</td>
<td>1.0</td>
<td>38.83</td>
<td>0.04</td>
<td>Most of the sample is SiO₂, small gibbsite, small chlorite and trace hematite.</td>
<td>Majorily is hematite and small SiO₂, trace of chlorite.</td>
</tr>
<tr>
<td>2</td>
<td>25</td>
<td>White coarse grained sandstone</td>
<td>39.66</td>
<td>3.62</td>
<td>20.24</td>
<td>10.65</td>
<td>0.66</td>
<td>Majority is hematite and small SiO₂.</td>
<td>Majority is hematite, little SiO₂ and trace chlorite.</td>
</tr>
<tr>
<td>3</td>
<td>15</td>
<td>Hard ferruginous sandy clay</td>
<td>4.37</td>
<td>2.09</td>
<td>55.12</td>
<td>16.40</td>
<td>0.42</td>
<td>Hematiteoliths, angular quartz grains are cemented by fine hematite.</td>
<td>Majority is hematite, little SiO₂ and trace chlorite.</td>
</tr>
<tr>
<td>4</td>
<td>16</td>
<td>Dolomite iron ore</td>
<td>7.05</td>
<td>3.47</td>
<td>49.11</td>
<td>10.75</td>
<td>1.74</td>
<td>Hematiteoliths, angular quartz grains are set in fine hematite matrix.</td>
<td>Majority is hematite, small SiO₂ and apatite.</td>
</tr>
<tr>
<td>5</td>
<td>17</td>
<td>Dolomite iron ore</td>
<td>9.28</td>
<td>5.36</td>
<td>47.01</td>
<td>12.21</td>
<td>2.02</td>
<td>Hematiteoliths, matrix is fine hematite with angular quartz grains</td>
<td>Majority is hematite, small SiO₂ and apatite.</td>
</tr>
<tr>
<td>6</td>
<td>18</td>
<td>Dolomite iron ore</td>
<td>12.49</td>
<td>3.54</td>
<td>45.33</td>
<td>12.91</td>
<td>1.95</td>
<td>Hematiteoliths, matrix is fine hematite with angular quartz grains</td>
<td>Majority is hematite, small SiO₂ and apatite.</td>
</tr>
<tr>
<td>7</td>
<td>19</td>
<td>Red brown ferruginous sandy clay</td>
<td>59.05</td>
<td>4.23</td>
<td>9.77</td>
<td>15.54</td>
<td>1.46</td>
<td>Angular quartz grains set in fine quartz and hematite matrix.</td>
<td>Majority is hematite, small SiO₂ and little hematite, trace chlorite.</td>
</tr>
<tr>
<td>8</td>
<td>20</td>
<td>White sandstone</td>
<td>65.01</td>
<td>0.77</td>
<td>8.47</td>
<td>14.29</td>
<td>0.05</td>
<td>Majority is hematite, small SiO₂, little apatite, trace of clay.</td>
<td>Majority is SiO₂, trace of clay.</td>
</tr>
<tr>
<td>9</td>
<td>21</td>
<td>Fine-medium sandstone with thin layers rich in white clay mineral</td>
<td>69.16</td>
<td>Tr.</td>
<td>6.97</td>
<td>17.17</td>
<td>0.05</td>
<td>Majority is hematite, small SiO₂.</td>
<td>Majority is SiO₂, trace of clay.</td>
</tr>
<tr>
<td>10</td>
<td>15</td>
<td>Fine-medium sandstone with thin layers rich in white clay mineral</td>
<td>75.00</td>
<td>Tr.</td>
<td>1.10</td>
<td>21.33</td>
<td>0.05</td>
<td>Majority is hematite, small SiO₂.</td>
<td>Majority is SiO₂, trace of clay.</td>
</tr>
<tr>
<td>11</td>
<td>22</td>
<td>Dolomite iron ore</td>
<td>61.10</td>
<td>0.98</td>
<td>8.12</td>
<td>19.89</td>
<td>0.14</td>
<td>Angular quartz grains cemented by very fine material</td>
<td>Majority is hematite, trace SiO₂.</td>
</tr>
<tr>
<td>12</td>
<td>23</td>
<td>Dolomite iron ore</td>
<td>8.46</td>
<td>1.78</td>
<td>51.62</td>
<td>5.90</td>
<td>0.52</td>
<td>Angular quartz grains cemented by very fine material</td>
<td>Majority is hematite, trace SiO₂.</td>
</tr>
<tr>
<td>13</td>
<td>24</td>
<td>Dolomite iron ore</td>
<td>2.38</td>
<td>4.11</td>
<td>53.32</td>
<td>18.44</td>
<td>0.95</td>
<td>Majority is hematite, trace SiO₂.</td>
<td>Majority is hematite, trace SiO₂.</td>
</tr>
<tr>
<td>14</td>
<td>25</td>
<td>Dolomite iron ore</td>
<td>2.91</td>
<td>4.05</td>
<td>50.85</td>
<td>8.75</td>
<td>2.91</td>
<td>Hematiteoliths, cemented by fine grains of apatite</td>
<td>Majority is hematite, trace SiO₂.</td>
</tr>
<tr>
<td>15</td>
<td>26</td>
<td>Dolomite iron ore</td>
<td>5.19</td>
<td>2.53</td>
<td>53.95</td>
<td>8.14</td>
<td>0.94</td>
<td>Majority is hematite, trace SiO₂.</td>
<td>Majority is hematite, trace SiO₂.</td>
</tr>
<tr>
<td>16</td>
<td>27</td>
<td>Dolomite iron ore</td>
<td>14.11</td>
<td>0.24</td>
<td>42.87</td>
<td>12.74</td>
<td>0.96</td>
<td>Majority is hematite, trace SiO₂.</td>
<td>Majority is hematite, trace SiO₂.</td>
</tr>
<tr>
<td>17</td>
<td>28</td>
<td>Dolomite iron ore</td>
<td>16.28</td>
<td>1.62</td>
<td>44.68</td>
<td>10.24</td>
<td>0.12</td>
<td>Majority is hematite, trace SiO₂.</td>
<td>Majority is hematite, trace SiO₂.</td>
</tr>
<tr>
<td>18</td>
<td>29</td>
<td>Dolomite iron ore</td>
<td>24.7</td>
<td>0.80</td>
<td>27.77</td>
<td>11.20</td>
<td>0.10</td>
<td>Dolomite of hematite, angular quartz grains are set in the matrix of very fine hematite grains.</td>
<td>Majority is hematite, trace SiO₂.</td>
</tr>
</tbody>
</table>

*THE CHEMICAL ANALYSIS WAS PERFORMED IN THE DEPT. OF METALLURGY LABS BY THE AUTHOR*
<table>
<thead>
<tr>
<th>No.</th>
<th>Depth/cm</th>
<th>Scanning Electron Microscope</th>
<th>Electron Microprobe Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Most of the sample is Si associated with Fe, angular and subangular grains of Ca.</td>
</tr>
<tr>
<td>As 1</td>
<td>1200</td>
<td></td>
<td>Most of the sample is Fe, angular Si grains, anhedral Si grains, and angular P grains associated with Ca.</td>
</tr>
<tr>
<td>As 2</td>
<td>25</td>
<td></td>
<td>Subangular P grains associated with Ca, and angular Si grains are cemented by fine particles of Fe associated with little Al.</td>
</tr>
<tr>
<td>As 3</td>
<td>10</td>
<td></td>
<td>Ooliths of Fe, angular Si grains, angular and subangular P grains surrounding the ooliths and the Si grains.</td>
</tr>
<tr>
<td>As 4</td>
<td>5</td>
<td></td>
<td>Ooliths of Fe associated with Al and Si.</td>
</tr>
<tr>
<td>As 5</td>
<td>16</td>
<td></td>
<td>Ooliths of Fe, angular Si grains associated with Ca and Al.</td>
</tr>
<tr>
<td>As 6</td>
<td>16</td>
<td></td>
<td>Ooliths associated with Si and Fe, cemented by Al associated with Si and little Fe. P occurs as very little fine particles.</td>
</tr>
<tr>
<td>As 7</td>
<td>20</td>
<td></td>
<td>Ooliths of Fe associated with Al and Si, anhedral grains of P associated with Ca occurs in the ooliths.</td>
</tr>
<tr>
<td>As 8</td>
<td></td>
<td></td>
<td>Ooliths of Fe associated with Si and Al, matrix is P associated with Ca.</td>
</tr>
<tr>
<td>As 9</td>
<td>15</td>
<td></td>
<td>Ooliths, matrix is mainly P associated with Ca, anhedral Si grains associated with Al.</td>
</tr>
<tr>
<td>As 10</td>
<td>5</td>
<td></td>
<td>Fe ooliths, matrix is mainly P associated with Ca, anhedral Si grains associated with Al.</td>
</tr>
<tr>
<td>As 11</td>
<td>15</td>
<td></td>
<td>Fe ooliths, anhedral P grains associated with Ca in the ooliths, subrounded and angular Si grains, matrix is fine Fe associated with Al and Si.</td>
</tr>
</tbody>
</table>
### Table 4.3: Lithology, Chemical Analysis, and X-Ray Studies for Ashan Iron Ore Core 2

<table>
<thead>
<tr>
<th>No.</th>
<th>Depth (cm)</th>
<th>Lithology</th>
<th>Fe%</th>
<th>CaO%</th>
<th>Mg%</th>
<th>Al₂O₃</th>
<th>SiO₂</th>
<th>X-Ray</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aa11</td>
<td></td>
<td>Overburden - sandstone and clays. Feruginous sandstone</td>
<td>2.84</td>
<td>1.00</td>
<td>41.16</td>
<td>8.58</td>
<td>8.9</td>
<td>Majority is hematite, small SiO₂, little apatite.</td>
</tr>
<tr>
<td>Aa12</td>
<td>11</td>
<td>Poor feruginous sandstone, pink sandstone occurs immediately above ironstone, zone above 6.1 cm.</td>
<td>0.65</td>
<td>2.80</td>
<td>42.44</td>
<td>8.91</td>
<td>9.69</td>
<td>Majority is hematite, trace of calcite and hematite and little apatite.</td>
</tr>
<tr>
<td>Aa13</td>
<td></td>
<td></td>
<td>1.21</td>
<td>4.50</td>
<td>41.21</td>
<td>20.88</td>
<td>6.69</td>
<td>Majority is hematite, trace of chlorite and little apatite.</td>
</tr>
<tr>
<td>Aa14</td>
<td>44</td>
<td>Oolitic iron-ore, metallic grey cobble, 0.3-1 mm across in fine red matrix</td>
<td>0.35</td>
<td>1.77</td>
<td>52.78</td>
<td>7.19</td>
<td>6.68</td>
<td>Most of sample is hematite, small SiO₂.</td>
</tr>
<tr>
<td>Aa15</td>
<td></td>
<td></td>
<td>0.66</td>
<td>1.97</td>
<td>50.92</td>
<td>7.12</td>
<td>6.16</td>
<td>Majority is hematite, little SiO₂ and apatite.</td>
</tr>
<tr>
<td>Aa16</td>
<td></td>
<td></td>
<td>1.38</td>
<td>5.18</td>
<td>50.89</td>
<td>9.05</td>
<td>4.93</td>
<td>Majority is hematite, little SiO₂ and apatite.</td>
</tr>
<tr>
<td>Aa17</td>
<td></td>
<td></td>
<td>2.16</td>
<td>3.92</td>
<td>51.60</td>
<td>7.47</td>
<td>5.52</td>
<td>Majority is hematite, little SiO₂ and apatite.</td>
</tr>
<tr>
<td>Aa18</td>
<td>11</td>
<td>Poorly laminated, reds feruginous mudrock</td>
<td>0.49</td>
<td>15.88</td>
<td>53.20</td>
<td>5.95</td>
<td>5.20</td>
<td>Majority is hematite, little SiO₂, trace of chlorite and calcite.</td>
</tr>
<tr>
<td>Aa19</td>
<td></td>
<td></td>
<td>1.11</td>
<td>2.87</td>
<td>41.83</td>
<td>7.56</td>
<td>12.03</td>
<td>Majority is hematite, little SiO₂, trace of chlorite and calcite.</td>
</tr>
<tr>
<td>Aa20</td>
<td></td>
<td></td>
<td>1.14</td>
<td>3.41</td>
<td>41.11</td>
<td>5.68</td>
<td>15.65</td>
<td>Majority is hematite, little SiO₂, little apatite and trace of chlorite.</td>
</tr>
<tr>
<td>Aa21</td>
<td></td>
<td></td>
<td>1.37</td>
<td>2.87</td>
<td>48.63</td>
<td>6.32</td>
<td>10.55</td>
<td>Majority is hematite, little SiO₂, little apatite, trace of chlorite.</td>
</tr>
<tr>
<td>Aa22</td>
<td></td>
<td></td>
<td>0.47</td>
<td>3.15</td>
<td>57.34</td>
<td>6.60</td>
<td>4.37</td>
<td>Majority is hematite, little SiO₂, trace of chlorite and calcite.</td>
</tr>
<tr>
<td>Aa23</td>
<td></td>
<td></td>
<td>0.90</td>
<td>5.15</td>
<td>56.55</td>
<td>7.15</td>
<td>3.36</td>
<td>Majority is hematite, little SiO₂, trace of chlorite and calcite.</td>
</tr>
<tr>
<td>Aa24</td>
<td></td>
<td></td>
<td>1.25</td>
<td>2.37</td>
<td>55.90</td>
<td>8.56</td>
<td>3.76</td>
<td>Majority is hematite, little SiO₂, trace of chlorite and calcite.</td>
</tr>
<tr>
<td>Aa25</td>
<td></td>
<td></td>
<td>0.97</td>
<td>2.35</td>
<td>56.63</td>
<td>7.64</td>
<td>4.35</td>
<td>Majority is hematite, little SiO₂, trace of chlorite and calcite.</td>
</tr>
<tr>
<td>Aa26</td>
<td>180</td>
<td>Oolitic iron ore, metallic grey cobble, 0.3-1 mm across in fine red matrix</td>
<td>1.09</td>
<td>1.65</td>
<td>56.84</td>
<td>4.60</td>
<td>5.86</td>
<td>Majority is hematite, little SiO₂, trace of chlorite and calcite.</td>
</tr>
<tr>
<td>Aa27</td>
<td></td>
<td></td>
<td>2.30</td>
<td>4.70</td>
<td>49.54</td>
<td>14.75</td>
<td>5.24</td>
<td>Majority is hematite, little SiO₂, trace of chlorite and calcite.</td>
</tr>
</tbody>
</table>

*The chemical analysis was performed in the Department of Metallurgy Labs by the Author.*
4.3.4.1 Lithology

The oolitic iron ore is a compact-dark-red hematite. In hand specimen it shows spheroidal or ellipsoidal grains of red hematite which are easily seen by the naked eye and whose average diameter is approximately 400 μm. The material cementing the ooliths is a fine hematitic material. The sandstones are reddish, brownish or white, fine to medium grained rocks which are hard and compact. (See Table 4.2).

4.3.4.2 Geochemical variation in the vertical sections of Aswan iron ore deposits (Tables 4.2 and 4.3)

Location I

In the oolitic iron ore

Iron: Chemical analyses show that the maximum iron content is 55.1% and the minimum is 42%. It is clear that the iron content shows a slight variation within the iron ore bed (Fig. 4.9).

Silica: The maximum silica content is 16.2% and the minimum is 2.3%. The vertical distribution of silica is irregular and often exhibits sharp variations.

Alumina: The maximum alumina is 18.4% and the minimum is 8.1%. The distribution is irregular and occasionally exhibits sharp changes in content.

Phosphorus: This distribution shows a vertical variation throughout the oolitic iron bed with a maximum phosphorus content of 2.9% and a minimum of 0.1% (Fig. 4.9).
FIG. 4.9
DISTRIBUTION OF IRON & PHOSPHORUS CONTENT IN ASWAN IRON ORE
Lime: The vertical distribution of lime is irregular and often exhibits sharp variations. The maximum CaO content is 6.3% and the minimum is 0.2%.

The sandstone bed
The maximum iron content is 8.1% and the minimum is 1%. The maximum SiO₂ content is 75.9% and the minimum is 53.3%, most of the samples which are high in SiO₂ are also high in Al₂O₃ content.

Lime varies from 2.3% to a trace; Alumina 38.8-19.8% and P 0.14-0.04%.

In the Ferruginous sandy clays

<table>
<thead>
<tr>
<th>Element</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iron</td>
<td>20.2 - 6.5%</td>
</tr>
<tr>
<td>SiO₂</td>
<td>69.1 - 39%</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>18.6 - 14.2%</td>
</tr>
<tr>
<td>P</td>
<td>1.4 - 0.5%</td>
</tr>
<tr>
<td>CaO</td>
<td>4.2 - trace</td>
</tr>
</tbody>
</table>

Location II
In the oolitic iron ore

Iron: 57.3 to 41.1%

The distribution of iron is irregular and slightly variable (see Fig. 4.10).

Silica: 15.6 to 3.3%

Its distribution is irregular and often exhibits sharp variations.
FIG. 4.10
DISTRIBUTION OF IRON & PHOSPHORUS CONTENT IN ASWAN IRON ORE DEPOSIT
Al₂O₃: 20.8 to 4.6%

Phosphorus: 2.3 - 0.35%

The distribution of phosphorus is irregular and occasionally exhibits a sharp variation.

CaO: 15.8 - 1.6%

The distribution of CaO is irregular and often shows sharp variation.

In ferruginous sandstone

Iron content 44.1 - 41.8%
SiO₂ content 12 - 8.9%
Al₂O₃ content 8.5 - 7.5%
Phosphorus content 3.8 - 1.1%
CaO content 2.8 - 1.0%

4.3.4.3 Results of X-ray diffraction mineralogical analyses are presented in Table 4.2

A typical X-ray diffractogram for two of the iron ore samples is shown in figures 4.11 and 4.12. From 15 samples examined, 4 minerals were identified within the iron ore samples - hematite, quartz, clay minerals and apatite.

Typically, two mineral phases were observed within any one sample of which hematite was the major phase especially in the oolithic iron ore sample, with varying amounts of quartz.

In the sandstone sample, quartz was the major phase with varying amounts of hematite and apatite.
Figure 4.11  Typical X-ray Diffractogram of One of Aswan Iron Ore Samples (Sample No. As 7) (Co Kα)
Small quantities of clay minerals were identified in some samples (characteristic peak at $7.17 \, \AA$ d space.)

4.3.4.4 Petrography

In oolitic iron ore thin sections, the ooliths are ellipsoidal in shape, and they display a concentric banding structure mostly, the average length of the major axis is 400 μm. The matrix may be a mixture of fine grains of hematite and clay minerals both of which cement the ooliths and angular quartz grains; or it may consist of fine grain hematite and partially agglomerated apatite crystals, both of which cement the ooliths.

The sandstone thin sections are mainly angular quartz crystals and little of muscovite, and very minor amounts of feldspar. Matrix is a mixture of fine grains of hematite and quartz with a little clay mineral (which is difficult to identify).

4.3.4.5 Scanning Electron Microscopy & EPMA

Examination of Aswan iron ore by stereoscan made possible the mapping of elemental distributions for Fe, Si, P, Ca, Al and S by employing electron beam scan techniques. This was done for fifteen samples from different beds throughout the body of the ore. The technique showed the structure and grain size of the iron oxides and the gangue minerals as may be seen in the data given below.
Figure 4.13 - Scanning electron micrographs of oolitic iron ore sample No. AS10. Fe, Si, Al, P and Ca. X-ray distribution map.

Ooliths are ellipsoidal and circular in shape; the length of the major axis varies from 75 μm to 250 μm. Many ooliths show a concentric banding structure but in a number of cases this cannot be seen. In several ooliths there is the nucleus of another mineral grain which subsequently appears as a subangular grain within the concentric layers. For the angular and anhedral grains, the length of the major axis varied from 25 μm to 80 μm. The matrix cements the ooliths and is Fe rich.

The Fe X-ray distribution map shows high concentrations of Fe in the ooliths and the matrix with the higher contents in the ooliths. It is shown also that Fe concentration is independent of the concentration of Si, Al, Ca or P. The Si occurs primarily as discrete areas of high concentrations and is not associated with the other elements.

The areas of high Si are associated with the angular detrital fragment seen in the scanning electron micrograph. However, less abundantly rounded areas (75 μm) of moderately high Si concentration are associated with moderately high Al concentration.

Ca and P are usually associated with each other, the discrete areas ranging in size from 20 μm to 100 μm.
Figure 4.13  Scanning Electron Micrographs of Oolitic Iron Ore
Sample No. As 10 and X-ray Distribution Maps x200

a - Scanning Electron Micrograph
b - Fe X-ray distribution map
c - Si X-ray distribution map
d - Al X-ray distribution map
e - P X-ray distribution map
f - Ca X-ray distribution map
Fig. 4.13
Fe appears in
a - as the matrix solely Fe rich which is shown from X-ray studies and petrographic studies to be hematite.

b - ooliths as only Fe, again shown from X-ray studies and petrographic studies as hematite.

Si shows
a - as discrete angular grains. From X-ray studies and petrographic studies these proved to be quartz.

b - associated with Al; proved from X-ray studies to be clay minerals.

Ca and P occur as discrete grains in the ooliths and in the matrix, but also locally cementing quartz grains, (bottom left hand corner) from X-ray studies these proved to be apatite.

Figure 4.14 - Scanning electron micrographs of oolitic iron ore sample No. AS9. Fe, Si, Al, P and Ca X-ray distribution maps

Ooliths are ellipsoidal in shape, and display a concentric banded structure, rounded grains are seen within the concentric layers.

Angular and subrounded grains; the length of the major axis varies from 25 μm to 150 μm.

The matrix cements the ooliths and also the detrital grains. Also there are a few hexagonal grains within the matrix.

-78-
Figure 4.14  Scanning Electron Micrographs of Oolitic Iron Ore
Sample No. As 9 and X-ray Distribution Maps x200

a - Scanning Electron Micrograph
b - Fe X-ray distribution map
c - Si X-ray distribution map
d - Al X-ray distribution map
e - P X-ray distribution map
f - Ca X-ray distribution map
The Fe X-ray distribution map shows high concentration of Fe in the ooliths and matrix. It shows that Fe concentration is independent of the concentrations of Si, Al, Ca or P.

The Si occurs primarily as discrete areas of high concentration associated with very small amounts of Al. The areas of high Si concentration are associated with the angular and sub-rounded detrital fragments seen in the scanning electron micrograph. However, less abundantly there exist rounded areas (40 μm) of low Si concentration associated with low Al concentration.

Both the Ca and P concentration are related to each other, the discrete areas ranging in size from 25 μm to 65 μm.

Fe shows in:
- matrix dominantly Fe (oxides)
- the ooliths are predominantly Fe (as oxides)

Si shows:
- as discrete angular and sub-rounded grains associated with a little amount of Al (mainly quartz associated with clays).
- discrete rounded grains low in concentration associated with low concentration Al (from X-ray studies proved to be clay minerals).

Ca and P occur as apatite which can be seen as discrete round grains in the ooliths, and very anhedral hexagonal grains in the matrix, suggest authigenic growth. Apatite
crystals ranging in size from 20 to 60 μm make up aggregates of 150 μm size.

Figure 4.15 - Scanning electron micrograph of Ferruginous sand clay sample No. As12-Z, and the X-ray distribution maps for Fe, Si, Al, P and Ca. Prismatic and subangular grains, the length of the major axis varies from 25 μm to 90 μm.

The matrix cements these grains, but there are two types of matrix materials.

The Fe X-ray distribution map shows high concentrations of Fe in the matrix. It also shows that the concentration of Fe is independent of the concentration of Si, Al, Ca or P.

The Si occurs primarily as discrete areas of high concentration and is not associated with other elements.

The areas of high Si concentration are associated with the prismatic and subangular detrital fragments seen in the scanning electron micrograph.

Al in the matrix is associated with Fe and Si.

Ca and P are usually associated with each other, the discrete anhedral grains ranging in size from 5 μm to 50 μm.

The Fe in the matrix is associated with Al and Si. X-ray studies indicate this to be chamosite.
Figure 4.15  Scanning Electron Micrograph of Ferruginous Sand Clay Sample No. As 12-2, and X-ray Distribution Maps x200

a - Scanning Electron Micrograph
b - Iron X-ray distribution map
c - Silicon X-ray distribution map
d - Aluminium X-ray distribution map
e - Phosphorus X-ray distribution map
f - Calcium X-ray distribution map
Fig. 4.15
Si shows:

a - as discrete prismatic and subangular grains which are shown from X-ray and petrographic studies to be quartz.

b - associated with Al and Fe; as previously this proved to be chamosite.

Ca and P occur as discrete anhedral grains in the matrix. They also exist in areas where the anhedral grains agglomerate to form aggregates, the length of the major axis of which is 80 μm.

Figure 4.16 - Scanning electron micrographs of Ferruginous sand clay sample No. AS12, and the X-ray distribution maps for Fe, Si, Al, P and Ca.

Angular and subangular grains; the length of the major axis varies from 6 μm to 80 μm.

The matrix cements the angular and subangular grains; but there are two types of matrix material, one is lighter coloured than the other.

The Fe X-ray distribution map shows a high concentration of Fe in the matrix. It also shows that the Fe concentration is independent of the concentration of Si, Al, Ca or P, although Si and Al are associated with Fe. The Si occurs primarily as discrete areas of high concentration and is not associated with other elements. The areas of high Si concentration are associated with the subangular and angular detrital fragments seen in the scanning electron micrograph.
Ca and P are usually associated with each other, the discrete subhedral and anhedral grains ranging in size of the major axis from 4 μm to 24 μm. Fe exists in the matrix. It is associated with Al and Si. From X-ray studies this is proved to be chamosite.

Si shows:

a - as discrete angular and subangular grains which are shown from X-ray studies and petrographic studies to be quartz.

b - associated with Fe and Al in the matrix, previously proved to be chamosite.

Apatite grains also make up aggregate areas; with the major axis approximately 40 μm.

Note: This sample is the same as one in Figure 4.15 showing variations in the ore.

Figure 4.17 - Scanning electron micrograph for oolitic iron ore sample No. AS25, and the X-ray distribution for Fe, Al, Si, P and Ca.

Ooliths are ellipsoidal in shape; the length of the major axis varies from 150 μm to 450 μm. Many ooliths show a concentric banding structure with many bands. In several ooliths there are nuclei of other mineral grains which appear as subangular grains.
Figure 4.17  Scanning Electron Micrograph for Oolitic Iron Ore
Sample No. As 25 and the X-ray Distribution Maps x100

a - Scanning Electron Micrograph
b - Iron X-ray distribution map
c - Silicon X-ray distribution map
d - Aluminium X-ray distribution map
e - Phosphorus X-ray distribution map
f - Calcium X-ray distribution map
Fig. 4.17
In addition there is a clastic component angular and subangular grains the length of the major axis varies from 80 μm to 300 μm.

The matrix cements the ooliths, but there are two types of matrix material, light and dark, both of which cement the ooliths. The Fe X-ray distribution shows a high concentration of Fe both in the ooliths and in the matrix. There is a slightly higher Fe concentration in the outer area of the ooliths. In some areas of the matrix these are the lighter regions shown in the scanning electron micrograph. It also shows that the iron concentration is independent of Si, Al, Ca or P content. The Si occurs primarily as discrete areas of high concentration not associated with the other elements. The areas of high Si concentration are associated with angular and subangular detrital fragments seen in the scanning electron micrograph. Also there are angular grains of Si in the cores of some ooliths.

Both the Ca and P content are very low and there is one small anhedral grain of P associated with Ca. The length of the major axis of the anhedral grain is about 10 μm.

Fe is shown in:

a - the matrix associated with moderately high Al and low Si. From X-ray studies this is identified as chamosite.
b - the ooliths have a high concentration of Fe in
the outer layer, but in the middle of the ooliths
the Fe is associated with Al and Si; as previously
these proved to be chamosite.

Si shows:

a - as discrete angular and subangular grains proved
from X-ray studies and petrographic studies to be
quartz.

b - associated with Al and Fe in the matrix and in the
middle of the ooliths. This, as previously, was
proved to be a chamosite.

Ca and P occur as apatite.

Figure 4.18 - Scanning electron micrograph for oolitic
iron ore sample No. AS8, and the X-ray
distribution maps for Fe, Al, Si, P and Ca

The single oolith is ellipsoidal in shape with a length of
the major axis of about 750 μm.

It shows a concentric banded structure with subhedral grains
found within the concentric layers; the length of the major
axis for these grains varies from 5 μm to 20 μm. Also there
is one subangular grain in the centre of the oolith lighter
than the others (just below centre of micrograph); the
length of the major axis is approximately 60 μm. The Fe
X-ray distribution map shows a high concentration of Fe in
the oolith. It also shows that the concentration of Fe is
independent of the concentration of Si, Al, Ca or P. The
Fig. 4.18
Si occurs primarily as discrete areas of high concentration and is not associated with other elements. The areas of high Si concentration are associated with the subangular detrital fragments seen in the scanning electron micrograph, also shown as areas of moderately high Si concentration in the matrix. (Top right of S.E.M.)

Al is seen as areas of moderately high Al concentration associated with moderately high Si concentration in the matrix and appears as subhedral grains of high Al concentration in the oolith for which the length of the major axis is about 15 µm.

High Ca concentration is always associated with areas of high P concentration in the oolith, the discrete subhedral grains ranging in size from 5 µm to 25 µm.

Fe is shown: in the ooliths which are rich in Fe and from X-ray studies and petrographic studies it was shown to be in the form of hematite.

Si shows:

a - as discrete subangular grain. X-ray studies proved it to be quartz.

b - as a, areas in the matrix in which it is associated with Al and proved from X-ray studies to be clay minerals.

c - as discrete subhedral grains of low Si concentration associated with Al and P (P bearing clay minerals).
Al shows:
   a - as areas in which it is associated with Si; as previously these were proved to be clay minerals.
   b - as discrete subhedral grains associated with Si and P (P bearing clay minerals).

P shows:
   a - a discrete subhedral grain associated with Al and low Si concentration.
   b - associated with Ca. From X-ray studies proved these to be apatite.

Figure 4.19 - Scanning electron micrograph for oolitic iron ore sample No. AS9-2 and the X-ray distribution map for Fe, Si, Al, P and Ca

Ooliths are ellipsoidal in shape; the length of the major axis varies from 70 µm to 500 µm. Many ooliths show a concentric banding structure. In some of the ooliths there are nuclei of certain mineral grains which appear as lighter areas in the core of the ooliths as seen in scanning electron micrograph. The oolith seen in the middle of the scanning electron micrograph is divided into two parts. The outer rim appears lighter in colour and better polished than the adjacent inner layers.

There are subangular grains in the matrix; the length of the major axis varies from 40 µm to 160 µm.

The matrix cements the ooliths and there are two types of matrix material - light and dark.
Figure 4.19  Scanning Electron Micrograph for Oolitic Iron Ore
Sample No. As9-2 and X-ray Distribution Maps x200

a - Scanning Electron Micrograph
b - Iron X-ray distribution map
c - Silicon X-ray distribution map
d - Aluminium X-ray distribution map
e - Phosphorus X-ray distribution map
f - Calcium X-ray distribution map
Fig. 4.19
The Fe X-ray distribution map shows a high concentration of Fe in both the matrix and the ooliths. There is a slightly higher Fe concentration in the outer rim of the middle oolith than the inner layers. Some Si occurs as discrete areas of high concentration not associated with the other elements. The areas of high Si concentration are associated with the subangular detrital fragments seen in the scanning electron micrograph.

Also there are anhedral grains high in Si concentration. These divide the oolith into two parts; the length of the major axis is about 10 μm. In each part there is a core of moderately high Si associated with moderately high Al.

Ca and P are associated with each other and are seen as irregular and variable size patches surrounding the oolith. They are also seen surrounding one subrounded high concentration Si grain. There are some anhedral Ca and P grains in the matrix; the length of their major axis is approximately 10 μm.

Fe is shown in:

a - the matrix which is rich in Fe, shown from X-ray studies and petrographic studies to be hematite.

b - ooliths as only Fe; again shown to be hematite.

Si shows:

a - as discrete subangular grains in the matrix and anhedral grains in the ooliths (quartz).
b - associated with Al in the core of the ooliths, proved to be clay minerals.

Ca and P show:

a - associated with each other; (apatite).

Apatite can be seen as irregular patches surrounding the ooliths and as anhedral grains in the matrix.

Figure 4.20 - Scanning electron micrograph for oolitic iron with sand sample No. AS11 and the X-ray distribution map for Fe, Si, Al, P and Ca.

Shows long subangular grains in the matrix as well as subrounded white grains for which the length of the major axis is about 10 µm. Also seen are irregular areas variable in size. The size varies from 30 µm to 60 µm. The matrix is uniform except for a small area in the top left of the scanning micrograph.

The Fe X-ray distribution map shows a high concentration of Fe in the matrix. It also shows that the concentration of Fe is lower in the area to the left of the Ca pocket. (In the middle of scanning electron micrograph). The Si occurs as discrete areas of high concentration and is not associated with the other elements. The areas of high Si concentration are associated with the two long subangular grains. The low Si concentration area to the left of the pocket has its Si associated with low Al and Fe concentration. Al is shown to be associated with Fe and Si in the matrix.
Ca occurs as discrete subrounded grains of high concentration and is not associated with the other elements. It is also seen as irregular pockets in which it is associated with P; the length of the major axis varies from 30 μm to 60 μm.

Also there is a Ca and P grain for which the length of the major axis is about 20 μm.

The Fe is shown to be in the matrix. From X-ray and petrographic studies the Fe was shown to exist as hematite.

Si shows:

a - as long subangular grains which were shown from X-ray studies to be quartz.

b - as matrix surrounding the Ca and P grains; proved to be quartz.

c - associated with Fe and Al; from X-ray studies it was proved to be clay minerals.

Ca shows:

a - as subrounded grains which are shown from petrographic studies to be calcite.

b - associated with P. X-ray studies proved these to be apatite.
Ooliths are ellipsoidal or circular in shape; the length of the major axis varies from 90 µm to 290 µm. Most of them show a concentric banded structure. In one oolith, in the middle of the scanning electron micrograph, there is the nucleus of another mineral grain which subsequently appears as two subangular grains in the core of the oolith; also there are fine anhedral grains within the concentric layers in the oolith. The matrix cements the ooliths and there are two types of matrix materials. The Fe X-ray distribution map shows a high concentration of Fe in the ooliths. It also shows that there are anhedral grains of Fe which occur in the matrix; the length of the major axis varies from 5 µm to 60 µm.

Si occurs as small anhedral grains of high concentration in the matrix and is not associated with other elements; the length of the major axis is about 10 µm. Also there are subangular grains of moderately high Si concentration associated with moderately high Al concentration in the core of the middle ooliths shown in the electron micrograph. There are fine anhedral grains of Si associated with Al within the concentric layer in the oolith. In addition to this, there are some fine particles of Si dispersed in the ooliths and the matrix.

Al occurs within subangular grains in the core of the middle oolith, as anhedral grains within the concentric
Figure 4.21  Scanning Electron Micrograph for Oolitic Iron Ore
Sample No. As 20 and X-ray Distribution Maps x200

a - Scanning Electron Micrograph
b - Iron X-ray distribution map
c - Silicon X-ray distribution map
d - Aluminium X-ray distribution map
e - Phosphorus X-ray distribution map
f - Calcium X-ray distribution map
layer and as fine grains dispersed in the ooliths and in the matrix.

Ca and P are associated with each other in the matrix and also seen as fine anhedral grains in the ooliths; the length of the major axis varies from 5 μm to 20 μm.

Fe is shown in:

a - ooliths rich in Fe (shown to be hematite)

b - as anhedral grains in the matrix; again shown to be hematite.

Si shows:

a - as anhedral grains (quartz)

b - associated with Al. Proved from X-ray studies to be clay minerals.

Ca and P are shown:

a - in the matrix associated with each other. Shown from X-ray studies to be apatite.

b - as anhedral grains in the ooliths. These proved to be apatite.

4.3.4.6 Summary of results

It will be recalled that this ore body is an oolitic iron bearing ore of the Minette type, occurring as bands in the middle group of the Nubian series. These horizontal bands are interbedded with clays and Ferruginous sandstones.
Fortunately, the iron ore bands are at the surface or close thereby and the open-cast method of mining is possible. The Aswan iron ore deposits can be classified into three different forms: the Ferruginous sandstones, the Ferruginous concretions and the Oolithic iron ore. From the chemical analysis of Aswan cores it is seen that the oolitic samples from the first core contains 42-55% Fe and 0.1-2.9% P and the second core analysis 41-57% Fe and 0.35-2.3% P. It is also seen that the analysis of the Ferruginous sandy clays in the two cores contained 6.5-20.2% and 44.1-41.8% Fe, with P content between 0.5-1.4% and 1.1-3.8% respectively, while the sandstone bed contained iron between 1-8% and phosphorus between 0.04-0.14%.

From X-ray examination the hematite was shown to be the major iron bearing phase with varying amounts of quartz and apatite, with only a little clay minerals.
5. FRODINGHAM IRONSTONE

5.0 Introduction

The iron making process as it is known today started in 1709 when Abraham Darby of Coalbrookdale successfully used coke as a fuel in his furnaces in Shropshire \(^{28,29}\). Many improvements in the making of iron and the perfecting of wrought iron followed during the next hundred years. Up to the middle of the nineteenth century almost the whole of the iron produced in the United Kingdom was smelted from home iron ores. Most of these were sedimentary ores known as ironstones. Since 1875 there has been a swing away from the high phosphorus containing home ores to foreign supplies with their higher iron contents and much lower phosphorus contents. The chief reasons for this movement were decreased unit cost of the iron in the richer ores with increased production and improved quality of the iron product. The proportion of imported to total ore consumed (Figure 5.1) increased from nil before 1860 to about 30\% by 1900, and remained constant at that level during the 1939-1945 war, during which imports were restricted. Since the war the proportion of imported ore started to rise again, until it is now about 60\% \(^{30}\).

The tonnage of home ores produced is shown in Figure 5.2 for 5-year periods since 1865 \(^{30}\). There has been considerable fluctuation, with a record production of nearly 20 Mt in 1942. From 1950 to 1965 output was in the range 15-17 Mt per year, but since that date it has fallen to 12.0 Mt in 1970. Thus the expansion and
FIG. 5.1
THE PROPORTION OF IMPORTED ORE
FIG. 5.2
AVERAGE CONSUMPTION OF BRITISH HOME IRON ORE IN
5-YEARLY PERIODS
restructuring of the iron and steel industry since 1950 has been largely based on imported ore and it is intended that this trend should continue. The reasons for this are as before resulting in a reduction of iron and steelmaking costs. Although the use of basic oxygen converters is recognised to be the most economical means of large scale steelmaking using molten iron, it is most profitable working with relatively low phosphorus content in the charge (not more than 0.25%).

The United Kingdom's iron ore reserves are approximately 3,000 Mt. Those in Northamptonshire are estimated at 1.050 Mt containing 0.3-1.0% P and 27-34% Fe. The Frodingham deposit in Lincolnshire contains reserves in excess of 1,000 Mt. It is also phosphoric with an average phosphorus content of 0.35% and iron content of 25% (31). Consequently it is clear that there are large iron ore resources with little practical value in the foreseeable future. These considerations have suggested that an investigation into the possibility of eliminating the phosphorus present in the iron ore or reducing its percentage to acceptable levels would be worthwhile. A number of research projects have been undertaken to reduce the concentration of phosphorus in Frodingham ironstone. One (32) attempted concentration by flotation and another(33) used roasting followed by magnetic separation. In both processes the phosphorus content in the ore and in the concentrated product was nearly the same.
Mineralogic examination of the structure of ironstone is an important part of ore-dressing research. Unfortunately, this technique is not sufficient to elucidate the complex intergrowth of gangue minerals with the valuable iron containing parts of the ore. These can be solved only by the use of advanced modern mineralogical examinations. Most of the analyses and examinations carried out in the earlier work were insufficient to meet the demands for the ore-dressing of Frodingham ores. These analyses do not define carefully enough the mineralogical character of ores showing the amount of impurities and the intergrowth of the useful and gangue components. It is not possible to solve the release size of grains, or, in the case of a complex ore, the combined technology of multi-stage separation.

The mineralogical examination of Frodingham ironstone carried out in this research programme utilised, for the first time, Electron microprobe and Scanning electron microscopy. Their introduction has helped to increase the description of the mineral distribution and sizes throughout the ore samples.

This application of modern electron metallographic techniques to elucidate the mineral particle sizes, quantity and distribution within an ore sample illustrates clearly the use of these techniques to practical problems in the field of mineral dressing for the production of improved mineral concentrates.
5.1 Stratigraphy
The Frodingham ironstone bed is situated in north Lincolnshire, east of Scunthorpe (Figure 5.3 a and b). It occurs in the lower Jurassic bed of the South Humberside area and the geology of the area is shown in Figure 5.4. The ore contains highly fossilised ferruginous ooliths, with ooliths of chamosite, siderite, and limonite, together with shell fragments, set in a matrix of either chamosite-sideritic mudstone or coarsely crystalline calcite. The beds range in thickness up to a maximum of about 32 feet. The average Fe content is 25% whilst the average SiO₂ content is 7.5%, CaO 22%, and Phosphorus 0.35%. It has a workable outcrop length of 7 miles north to south (34). The beds extend, under increasing cover, at least for 20 miles to the east.

A generalised section of rocks in this ground with their variation in thickness is as shown in Figure 5.5.

5.2 Lithology (35)
In the southern half of the quarried area the ironstone as a whole is made up of green and brown beds, but in the northern part of the field the general colour is brown with many beds approaching red in colour.

On the whole the ironstone is remarkably free from argillaceous matter. The upper beds near the surface are usually darker and more oxidised than the beds beneath. They are more broken up and carry a considerable amount of natural fines; i.e. the average size of the ore is less
Illustration removed for copyright restrictions
Illustration removed for copyright restrictions
Fig 5.5 THE GEOLOGY OF THE SCUNTHORPE AREA

GEOLOGICAL SUCCESSION

LITHOLOGY

3"-8" CORMBRASH
Buff Limey Sandstone

17"-50" GREAT OOLITE SERIES
Mainly Clays

15"-32" UPPER ESTUARINE SERIES
Limestone Locally Sands and Sandy Clays with Lst. Sands,
Silt and Marls

HIBALDSTON OOLITE
Buff and Grey Oolitic Limestone

43"-71" LINCOLNSHIRE LIMESTONE
Occasional Beech Knots

KIRTON CEMENT STONE Alternating Grey Calcareous Clays and
SANDY OOLITE Compartments

0"-24" LOWER ESTUARINE SERIES
Ferruginous with Boxstones of Northampton Sands
NORTHAMPTON SANDS Locally as Limey Sandstone - basal Fossil

40"-60" UPPER LIAS CLAYS
Grey Clay and Brown Shale

66"-73" LOWER LIAS CLAYS
Grey Clays

21"-61" PECTEN RED
Brown Oolitic-Lime Ironstone

40"-100" LOWER LIAS CLAY Dark Grey Clays

50" PRODINGHAM IRONSTONE
Clayey Ironstone
Limonite Ooliths in Clay Matrix with Siderite and Chamosite Linen Ironstone

APPX. LOWER LIAS CLAYS Shales, Clays and Limestones

E = Erosion surface
than three quarters of an inch, (20 mm) this is undoubtedly due to the greater effects of weathering in the higher beds. The ore is highly fossilised and contains thick shells, mainly the lamellibranchs cardinia and gryphaea, which are preserved in crystalline calcite. The excessively calcareous nature of the stone is largely due to the presence of these fossils.

Ooliths of almost pure limonite are found throughout the ironstone, but their distribution is not uniform; they may be patchy in one place, uniform in another and sporadic elsewhere. The individual ooliths also are variable in size and shape; generally they are small with a tendency to be elongated rather than spherical.

The uppermost beds are chamositic mudstones consisting of brown limonitic ooliths with shell fragments and occasional scattered sand grains in a matrix made up of innumerable minute flakes of chamosite roughly parallel with the bedding together with a few flakes of mica and fine quartz plus numerous small rhombs of siderite with dolomite nuclei.

The lower beds are generally more calcareous and their predominantly green or greenish-brown colour is due to the fact that the matrix is essentially chamositic mudstone. Parts of individual beds are frequently greyish-blue in colour where the matrix is limestone.

The more limey beds, occurring chiefly in the lower part of the ironstone, are made up of an aggregate of brown
limonitic ooliths, rounded shell fragments and crinoid ossicles cemented with clear crystalline calcite which frequently shows a comby structure indicating the growth of crystals from the walls of the interstitial spaces. Complete lamellibranch shells are often lined with calcite crystals and sometimes wholly or partly filled with a smooth chamositic mud. Some of the shells are worn and riddled with the branching tubules of 'algues perforantes', while others are uninjured or only superficially chloritized.

5.3 Mineralogy

The unweathered mineralogical constitution of the Frodingham ironstone, taken from Davies and Dixie (36) (1951, p.89), is shown in Table 5.1.

It shows that the ironstone varies according to the nature of the matrix. The ironstone with a calcite matrix is essentially a ferruginous limestone and corresponds to the "limey ore" while that with a chamosite-siderite matrix may be described as clayey ironstone. The shells (principally Cardinia) tend to occur in well-defined bands, usually 6 to 12 inches (15-30 cm) thick, the remainder of the ironstone being almost devoid of shells. Consequently, some parts of the clayey ironstone contain little or no calcite.

The nature of ooliths and the shell fragments has been described by Davies and Dixie (36), and the following is summarised from their work. The majority of the ooliths are ellipsoidal, with major axes about 0.30 mm long and twice the length of the minor axis. Locally, thin bands may contain ooliths (or pisoliths) up to 5 mm across. The
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<thead>
<tr>
<th>Principal Constituents</th>
<th>Calcite matrix</th>
<th>Chamosite-Siderite matrix</th>
</tr>
</thead>
<tbody>
<tr>
<td>Limonite - mainly as ooliths</td>
<td>30</td>
<td>35</td>
</tr>
<tr>
<td>Quartz - as small grains</td>
<td>-</td>
<td>0-10</td>
</tr>
<tr>
<td>Calcite - as shells</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>as matrix</td>
<td>30</td>
<td>-</td>
</tr>
<tr>
<td>Siderite</td>
<td>10</td>
<td>17</td>
</tr>
<tr>
<td>Chamosite</td>
<td>0-5</td>
<td>18-23</td>
</tr>
<tr>
<td>Minor Constituents</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sericite</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td>Collophane</td>
<td>1.5</td>
<td>2.0</td>
</tr>
<tr>
<td>Pyrites</td>
<td>0.4</td>
<td>0.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(locally up to 7.0)</td>
</tr>
<tr>
<td>Rutile</td>
<td>-</td>
<td>1</td>
</tr>
</tbody>
</table>
Ooliths in other bands are exceptionally small.

5.4 Mineralogical classification of the ironstone

The ironstone is classified into 4 principal types, viz. A, B, C and D (Types A, B and C are 'clayey' ironstones, whilst type D is a limey ironstone). These types are defined as follows:

**Type A**

In this, calcite shells and limonite-chamosite ooliths, more or less replaced by siderite, all coated with a thin film of chamosite, are closely packed together with the interstices filled with siderite.

The hand specimen, especially when moist, has a distinctive appearance. The siderite has been superficially oxidised to limonite so that the ironstone appears as a sponge-like mass of dark green chamosite, the pores in the sponge being filled by highly polished ooliths of brown limonite and brown aggregates of siderite.

**Type B**

In this type, small rhomb-shaped crystals of siderite are evenly distributed among linearly arranged flakes of chamosite. The hand specimen is a dense, tough, compact, dark bluish-grey rock which breaks with a granular fracture due to the siderite rhombs.
Ooliths and other coarse material are absent. It occurs usually as discontinuous bands, an inch or two thick (2-5 cm), among other types of ironstone and is far less abundant than the others.

**Type C**

This type is characterised by limonite-chamosite ooliths, more or less replaced by siderite, and calcite shells embedded in an extremely fine-grained matrix composed of chamosite and siderite. In the hand specimen, highly polished limonite ooliths are scattered irregularly through a dark bluish-grey matrix. Unlike Type B, this matrix is quite soft when wet, and unlike Type A, chamosite has partly replaced, and not merely coated, the calcite shells. Locally, small subangular quartz grains may be present.

**Type D**

In this type, limonite-chamosite ooliths and calcite shells, more or less replaced by siderite, are set in a matrix composed of coarsely crystalline calcite, some siderite and, less commonly, pale-green chamosite may be present in the matrix. This type is essentially a hard, shelly limestone containing coarse iron bearing particles. The calcite matrix, though often limonite stained, can usually be distinguished in the hand specimen. This ironstone has been formed mainly by the mechanical disintegration of ironstone of the three preceding types, the debris so formed being cemented together with calcite. The occurrence of type D ironstone indicates that, after a period of quiescent conditions on the sea-floor during which ironstones of types A, B and C were deposited, turbulent conditions supervened and churned up the last-formed deposits.
5.5 Experimental Work

5.5.1 Material

The material used in this study was taken from split cores provided by British Steel Corporation. Cores Y 112, and Y 113 were drilled from the Yarborough pit, (Figure 5.3b). Core drilling was done using 101 mm or 116 mm drilling crows, and core recovery was approximately 100 percent.

Ironstone cores were split longitudinally and one half of the core was boxed in one foot increments. These were collected from Scunthorpe and the following investigations made:

- Descriptive logging
- Photography
- Chemical analyses
- Microscopic examination
- X-ray mineralogical examination
- Microprobe analysis
- Scanning electron microscopy

The other portion of the core was crushed in one foot increments by B.S.C. for chemical analysis and the results are presented in Tables 5.2 and 5.3.

Photographs of Frodingham ironstone cores Y 112 and Y 113 are shown in Figures 5.6 and 5.7, showing the various ironstone types which are readily recognised.

5.5.2 Core logs

Detailed logging of the cores (Figs. 5.8 and 5.9) has shown that a variety of different lithologies are present in each.
<table>
<thead>
<tr>
<th>No.</th>
<th>Depth</th>
<th>Iron %</th>
<th>L.H.o.</th>
<th>Insol %</th>
<th>Sal %</th>
<th>Phos %</th>
<th>X-ray</th>
<th>Scanning Electron Microscope</th>
<th>Electron Microprobe analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>76’3”</td>
<td>32.1</td>
<td>1.5</td>
<td>23.0</td>
<td>0.050</td>
<td>0.46</td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>77’3”</td>
<td>32.1</td>
<td>1.5</td>
<td>23</td>
<td>0.050</td>
<td>0.46</td>
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<td>-1</td>
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<td></td>
<td>Majority is SiO₂, trace of</td>
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<td>chamosite and siderite,</td>
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<td></td>
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<td></td>
<td>and little apatite. Most</td>
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<td></td>
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<td></td>
<td>of the sample is SiO₂,</td>
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<td></td>
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<td></td>
<td></td>
<td>small amount of calcite and</td>
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<td></td>
<td></td>
<td></td>
<td>little of chamosite, apatite</td>
<td></td>
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<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>hemaite and siderite.</td>
<td></td>
</tr>
<tr>
<td>A7-2</td>
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<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>A8</td>
<td>79’3”</td>
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<td>11.5</td>
<td>17.0</td>
<td>0.037</td>
<td>0.16</td>
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<td>Most of the sample is SiO₂,</td>
<td></td>
</tr>
<tr>
<td></td>
<td>80’3”</td>
<td>26.6</td>
<td>12.1</td>
<td>22.9</td>
<td>0.060</td>
<td>0.18</td>
<td></td>
<td>and calcite, little siderite,</td>
<td></td>
</tr>
<tr>
<td></td>
<td>81’3”</td>
<td>22.4</td>
<td>11.4</td>
<td>28.8</td>
<td>0.578</td>
<td>0.23</td>
<td></td>
<td>chamosite, hemaite and</td>
<td></td>
</tr>
<tr>
<td></td>
<td>82’3”</td>
<td>23.4</td>
<td>17.8</td>
<td>11.5</td>
<td>0.408</td>
<td>0.10</td>
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<td>apatite.</td>
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<td>83’3”</td>
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<td>15.5</td>
<td>7.3</td>
<td>2.308</td>
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<td>12.8</td>
<td>16.4</td>
<td>0.343</td>
<td>0.23</td>
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<tr>
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<td>85’3”</td>
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<td>17.4</td>
<td>6.3</td>
<td>0.458</td>
<td>0.16</td>
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<td>86’3”</td>
<td>24.8</td>
<td>25.0</td>
<td>4.1</td>
<td>0.730</td>
<td>0.20</td>
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<td>87’3”</td>
<td>23.3</td>
<td>24.1</td>
<td>8.2</td>
<td>0.765</td>
<td>0.12</td>
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<td>88’3”</td>
<td>21.7</td>
<td>30.0</td>
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<td>0.825</td>
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<td>37.1</td>
<td>11.3</td>
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<td>0.417</td>
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<tr>
<td></td>
<td>90’3”</td>
<td>29.0</td>
<td>21.0</td>
<td>3.9</td>
<td>0.174</td>
<td>0.37</td>
<td></td>
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</tr>
<tr>
<td></td>
<td>91’3”</td>
<td>36.6</td>
<td>11.2</td>
<td>4.7</td>
<td>0.130</td>
<td>0.38</td>
<td></td>
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</tr>
<tr>
<td>A53</td>
<td>92'10&quot;</td>
<td>43.3</td>
<td>6.0</td>
<td>0.1</td>
<td>0.501</td>
<td>0.49</td>
<td>Majority is SiO$_2$, small amount of calcite and hematite, trace of apatite.</td>
<td>Ooliths of Fe, matrix is mainly Ca associated with little Si and Al.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>93'10&quot;</td>
<td>21.7</td>
<td>28.6</td>
<td>3.7</td>
<td>0.701</td>
<td>0.32</td>
<td>Most of the sample is calcite and SiO$_2$, little hematite and apatite.</td>
<td>Ooliths of Fe, shell fragment of Ca, matrix is mainly Ca with little Fe, Si, Al and P.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>94'10&quot;</td>
<td>25.2</td>
<td>21.8</td>
<td>8.9</td>
<td>1.260</td>
<td>0.20</td>
<td></td>
<td>Ooliths of Fe, one oolith of Fe associated with Ca, Si, Al and P, matrix is mainly Ca with Fe, Si, Al and P.</td>
<td></td>
</tr>
<tr>
<td>A55</td>
<td>95'0&quot;</td>
<td>15.9</td>
<td>36.3</td>
<td>3.5</td>
<td>0.348</td>
<td>1.12</td>
<td>Most of sample is calcite and SiO$_2$, little hematite and apatite.</td>
<td>Ooliths of Fe, associated with Si, Al and Ca, P occurs as fine grains and as amelhedral grains (3µm), associated with Ca.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>96'0&quot;</td>
<td>16.6</td>
<td>35.7</td>
<td>2.8</td>
<td>0.321</td>
<td>0.32</td>
<td></td>
<td>Ooliths of Fe associated with Si and Al, Ca and P; matrix is Ca, in one oolith amelhedral grains of P and Ca (15-33µm)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>97'0&quot;</td>
<td>27.7</td>
<td>19.8</td>
<td>6.0</td>
<td>0.551</td>
<td>0.48</td>
<td></td>
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</tr>
<tr>
<td>No.</td>
<td>Depth</td>
<td>Iron %</td>
<td>Lime %</td>
<td>Insol %</td>
<td>Sul %</td>
<td>Phase</td>
<td>X-ray</td>
<td>Scanning Electron Microscope</td>
<td>Electron Microprobe Analysis</td>
</tr>
<tr>
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<tr>
<td>A106</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Calcite and little siderite, chamosite, SiO₂ and hematite</td>
<td>Majority is calcite and little SiO₂, hematite and trace of chlorite and chamosite.</td>
</tr>
<tr>
<td>107</td>
<td>106'3&quot;</td>
<td>20.5</td>
<td>20.8</td>
<td>17.2</td>
<td>0.302</td>
<td>0.52</td>
<td></td>
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<tr>
<td></td>
<td>107'3&quot;</td>
<td>18.2</td>
<td>32.8</td>
<td>3.3</td>
<td>0.020</td>
<td>0.18</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Majority is hematite and little SiO₂ and trace apatite.</td>
<td>Majority is calcite and little hematite and apatite.</td>
</tr>
<tr>
<td>A116</td>
<td>108'3&quot;</td>
<td>21.6</td>
<td>24.6</td>
<td>9.2</td>
<td>0.087</td>
<td>0.24</td>
<td></td>
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</tr>
<tr>
<td>A117</td>
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</tr>
</tbody>
</table>

From 76'3" - 108'3"

SiO₂ 7.88%

Loss on ignition 24.25%
<table>
<thead>
<tr>
<th>No.</th>
<th>Depth</th>
<th>Iron %</th>
<th>Lin %</th>
<th>Iron %</th>
<th>Sulph %</th>
<th>Phos %</th>
<th>Petrography</th>
<th>Scanning Electron Microscope</th>
<th>Electron Microprobe Analyzes</th>
</tr>
</thead>
<tbody>
<tr>
<td>08</td>
<td>30.2</td>
<td>8.2</td>
<td>21.9</td>
<td>0.082</td>
<td>0.35</td>
<td></td>
<td>Limestone occurs as ooliths and a fine matrix material. Ooliths are generally ellipsoidal, some are circular, some are broken, matrix in general siderite with some ferroan calcite and shell fragments</td>
<td>Fe rich ooliths with Si; some ooliths replaced partially by Ca; fine grains of P associated with Ca in the ooliths. Matrix is mainly Ca.</td>
<td></td>
</tr>
<tr>
<td>09</td>
<td>30.5</td>
<td>6.2</td>
<td>21.6</td>
<td>0.082</td>
<td>0.35</td>
<td></td>
<td>Limestone ooliths, matrix is limonite and siderite in some areas, and ferroan calcite in other areas. Also there are angular grains of quartz and shell fragments of calcite.</td>
<td>Fe rich ooliths, fine grains of phosphorus associated with fine grains of Ca occur in the ooliths. Matrix is fine grains of Fe, Si, Al and Ca.</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>32.0</td>
<td>6.5</td>
<td>21.6</td>
<td>0.037</td>
<td>0.33</td>
<td></td>
<td>Limestone ooliths ellipsoidal to circular in shape. Occasionally ooliths have cores of subangular quartz grains or calcite fragment. Variable size patches of limonite matrix, most of the matrix is ferroan calcite</td>
<td></td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>27.4</td>
<td>9.1</td>
<td>25.7</td>
<td>0.073</td>
<td>0.36</td>
<td></td>
<td>Chamosite ooliths; calcite shell fragments are set in the matrix of fine grained chamosite, siderite, ferroan calcite and siderite</td>
<td></td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>22.2</td>
<td>11.9</td>
<td>23.8</td>
<td>1.379</td>
<td>0.21</td>
<td></td>
<td>Anglular and subrounded of quartz grains, matrix is chamosite, shell fragment of calcite, ferroan calcite occurs in some areas in the matrix, pyrite occurs in the matrix and in the shell fragment.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>13</td>
<td>31.4</td>
<td>12.5</td>
<td>8.2</td>
<td>0.147</td>
<td>0.17</td>
<td></td>
<td>Limestone ooliths set in chamosite matrix, siderite grains and shell fragment in the matrix.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>27.6</td>
<td>7.8</td>
<td>7.2</td>
<td>0.655</td>
<td>0.39</td>
<td></td>
<td>Limestone ooliths surrounded by thin rim of chamosite, matrix is siderite and chamosite, in some ooliths limonite is partially replaced by siderite</td>
<td></td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>34.0</td>
<td>14.0</td>
<td>6.3</td>
<td>0.073</td>
<td>0.32</td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>No.</td>
<td>Depth</td>
<td>Iron %</td>
<td>Lado</td>
<td>Inosil</td>
<td>Sulph %</td>
<td>Phos %</td>
<td># Petrography</td>
<td></td>
<td></td>
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<td>-----------------------------------------------------------------------------</td>
<td></td>
<td></td>
</tr>
<tr>
<td>72</td>
<td>30.1</td>
<td>19.6</td>
<td>4.6</td>
<td>0.03</td>
<td>0.35</td>
<td></td>
<td>Scanning Electron Microscope</td>
<td></td>
<td></td>
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<tr>
<td>73</td>
<td>27.1</td>
<td>23.4</td>
<td>4.0</td>
<td>0.101</td>
<td>0.40</td>
<td></td>
<td>Lithoite ooliths surrounded by thin rim of chamosite, small patches of siderite, shell fragment of calcite occur in the matrix. Matrix is siderite with variable amount of ferroan calcite.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>74</td>
<td>30.8</td>
<td>8.1</td>
<td>6.5</td>
<td>0.467</td>
<td>0.60</td>
<td></td>
<td>Ooliths of Fe associated with little of Si, Al, P and Ca, angular grains of Ca, matrix is mainly S with fine particles of Si.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>B54</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Electron Microprobe Analysis</td>
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<td></td>
</tr>
<tr>
<td>75</td>
<td>35.7</td>
<td>13.7</td>
<td>4.4</td>
<td>4.188</td>
<td>0.40</td>
<td></td>
<td>Ooliths of Fe associated with St, Al, P and Ca. The ooliths surrounded by a rim of high Si concentration, matrix mainly fine Fe.</td>
<td></td>
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</tr>
<tr>
<td>76</td>
<td>18.3</td>
<td>31.4</td>
<td>5.7</td>
<td>0.458</td>
<td>0.20</td>
<td></td>
<td>Ooliths of Fe with Si and Al; fine P associated with Ca occur in the ooliths. Matrix is mainly Ca.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>B63</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Ooliths of Fe associated with fine particles of Si, Al, P and Ca. Matrix is Ca.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>77</td>
<td>22.7</td>
<td>24.8</td>
<td>9.1</td>
<td>0.115</td>
<td>0.50</td>
<td></td>
<td>Fe ooliths associated with Si and Al. Matrix is mainly Ca. Small particles of P are associated with Ca.</td>
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<tr>
<td>B69</td>
<td></td>
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<td></td>
<td>Ooliths of Fe with little fine grains of Si, Al, P and Ca. Matrix is Ca.</td>
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<td>78</td>
<td>15.7</td>
<td>35.9</td>
<td>3.3</td>
<td>0.101</td>
<td>0.20</td>
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<td>Ooliths of Fe associated with Al and Si. Matrix is mainly P and Ca associated with fine particles of Fe, Si and Al.</td>
<td></td>
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</tr>
<tr>
<td>79</td>
<td>24.7</td>
<td>22.4</td>
<td>5.7</td>
<td>0.033</td>
<td>0.35</td>
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<td>80</td>
<td>22.2</td>
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<td>6.0</td>
<td>0.082</td>
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<td>82</td>
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<td>83</td>
<td>18.6</td>
<td>31.3</td>
<td>3.5</td>
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<td>B100</td>
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<tr>
<td>85</td>
<td>23.5</td>
<td>18.4</td>
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<thead>
<tr>
<th>No.</th>
<th>Depth</th>
<th>Iron %</th>
<th>Lime %</th>
<th>Kao %</th>
<th>Soln %</th>
<th>Sulph %</th>
<th>Phos %</th>
<th>Petrography</th>
<th>Scanning Electron Microscope</th>
<th>Electron Microprobe Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>B113</td>
<td>86</td>
<td>16.7</td>
<td>33.8</td>
<td>4.5</td>
<td>0.133</td>
<td>0.28</td>
<td></td>
<td>Ooliths of Fe; matrix is mainly Ca with little Si.</td>
<td></td>
<td>Fe ooliths associated with</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>In the lower part, limonite ooliths, two types of matrix material, fine</td>
<td></td>
<td>fine particles of Si, Al, P</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>grained limonite; fine grained limonite with foraminiferal calcite. In</td>
<td></td>
<td>and Ca; matrix is Ca.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>upper part limonite ooliths calcite shell fragment set in foraminiferal</td>
<td></td>
<td>In some ooliths Fe replaced</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>calcite matrix.</td>
<td></td>
<td>partially by Ca.</td>
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<td>B117</td>
<td>87</td>
<td>20.4</td>
<td>27.6</td>
<td>8.5</td>
<td>0.115</td>
<td>0.41</td>
<td></td>
<td>Ooliths of Fe, angular Ca grains, matrix is mainly Ca.</td>
<td></td>
<td>Oolith of Fe associated with</td>
</tr>
<tr>
<td></td>
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<td></td>
<td></td>
<td></td>
<td>Quatzt grains, limonite ooliths shell fragment are set in a chamosite</td>
<td></td>
<td>Si, Al, P and Ca; matrix is Ca.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>cement.</td>
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<tr>
<td>B123</td>
<td>88</td>
<td>14.5</td>
<td>20.2</td>
<td>13.5</td>
<td>0.192</td>
<td>0.25</td>
<td></td>
<td>Ooliths of Fe with little Si, Al, P and Ca. matrix is mainly Ca.</td>
<td></td>
<td>Ooliths of Fe associated with</td>
</tr>
<tr>
<td></td>
<td>89</td>
<td>19.8</td>
<td>28.8</td>
<td>6.2</td>
<td>0.057</td>
<td>0.31</td>
<td></td>
<td></td>
<td></td>
<td>Si, Al, P and Ca; unchaidral</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>17.5</td>
<td>30.7</td>
<td>7.9</td>
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<td>grains of Si associated with</td>
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<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Al occur in the matrix, matrix</td>
</tr>
<tr>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>is Ca.</td>
</tr>
</tbody>
</table>

From 39°0' - 90°0'

\( \text{SiO}_2 \quad 8.58\% \\
\text{Loss on ignition} \quad 24.15\%

*Petrography was performed on 40 thin polished sections & 30 polished sections*
Figure 5.6  Typical Reference
Photograph of Split Core Y 112
(Frodingham Ironstone)
Figure 5.7  Typical Reference
Photograph of Split Core Y 113
(Frodingham Ironstone)
Illustration removed for copyright restrictions
### Lithology of Appleby and Ingamhám Cores

A geologist (S. James) was working on the structure of ooliths in ironstones. Consequently, he was very interested in the two cores from Yarborough which are the subject of a part of the present work. Consequently, he prepared a complete lithology of the two cores and these are included here in order to locate the samples described in section 5.5.2.

**Figure 5.8** shows the descriptive lithology of core Y III from 76' 3" - 109' depth.

<table>
<thead>
<tr>
<th>Commence Core</th>
<th>76' 3&quot;</th>
<th>109' 0&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Chamosite lenses</strong></td>
<td>Rust brown, limonite. Ooliths are elliptical, 0.2m across, with polished surfaces. They are set in a limonite matrix. Black to dark grey, fine-grained material forms thin lenses. <strong>Grey material</strong> forms thicker lenses in place of black. Bivalve shells infilled with fine grey material.</td>
<td></td>
</tr>
<tr>
<td>Light grey material disappears. Fine dark red brown material forms thin, short lenses. Black/dark grey material returns. Black/dark grey material forms thicker lenses than before and contains a number of limonite ooliths.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Burrow infilled with fine grey material. The limonite matrix becomes reddish brown and between black lenses is dark grey. Black lenses in frequency and are 0.7/1cm thick.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lense of grey fine-grained material. Black lenses increase in frequency, but are considerably thinner, shorter and often curved. Black/dark grey material disappears, red takes over as thin lenses. Lense of grey fine-grained material. Red fine-grained material appears forming short and long thin lenses. Red material disappears, grey and grey green takes over. Grey material occurs.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

---

**Figure 5.8** shows the descriptive lithology of core Y III from 76' 3" - 109' 0" depth.
Lenses decrease rapidly in size and are all of green chamosite calcite rich shell band.

- Numerous irregular lenses of different thickness of fine grained grey material and greenish chamosite material are present. These lenses occur as small rounded patches. The lenses contain a few ooliths. Surrounding these limonite ooliths is a matrix made up of discoidal and elliptoidal 0.1mm ooliths.

Limonite ooliths is cemented with calcite above and below the shell band.

- The greenish grey fine grained material increases very rapidly in quantity and makes up 70% of the rock at the top of the unit. The limonite ooliths are altered to a dull yellow colour and are seldom in appearance.

Shell band

- Grey green chamosite occurs in two intercalated forms (i) massive and fine grained (city) lighter coloured and pitted. The latter contains numerous small dull yellow white oolith shaped grains. It is very fine grained. Many complex shell fragments occur.

Ellipsoids with hematite skins

- Fine grained greenish grey lenses with rounded ends are surrounded by green white oolitic containing dull white ooliths in a green grey fine grained matrix. Spots and ellipsoidal lenses are margined by hematite material.

Pyrite cubes common

- Greenish white colour to rock with small 0.1mm white ooliths in a chamosite matrix.

Pyritic pisoliths

- Pisolith contains pyrite distributed parallel to nucleus.

Shell band of much fragmented material.

- Fine grained brown material forms thin lenses and infills to shells.

Pyrite and silver metallic material forms matrix to ooliths.

- Yellowish green limonite ooliths are generally absent leaving pits. The matrix is of limonite coloured yellowish white and not chamosite giving a greenish colour. Occasional dull light green and light green ooliths occur. Limonite lenses or clasts of light green, fine grained, chamosite are present and often cavernous. They may be surrounded by a fine dark red brown skin.

Cavernous chamosite clasts

- Ooliths become rather more common. Although some are still absent.

They are discoidal and elliptoidal, 0.1mm across of a dull yellow limonite end of chamosite. Associated with the shell portion as clasts or lenses of chamosite occur and are rimmed by dark brown material. This may also infill shells. Limonite pisoliths occur. The lowest 2m is poor in ooliths, rich in chamosite.

Pisoliths and chamosite clasts

- Ooliths become rather more common. Although some are still absent.

They are discoidal and elliptoidal, 0.1mm across of a dull yellow limonite end of chamosite. Associated with the shell portion as clasts or lenses of chamosite occur and are rimmed by dark brown material. This may also infill shells. Limonite pisoliths occur. The lowest 2m is poor in ooliths, rich in chamosite.

Barn, fine grained lens...

- Massive, grey green rock. Fine grained limonite ooliths are present but not closely packed. Small spots of a light green chamosite are scattered throughout. Lenses of olive brown limonite oolite occur in the lower half. Ooliths in a this are discoidal, 0.1mm across with polished surfaces. They are set in a chamosite matrix. Little shell material is present.
<p>| 84’31’   | Shell band with limonite-oolite clast. | Greenish grey shell rich material. The matrix is of fine-grained greenish grey chalk. Occasional limonite ooliths occur. Limonite oolite clasts, with limonite matrix. Other coloured ooliths. Ooliths are ellipsoidal, 0.5mm across, with slightly tarnished surfaces. These are set in a matrix of limonite, chamosite and calcite. Limonite and chamosite pisoliths occur. Lenses of dark grey green chamosite and a fine red brown material occur. Chamosite post-dates this as it is often made around it. Rock is altered. Large areas of a fine, dull limonite occur and contain relicert. Small, fine-grained, grey patches. Chamosite rich areas are dark grey green. Chamosite matrix areas are pitted, the ooliths being generally altered. Yellowish olive brown limonite oolite. Ooliths are ellipsoidal and dolomitic with polished surfaces, 1mm across. The upper portion has limonite matrix, the lower portion a dominantly calcite cement. Chamosite is occasionally a matrix mineral. Small fine-grained pisoliths occur. Lower 3m olive brown limonite oolite. Ooliths are ellipsoidal, 1mm across, with polished surfaces. Chamosite also occur. They are set in a chamosite matrix. Brownish grey lime-rich oolite. Ooliths are ellipsoidal, 1mm across, with polished surfaces. The matrix is generally grey calcite but in places near the top is of olive brown limonite. Shell material occur with coarsely crystalline interstitial calcite. Occasionally limonite ooliths are present in this. Lenses of dark black/brown material with areas of chamosite and few ooliths. Olive green limonite. Ooliths are ellipsoidal, 0.5mm across, and dull yellow brown in colour. They are set in a chamosite matrix. Irregular patches of black, fine-grained, reflective material perhaps hematite occur. Olive brown limonite ooliths are discoidal and ellipsoidal, 1mm across and dull yellow brown in colour. These are in a chamosite matrix. Small lenticles of black chamosite only occur. Shell rich band. Brownish grey limestone of shell material and coarse calcite. Polished 0.5mm ooliths. Lenses of dark black/brown material. Ooliths are ellipsoidal and discoidal, 0.5-0.6mm across, with a dull brown colour. In places these ooliths have a relieff appearance. They are set in a matrix of chamosite and limonite. Immediately above the shell rich band are lenses of fine grey green material often occurring within bivalve shells. Thin, black to dark grey, fine grained matrix. The matrix is distributed throughout the unit, but is concentrated in a band at the base. | Shell band. Olive brown limonite oolite. Ooliths are ellipsoidal and discoidal, 0.5-0.6mm across, with a dull brown colour. In places these ooliths have a relieff appearance. They are set in a matrix of chamosite and limonite. Immediately above the shell rich band are lenses of fine grey green material often occurring within bivalve shells. Thin, black to dark grey, fine grained matrix. The matrix is distributed throughout the unit, but is concentrated in a band at the base. | 84’35’ | 85’10’ | 85’15’ | 86’0’ | 86’14’ | 86’19’ | 87’0’ | 87’10’ | 87’30’ | 88’ | 88’30’ |</p>
<table>
<thead>
<tr>
<th>Location</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>89°</td>
<td>Black markings commence</td>
</tr>
<tr>
<td>90°</td>
<td>Shell-rich band</td>
</tr>
<tr>
<td>90°9&quot;</td>
<td>Hellomite</td>
</tr>
<tr>
<td>91°</td>
<td>Shell band</td>
</tr>
<tr>
<td>92°</td>
<td>Thin, black, fine-grained tuff, parallel to bedding containing few shell fragments and 0.5-0.5mm ooliths. Some pyrite. Olive green limonite oolite. Ooliths generally a dull yellow brown in colour (relict) but occasionally brown and polished. They are set in a matrix of chamosite and limonite. Black to dark brown fine-grained material is present in the upper portion of the unit and may also act as a matrix material. It may also form long, thin, lenses - lenses thicker than 0.5cm and are irregular-shaped. The material contains a few ooliths. Where several dark lenses occur together, interstitial oolite is of relict limonite ooliths in a limonite matrix. Little shell material occurs.</td>
</tr>
<tr>
<td>92°20'0&quot;</td>
<td>Thin shell band</td>
</tr>
<tr>
<td>93°</td>
<td>Lenses of fine-gray material: polished limonite ooliths in a calcite matrix</td>
</tr>
</tbody>
</table>
Cont. Figure 5.8

| Chamisite matrix | Brownish grey limonite oolite. Ooliths are ellipsoidal, 0.2mm across, with polished surfaces. In the upper portion they are occasionally ellipsoidal. The upper portion has an entirely chamisite matrix; the lower calcite. The lower portion contains numerous patches of

| Calcite matrix | Shell material: Fine grey material forming lenses. Few ooliths. Patches of chamisite are also associated.

| Shell material: Fine grey material forming lenses. | Brownish olive green limonite oolite. Ooliths are ellipsoidal, 0.2mm across. They are often dull. Chamisite matrix: Fine grey: irregular lens. Greenish grey limonite oolite. Ooliths are ellipsoidal, 0.2mm across, and are dull brown in colour. They are often isolated and in places of a chalky matrix. Greenish grey limonite oolite. Ooliths are isolated and in places of a chalky matrix. They are dull brown in colour. The ooliths are set in a grey green chamisite and limonite matrix in the upper portion, which gives way to a calcite matrix.

Lense of fine grey material: Shell rich base. Poorly rounded, 1mm grains are coated with a reddish-brown matrix and set in a chamosite and limonite matrix. Ochrous patches of limonite occur. Shelly: For 1mm: has ellipsoidal 1mm ooliths. Chamisite: Fine grey matrix. Olive brown limonite oolite. Ooliths are ellipsoidal, 0.15mm across, dull brown or reddish; they are set in a grey green chamisite matrix. Olive green limonite oolite with rust markings. Ooliths are ellipsoidal, 0.2-0.3mm across and are dull brown; frequently reddish; they are set in a greyish green matrix. The matrix is limonitic with a dull yellow ochre. Patches in the upper portion contain polished ooliths. Rusty brown markings occur throughout. These are irregular in shape and due to the presence of polished reddish-brown ooliths and red brown shell fragments. The matrix is chamosite and occasionally chamisite. Shell fragments are scattered throughout and form two distinct inclines bands at the base; between and immediately above these same patches of hematite and olivine fine grained material occur. Micropisoliths of limonite 0.3mm across and polished ooliths sometimes occur. Where shell fragments are close together coarse interstitial calcite occurs.
| 101°50' | **Case of fine grey material:** Olive grey green limonite-coalite. Ooliths are discoidal and ellipsoidal; 0.5 mm across, with polished surfaces. The matrix is grey, green, and contains calcite in an irregular manner. Chamosite is also a matrix mineral. Chamosite forms lamellae where few ooliths occur. These are 1-2 mm thick and cover between the two grey bands. Shell fragments are present throughout and form two shell-rich bands. An overturned bivalve has trapped fine grey material below it.

| 102° | **Olive green, reddish tinged, limonite-coalite. Ooliths are ellipsoidal, 0.2-0.5 mm across, with polished surfaces. They are set in a matrix of greenish grey chamosite. Numerous burrows are present, infilled with other fine materials or more commonly with reddish brown material. This is composed of a red, fine grained matrix containing many ooliths. Two thirds of the way down, dark black markings occur.**

| 102°75' | **Black markings:** Black markings with a black/dark grey fine material making them up, occur subparallel to bedding but are discontinuous. The lowest 1" contains much dark green chamosite with sparse ooliths. No fauna.

| 103° | **Chamosite rich bivalve:** Olive brown limonite-coalite. Ooliths are ellipsoidal, 0.1-0.5 mm across, dull brown, and often yellow ochre in colour. The typical micro-fossils have a reticulate appearance. They are set in a dull grey green chamosite matrix. The central portion is limonitic in places. Dark red hematite markings occur in the central portion. The lower 1" is more coarse grained ooliths being 1 mm across and occasionally polished. Chamosite is present in trace quantities. Shell fragments are not uncommon, and a shell fragment-rich band occurs.

| 104° | **Shell fragment-rich band:**

| 104°57' | **Dividing line in life position with fine grey material:** Olive brown limonite-coalite. Ooliths are 0.1-0.2 mm across, discoidal and polished. They are set in a dominantly calcite matrix, with some chamosite.

| 104°77' | **Red brown limonite-coalite. Ooliths are difficult to distinguish from the matrix. They are 0.1-0.5 mm across, some are brown or red-brown and polished, others dull brown. The matrix is of limonite, chamosite and a fine red-brown material. The limonite and red-brown material forms small areas with very few ooliths. Occasional small patches of a fine grey material occur.**

| 105°3' | **Shell fragment coated red.**

| 105°75' | Fauna is present but not common. |
| 105.3" | Olive brown patch | Dark grey green rock with olive brown patches. It is chamosite rich. Dark grey fine-grained chamosite with a massive habit forms the matrix to:
Olive brown patch
Lense of fine grey material
Olive brown patch
Lense of lime-osite oolite
Lense of fine grey material |
<p>| 106&quot; | Calcite matrix Lense of fine grey matrix Lense of lime-osite oolite clast. |
| 106.1&quot; | Calcite matrix Lense of fine grey matrix Lense of lime-osite oolite clast. |
| 107.9&quot; | Shell fragment rich band |
| 108&quot; | Burrow infilled with fine grey material |
| 108.21&quot; | Dark grey green chamosite rock. The lower half is entirely of chamosite with a number of dull, relite, ooliths. The upper half is lighter in colour. Chamosite forms numerous discontinuous lenses. In between are dull and relite 0.5mm limonite ooliths set in a greenish white matrix. |</p>
<table>
<thead>
<tr>
<th>Page</th>
<th>Lithology of Frongham Ironstone Core Y 113</th>
</tr>
</thead>
<tbody>
<tr>
<td>59^</td>
<td>Shell band, thin lens of fine grey material</td>
</tr>
<tr>
<td>60^</td>
<td>Black markings become prominent.</td>
</tr>
<tr>
<td></td>
<td>Black to dark grey markings, sparse with branching and coalescing form. Oolith texture in places. Colour perhaps due to ooliths being of hematite.</td>
</tr>
<tr>
<td></td>
<td>Bivalve shells and fragments become infrequent.</td>
</tr>
<tr>
<td>61^</td>
<td>Burrows infilled with fine grey material.</td>
</tr>
<tr>
<td></td>
<td>Thin band of small ooliths burrows. Occasional black bivalve and oolithy valves.</td>
</tr>
<tr>
<td></td>
<td>Very small (1&quot;) lenses of grey fine-grained material occur. Bivalve fragments more common.</td>
</tr>
<tr>
<td>62^</td>
<td>Shell band, broken bivalves.</td>
</tr>
<tr>
<td></td>
<td>Occasional complete bivalves, not apparently in life position, with red ooliths. Small patches of black ooliths (hematite). More common bivalve fragments.</td>
</tr>
</tbody>
</table>

-132-
Grey fine grained material increases in amount from where red disappears. Rust coloured limonite ooliths richly cemented. Lime, the dominant component, correspondingly decreases.

Rust coloured limonite oolite forms lenses and stringers in dark grey material, in 80% rock.

Dark grey, fine grained material forms lenses and infills burrows. Greenish grey chamosite rich rock. Chamosite is present as fine ellipsoidal oolite. White material also forms a number of ooliths. These are set in a matrix of grey green fine grained chamosite.

Bivalve shell band. Lateral extensive portions of fine chamosite. Lenses like patches of brownish chamosite occur. Bivalve shells and fragments occur and form bands of shells in the life position.

Bivalve shell band

Bivalve shell band

Pyrite tubes. Greenish grey chamosite oolite. Ooliths 3-4 mm across and ellipsoidal. Calcite the dominant cement and may have iron in calcite. Ooliths may be absent, leaving disseminated chamosite. Brown pisoliths containing calcite bands 1-2 cm. Grey fine grained lenses. Large bivalves and bivalve fragments present. Massive, greenish grey fine grained chamosite rich rock. Occasional ooliths. Grey fine grained material forms small, irregular, lenses at the base of the unit. Shell fragments are common. Gradational base.

Small irregular lenses of fine grey material. Rusty brown limonite oolite. Ooliths are ellipsoidal, 3-4 mm across, with polished surfaces. These are set in a limonite matrix. Fine grained grey material forms irregular lenses varying from 1/2-1/4" thick, thinning to zero. Fauna is sparse. A shell lens occurs. This is rich in interstitial calcite. On occasion fine grey material is concentrated beneath "overturned" bivalve valves.

Small irregular lenses of fine grey material. Thick shell band of bivalves. Some complete P."
Small irregular lenses of fine grey material.

Brown limonite oolite. Ooliths are ellipsoidal, jam across; with polished surfaces. They are set in a calcite cement. Bivalve shells and fragments are present but not common. A dark grey to black fine grained material forms a lens at the base and contains a few limonite ooliths.

Irregular fine grain lens.

Olive green brown limonite oolite. Many irregular lenses of fine grey material and chamosite cores occur. The oolite and lenses each make up 5% unit. The core contains discoidal limonite forms lenses with polished surfaces. These are set in a matrix of either limonite or chamosite. The lenses are of a fine grained, generally non-oolitic, black/dark grey envelope around a fine grained green chamosite core with a few limonite ooliths. Chamosite nodules occur. Very little fauna. Brown limonite oolite with calcite cement.

Fine grey material and many shells.

Olive green brown limonite oolite. Ooliths are ellipsoidal, jam across; with polished surfaces. These are set in a matrix of limonite or fine grained green chamosite. Occasional thin lenses of a fine grey material occurs. Fauna is infrequent, containing small bivalve shells and a bolomphite. Both elements are coated with chamosite.

Fine grey material forming lens.

Belemnite with chamosite coat.

Limonite oolite. Ooliths are jam across. They are set in a matrix of limonite and calcite. A shell rich band of chamosite and fragmentary bivalves occur at the base.

Shell rich band lens with chamosite ooliths set in calcite matrix.

Olive green brown limonite oolite. Ooliths are discoidal, jam across; with polished surfaces. These are set in a matrix of limonite and green fine grained chamosite. Small patches with few ooliths, but rich in chamosite matrix occur. Near the top a lens containing 5%, chamosite ooliths set in a calcite matrix occurs. Occasional black fine grey short lenses and spots are present.
<table>
<thead>
<tr>
<th>Layer</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>71''</td>
<td>Limestone, calcite is present as broken shell fragments and coarse, crystalline calcite. Instostrially, limonite oolite occurs. Ooliths are discoidal, 1 cm across and set in a calcite matrix.</td>
</tr>
<tr>
<td>71'11''</td>
<td>Olive brown limonite oolite. Ooliths are discoidal, 1 cm across, with polished surfaces. The matrix is generally limonite but a chamosite matrix is not uncommon. A calcite-rich zone occurs at the base, rich in shell fragments with limonite ooliths in a calcite matrix. Occasional blue, fine-grained markings are present.</td>
</tr>
<tr>
<td>72'7''</td>
<td>Red-brown clasts shell-rich band. Limonite oolite. Ooliths are discoidal, 1 cm across, with polished surfaces. They are set in a calcite matrix. The upper third is rich in calcite, as shells and coarse calcite. Hematite streaks 1 cm across with red brown colour in this position also.</td>
</tr>
<tr>
<td>72'11''</td>
<td>Olive brown, massive, limonite rich rock. Oolite texture is poor, deformed. Ooliths are poorly formed, varying from discoidal to ellipsoidal. The majority are of limonite, but some are of chamosite. Chamosite forms thin, irregular veinslets within the rock. Occasional reddish calcite clasts 2-3 cm across occur. The matrix is generally limonite but some chamosite occurs. Bivalve fragments occur throughout but are not frequent.</td>
</tr>
</tbody>
</table>

[Cont. Figure 5.0]
<p>| 75.1 | Revalve shell band | Calcite rich rock. Calcite occurs as coarsely crystalline material around numerous shell fragments. This tends to form three bands between which limonite-oolite occur. The ooliths are ellipsoidal, 0.3 mm across and set in a limonite and calcite matrix. |
| 75.9 | Revalve shell band |
| 76.9 | Overall vertical colour change from olive brown at top to greyish green at base as chamosite content of matrix increases. Limonite-oolite. Ooliths are ellipsoidal and discoidal, 0.1-0.3 mm across, with polished surfaces. They are set in a matrix of chamosite and limonite. |
| 77.6 | Thin lens of fine grey material |
| 77.7 | Shell band |
| 77.8 | Shell band |
| 77.9 | Limonite-oolite clasts |
| 79.7 | Shell fragment band, rich in calcite. Matrix becomes entirely chamosite. Ooliths are discoidal, varying from 3 mm across with polished surfaces. These are set in a dominantly limonite matrix, although chamosite is the dominant matrix mineral in places. Greenish-grey fine-grained chamosite forms small irregular lenses and coats shells. Irregular, large, complete and fragmentary are scattered throughout the rock. Olive brown limonite-oolite. Ooliths vary in size from 0.1-0.5 mm and are ellipsoidal and discoidal in shape. These are set in a matrix of chamosite and limonite matrix. Occasional dark green, irregular, lenses of chamosite occur. These contain very few ooliths. |
| 79.9 | Thin lens of fine grey material |
| 79.91 | Limonite-oolite clasts |
| 79.1 | Cont. Figure 5.9 |
| Burrows infilled with fine grey material | Brownish grey limonite; ooliths are discoidal, varying in size from 0.2-0.5mm. Approximate equal amounts. A shell band with interstitial grey green chamosite occurs at the base. |
| Shell fragment | Olive brown limonite; ooliths are discoidal, 0.2-0.5mm across, with polished surfaces. They are set in a grey-green chamosite and calcite matrix. Small, thin, irregular lenses of a fine grey material occur. Shell fragments are scattered throughout. |
| | Greyish brown limonite; ooliths are brown, discoidal, 0.2-0.5mm across. They are set in a greenish grey chamosite matrix. Numerous fragmentary bivalve shells occur. |
| | Grey, shelly limestone. Coarse crystalline calcite occurs in the interstices. Occasional limonite ooliths and patches of light greyish-green chamosite occur. |
| Oocystropl. Limonite oolite clast. | Olive brown, coarse grained limonite; oolite, roughly spherical to pumice limonite ooliths, 1.5mm across. They are set in a chamosite, calcite and limonite matrix. Fine grained grey material also acts as a matrix. |
| | | Olive green limonite oolite. Ooliths are ellipsoidal, 0.2-0.4mm across. They are set in a generally chamosite matrix, with patches of limonite matrix. Thin lenses of a black-dark grey material run sub parallel to bedding and containing a little white mica. They are laterally discontinuous. |
| | Brownish grey limonite oolite. Ooliths are discoidal, 0.5mm across, with polished surfaces. In the upper half these are set in a calcite matrix. Patches of limonite matrix occur throughout. Occasional clasts of limonite oolite of limonite oolite matrix occur in the upper portion. Numerous complete and fragments of bivalves are present throughout. |
| Chamosite and calcite in equal amounts as matrix minerals. Melanite. Small red-brown clasts of Chamosite clasts. Complete, large bivalve, infilled with calcite. Overturned. | All coloured red due to ooliths being coloured red, and the presence of small red clasts. Chamosite and calcite make up equal amounts as matrix minerals. |</p>
<table>
<thead>
<tr>
<th>Layer</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>81'</td>
<td>Matrix entirely chamosite. Calcite disappears as a matrix mineral, chamosite the dominant matrix mineral.</td>
</tr>
<tr>
<td>83'1/4</td>
<td>Patches of red ooliths. Shell material is rare in the lower 5'.</td>
</tr>
<tr>
<td></td>
<td>Class or lenses of chamosite. Greyish green limonite oolite. Ooliths are ellipsoidal, 0.3-0.5mm across, and have polished surfaces. They are set in a fine-grained greyish green chamosite matrix. Near the top, lens-like or clasts of a fine grey-green chamosite occur. There are occasional thin, fine-grained grey lenses, the same material infilling a burrow. Shell fragments occur sparsely.</td>
</tr>
<tr>
<td>84'</td>
<td>Burrows infilled with either fine grey material or limonite. Olive brown limonite oolite. Ooliths are ellipsoidal, 0.3-0.5mm across with polished surfaces. They are set in a limonite matrix. Chamosite may also occur as a matrix mineral. Burrows are infilled with either fine grey material containing a few ooliths or limonite. With either bone, thin lenses of a fine-grained grey material occur. Limestone. Many shell or shell fragments in a matrix of coarse calcite in places. Limonite oolite occurs with ooliths set in a calcite and chamosite. Chamosite rich limonite oolite. The chamosite is green and fine-grained, occurring as splitting and coalescing lenses, 1-2cm thick at maximum, in between limonite oolite of ooliths set in chamosite occurs. Ooliths here are not found.</td>
</tr>
<tr>
<td>85'</td>
<td>Limonite pisoliths. Greyish brown, bioclastic rich, limonite oolite. Ooliths are ellipsoidal, 0.5mm across. They are filled in pits, fine-grained yellow material. These set in a matrix of calcite and calcite matrix. The chamosite component decreases towards the top. Occasional limonite pisoliths occur near the top. Occasional limonite is common. Coarse calcite is common in these shells and fragments are very common.</td>
</tr>
<tr>
<td></td>
<td>Limonite clast near the top. Occasional limonite clasts 5cm across occur in the lower portion. Coarse calcite is common between these.</td>
</tr>
<tr>
<td>85'3/4</td>
<td>Olive brown limonite oolite. Ooliths are ellipsoidal, 0.8-1.0mm across, and are set in a matrix and matrix mat. Brownish grey limonite oolite. Ooliths are ellipsoidal, 0.5-0.7mm across, and are set in a yellowish green limonite oolite. Ooliths are ellipsoidal, 0.5-0.7mm across, and are set in a matrix of matrix rich in calcite and calcite matrix. The rock is rich in bioclastic material throughout and has two layers particularly. Rich in shell and shell fragments.</td>
</tr>
<tr>
<td>86'</td>
<td>Shell rich band. Red brown limonite oolite. Ooliths are ellipsoidal, 0.5mm across, with polished surfaces. These are set in a matrix of matrix cement. A band rich in chamosite as a matrix occurs. There are abundant thin red marks. There are irregular branching bands of a fine reddish material. It may also set as matrix.</td>
</tr>
</tbody>
</table>

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Cont. Figure 5.0

Large bivalves in life-position-with
camouflage banked
up in them

Olive brown limonite oolite. Ooliths are
ellipsoidal, 0.5-0.3 mm across, with
polished surfaces. Shell material of the
same grade is coated in limonite. The
matrix is dominantly of chamosite with
subordinate occasional limonite. Fine
grained, dark green, chamosite containing
very few ooliths infills burrows and is
banked up against, and in, bivalve shells
in their "live" position.

Shell concentration
Chamosite in a
bivalve "cup"

Lens of fine
grey material

Brownish grey limonite oolite. Ooliths are
ellipsoidal, 0.3 mm across. They are set in a
dominantly coarse calcite matrix with patches of
in which the matrix is chamosite. The rock
is high in bioclastic components; complete
and fragments of shell material occurring
throughout. There are four bands where shell
material is concentrated. Occasional shells
in the "live" position contain chamosite.

Shell concentration
Chamosite in a
bivalve "cup"

Lens of fine
grey material

Olive brown limonite oolite. Ooliths are ellip-
soidal, 0.3 mm across. They are altered to
dull yellow material. The matrix is of a
greenish grey chamosite and minor limonite.
Patches of fine red brown material occur as
blobs and stringers. Little shell material.

39.5

Chamosite in bivalve "cup"

Brownish grey limonite oolite. Ooliths are dis-
coidal, 0.3 mm across. They are set in a calcite
matrix. Chamosite occurs in "live" position in
bivalve shells. Shells red brown lenses occur.

Lens of fine
grey material

Yellowish brown limonite oolite. Ooliths are
ellipsoidal, 0.3 mm across with polished surfaces.
The matrix is of chamosite. Very thin red brown
lines mark the rock. A fine grey lenses with a
flat base and irregular top occurs.

Cine grey material
Forming rounded
ions

The matrix is of calcite and chamosite. Fragments
shell material occurs.

29.51

Limonite pisoliths

Olive brown limonite oolite. Ooliths are ellip-
soidal, 0.2 mm across and often altered to a dull
yellow brown. Set in a limonite and chamosite
matrix. Lenses of chamosite poor in ooliths
occur. A shell band occurs generally. Large
limonite pisoliths with chamosite cortex and
ooliths tangential to the nuclei occur above this.

Shell band

Massive, fine greenish grey chamosite. This has
an irregular top. The rock contains ooliths
which are wedges, and are often altered
to dull yellow material.

Lens of fine
grey material

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The overall mineral assemblage remains constant, comprising calcite, siderite, limonite, chamosite. Calcite forms the dominant matrix mineral in a number of instances, and is also present as numerous shell fragments. Siderite occurs in dark grey lenses of siderite mudstone. Limonite forms ooliths and may be common in the matrix. Chamosite occurs in a massive form as chamosite mudstone, and as a dominant matrix mineral.

The detailed logs have been simplified into four lithologies as shown below:

A - limonite oolite with a dominantly calcite matrix,
B - chamosite mudstone,
C - limonite oolite having a dominantly chamosite matrix,
D - Bioclastic limestone.

The petrography of lithological types A, B, C and D is described below:

Lithological type A
Limonite ooliths, pisoliths of limonite, chamosite and siderite, several different intraclast types, and shell fragments are set more or less randomly in a cement of low-iron ferroan calcite. All coated with a thin film of chamosite.

Examination of the arrangement of the component allochemical grains shows that the larger shell fragments are oriented with their long axes parallel to bedding. Where ooliths are
in contact with such fragments they lie with their major axis parallel to the shell.

**Lithological type B**

This lithological type is a chamositic mudstone. Siderite, limonite and calcite also occur but are very subordinate. The overall colour of the lithological type is a dull greyish green. Allochemical grains are rather less common than in the lithological types A, but comprise ooliths, pisoliths and complete and fragmentary bivalve shells.

Two oolith types have been recognised:

1 - Limonite ooliths. These are characterised by a dull yellow colour.

2 - Chamosite ooliths. These ooliths are coloured greyish green.

The limonite ooliths are far more abundant than the chamosite ooliths. The shell material present is represented by barids consisting of shell fragments and complete, but disarticulated, shells. The dominant matrix mineral is fine grained chamosite. In addition to this, siderite - a dull white mineral - and pyrite also participate in the matrix. Where siderite acts as a matrix mineral, the rock is coloured a greyer shade of green and is much harder. In addition, to its matrix occurrence, siderite also forms dark grey lenses.

**Lithological type C**

Limonite ooliths, three different interclast types, shell fragments, diagenetic siderite grains, and detrital quartz
grains are set in a fine grained matrix of chamosite and limonite. Low iron ferroan calcite forms small patches of matrix. Ooliths may be oriented with their major axis parallel to bedding, but more commonly are not oriented with any preferred direction. Large shell fragments are oriented with their long axis parallel to bedding. Chamosite mud often forms thin discontinuous lenses in which quartz and siderite detrital grains may be common, but all other allochemical grains are absent. In other areas this distribution is reversed; quartz and siderite are uncommon whilst the other allochems are common.

**Lithological type D**

This is a bioclastic limestone, composed predominantly of complete and fragmentary bivalve shells set in a coarsely crystalline calcite matrix. The colour of this lithology varies from grey-brown to grey. A small proportion of the component allochems are limonite ooliths. These are discoidal in shape with polished surfaces and a grain size variation of 300 to 500 μm. In some instances the surfaces of the shell material may be coated with limonite. Lenses of dark grey siderite mudstone are occasionally present.

The most important points for Davies and Dixie (36) classification of the Frodingham ironstone and the present classification are described below in Table 5.4.
<table>
<thead>
<tr>
<th>Principal Types According to Davies and Dixie</th>
<th>Detritus</th>
<th>Matrix</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Limonite-chamosite ooliths (closely packed), calcite shells - all coated with a thin film of chamosite.</td>
<td>Dominantly granular siderite</td>
</tr>
<tr>
<td>B</td>
<td>-</td>
<td>Rhomb-shaped crystals of siderite distributed between linearly arranged flats of chamosite.</td>
</tr>
<tr>
<td>C</td>
<td>Limonite-chamosite ooliths, and calcite shells partly replaced by chamosite</td>
<td>Very fine-grained, of chamosite and siderite some quartz grains at times</td>
</tr>
<tr>
<td>D</td>
<td>Calcite shells, limonite-chamosite ooliths</td>
<td>Coarse crystalline calcite (some siderite and chamosite)</td>
</tr>
</tbody>
</table>

Present Classification

| A                                             | Limonite ooliths, pisoliths of limonite, chamosite and siderite. Calcite shell fragments. All coated with a thin film of chamosite | Low-iron ferroan calcite |
| B                                             | Chamosite and limonite ooliths. Calcite shells | Fine grained of chamosite, siderite and pyrite occur also in the matrix. Siderite lenses |
| C                                             | Chamosite limonite ooliths, calcite shell | Fine grained chamosite and limonite and low iron ferroan calcite forms small patches of matrix. Thin discontinuous lenses mainly quartz. Siderite grains occasionally present. |
| D                                             | Calcite shells, limonite ooliths. Shell material coated by limonite | Coarsely crystalline calcite. Lense of siderite mudstone are occasionally present. |
5.5.3 The Chemistry of the Ironstone

Since 1958 many boreholes have been drilled close to and to the east of the B.S.C. steel plants in Scunthorpe. The opencast and underground working ironstone core recovery from these boreholes is frequently between 95 and 100 percent.

As for the B.S.C. operations in South Lincolnshire, each foot (30 cm) of the ironstone cores, after drying was analysed for total iron, lime, silica, and sulphur; silica was determined as a perchloric acid insoluble. In this work the chemical analysis for phosphorus was completed and added to the above list (see Tables 5.2 and 5.3).

An average chemical analysis for the total thickness of the ironstone is as follows:

<table>
<thead>
<tr>
<th>Core</th>
<th>Y 112</th>
<th>Y 133</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>25.6%</td>
<td>24.6%</td>
</tr>
<tr>
<td>Lime</td>
<td>20.5%</td>
<td>21.2%</td>
</tr>
<tr>
<td>SiO₂</td>
<td>7.88%</td>
<td>8.58%</td>
</tr>
<tr>
<td>Sulphur</td>
<td>0.38%</td>
<td>0.43%</td>
</tr>
<tr>
<td>Phosphorus</td>
<td>0.33%</td>
<td>0.32%</td>
</tr>
<tr>
<td>Loss on ignition</td>
<td>24.2%</td>
<td>24.1%</td>
</tr>
</tbody>
</table>

5.5.3.1 Vertical chemical variation within the ironstones

In Tables 5.2 and 5.3 the chemical analyses are shown for two cores Y 112 and Y 113, the analyses being done for each foot (30 cm) from the top of the ironstone.

Vertical variation in chemistry, principally in terms of variation in Fe content and phosphorus content, are shown in Figures 5.10 and 5.11.
FIG. 5.10
DISTRIBUTION OF IRON & PHOSPHORUS CONTENT IN FRODINGHAM IRONSTONE
From the data shown in Tables 5.2 and 5.3 the upper type C is seen to contain generally higher iron values than the type D, and generally higher silica values occur in the upper part of the cores compared with the lower bed except where transition beds are found. Phosphorus shows a vertical variation in the two cores. It is irregular and often exhibits sharp variations.

From Table 5.2 the maximum iron content is 43.3% and the minimum 15.9%, lime content is between 36.3% and 6%, sulphur content is between 2.3% and 0.037%, phosphorus between 1.12% and 0.05%. From Table 5.3 the iron content is between 39.8% and 15.7%, lime between 35.9% and 6.2%, sulphur between 4.1% and 0.019% and phosphorus content is between 1.1% and 0.11%.

5.5.4 Result of X-ray diffraction mineralogical analyses

18 Frodingham ironstone samples and two concentrated ironstone samples were analysed by X-ray diffraction methods, but the two concentrated ironstone samples were subjected to the following treatment before X-ray analysis.

1 - Dry sample No. B100 was ground using pestle and mortar (uniform grinding times)

2 - Samples were analysed for their phosphorus content

3 - Dry sample powdered dissolved in 20% Acetic Acid to dissolve the calcite (37). After the reaction stopped, the filtered, dried powdered samples were concentrated by
high intensity magnetic separation, followed by the determination of the phosphorus content in the magnetic and non-magnetic product.

The magnetic sample product contained 4.55% P and the non-magnetic sample product contained 3.48% P. Both were analysed by X-ray diffraction.

X-ray analyses
Samples were examined on a diffractometer with Fe filtered, Co radiation, and Ni filtered, Cu radiation using operating potentials of 30 kV with a current of 30 mA, and 40 kV with 20 mA respectively. Goniometer scans from 3° to at least 60° were run at 1° 2θ per minute. Diffractograms were studied visually to identify as many mineral phases as possible.

Four mineral phases were identified within the two samples, Hematite, Apatite, Quartz and Clay mineral. Apatite was the major phase.

A typical X-ray diffractogram for one of the ironstone samples is shown in Figure 5.12. Seven mineral phases were identified by X-ray techniques within the ironstone.

The percentage of the phases in descending order were as follows: calcite, quartz, chamosite, hematite, siderite, apatite and chlorite.
Figure 5.12  Typical X-ray Diffractogram of One of Frodingham Ironstone Samples (Sample No. A 8)

\( \text{Co K}\alpha \)
5.5.5 Scanning Electron Microscopy

Examination of Frodingham ironstone by Stereoscan enables the mapping of elemental distributions for Fe, Si, P, Ca, and Al by employing electron beam scan techniques. This was done for 40 samples from different beds throughout the body of the ore. This technique showed the structure and the grain size of the iron oxides and the gangue minerals as may be seen in the example given below.

Figure 5.13 Scanning electron micrographs of Frodingham ironstone Sample No. B 100-2 Fe, Si, Al, P and Ca, X-ray distribution map. This figure shows that the ooliths were ellipsoidal in shape; the length of the major axis varied from 100 μm to 250 μm. Some of the ooliths were broken. They showed a concentric banding structure but in a number of cases this could not be seen. The matrix cemented the ooliths, and there were two types of matrix material—light, poorly polished and a smaller amount of darker, better polished material.

The Fe X-ray distribution shows a high concentration of Fe in the ooliths. Fe is also shown as very fine grains dispersed throughout the matrix.

The Si occurs finely disseminated in the matrix associated with moderately high concentration of Ca and P and low concentration of Fe and Al.

Al occurs within the matrix in low concentrations associated with Si, Fe, Ca and P.
Figure 5.13  Scanning Electron Micrograph of Frodingham Ironstone Sample No. B 100-2 and X-ray Distribution Maps x200

a - Scanning Electron Micrograph
b - Iron X-ray distribution map
c - Silicon X-ray distribution map
d - Aluminium X-ray distribution map
e - Phosphorus X-ray distribution map
f - Calcium X-ray distribution map
Fig. 5.13
The matrix contains high concentrations of Ca associated with P and low concentration of Si, Al and Fe. In addition, there are anhedral Ca grains in some ooliths not associated with P.

P occurs in the matrix in high concentrations associated with Ca, and smaller amounts of Al, Si and Fe.

Fe is shown:-

a - as high concentration in the ooliths. X-ray studies proved these to be hematite.

b - as low concentration associated with Ca, P, Al and Si in the matrix. From X-ray studies this was likely to be chamosite.

Si was associated with Al, Fe, Ca and P in the matrix. Proved from X-ray studies to be chamosite.

Ca is shown in:-

a - large calcite crystals and also as anhedral grains in some ooliths. X-ray studies proved these to be calcite.

b - associated with P, Al, Si and Fe in the matrix. From X-ray studies this was shown to be calcite with apatite.

P is shown in the matrix associated with Ca, Fe, Si and Al and, as previously, proved to be apatite. Matrix is apatite, chamosite and calcite intimately intergrown.
Figure 5.14: Scanning electron micrographs of Frodingham ironstone sample No. B 55, Fe, Si, Al, P, Ca and S. Ooliths are ellipsoidal in shape; the length of the major axis varies from 225 µm to 350 µm. The ooliths show a concentric banding structure. The matrix cements the ooliths but there are two types of matrix materials, light and dark both well polished.

The Fe X-ray distribution map shows a high concentration of Fe in the ooliths and in most of the light coloured matrix. It also demonstrated that the concentration of Fe was independent of the concentration of Si, Al, Ca or P. The Si occurred as discrete areas of high concentration in which it was associated with Al. The areas of high Si concentration are present in the outer rim surrounding the Fe rich ooliths. There are also very low concentrations of Si dispersed in the ooliths and the matrix.

Al is shown to be associated with Si in the outer rim of the ooliths, and as very low concentrations dispersed in the ooliths and the matrix.

Ca is shown in the matrix in high concentration as rims surrounding the ooliths and as discrete large areas. It is also shown as anhedral grains in the matrix associated with P; the length of the major axis is about 20 µm (top right).

P is shown to be associated with Ca in the matrix.
Figure 5.14  Scanning Electron Micrograph of Frodingham Ironstone
Sample No. B 55 and X-ray
Distribution maps x200

a - Scanning Electron Micrograph
b - Iron X-ray distribution map
c - Silicon X-ray distribution map
d - Aluminium X-ray distribution map
e - Phosphorus X-ray distribution map
f - Calcium X-ray distribution map
g - Sulphur X-ray distribution map
S, in high concentration, is found in some parts of the matrix associated with Fe.

The Fe is shown to be in:

a - the matrix associated with S. Petrographic studies showed this Fe to exist as pyrite.

b - the ooliths are rich in Fe (shown to be hematite).

Si shows in the outer rim of the ooliths associated with Al. Proved from X-ray studies to be clay minerals.

Ca shows:

a - in the matrix - shown to be calcite

b - as anhedral grains in the matrix associated with P. These proved to be apatite.

P is shown in the matrix as anhedral grains associated with Ca (apatite).

S is shown in the matrix associated with Fe (shown to be pyrite).

Figure 5.15: Scanning electron micrographs of Frodingham ironstone sample No. A.85, Fe, Si, Al, P and Ca, X-ray distribution map. The ooliths are ellipsoidal in shape: the length of the major axis varies from 180 μm to 340 μm. The matrix cements the ooliths. The Fe X-ray distribution map shows high concentration of Fe in the ooliths. The Fe in the matrix is seen in the right hand side of the photo-
Figure 5.15  Scanning Electron Micrograph of Frodingham Ironstone

Sample No. A 85, and X-ray Distribution maps, x200

a - Scanning Electron Micrograph
b - Iron X-ray distribution map

c - Silicon X-ray distribution map
d - Aluminium X-ray distribution map
e - Phosphorus X-ray distribution map
f - Calcium X-ray distribution map
micrograph in low concentrations associated with Si, Al, Ca and very little P.

The Si occurs in the matrix and the ooliths, the higher concentration being at the right hand side of the map. In the ooliths the Si is associated with Al and Fe. In the matrix it is associated with Al, Fe, Ca and very little P.

Al occurs in low concentration in the ooliths and the matrix. In both cases it is associated with Si and Fe. In the matrix, the left hand side of the map shows a higher concentration of Ca than the right hand side. Ca is also associated with Fe, Si, Al and a little P in the right hand part of the matrix.

P is shown as very low concentration mainly within the matrix.

Fe is shown in:
a - the right hand side of the matrix associated with Si, Al, Ca and little P. Shown from X-ray studies to be a mixture of siderite and chamosite.
b - ooliths associated with Si and Al (chamosite).

Si shown in:
a - the matrix associated with Al, Fe, Ca and little P. (chamosite).
b - ooliths associated with Fe and Al (chamosite).
Ca is shown in:

a - the left hand side of the matrix in high concentration (calcite).

b - the right hand side of the matrix associated with Fe, Si, Al, and a little P (shown to be calcite)

P is shown as low concentrations dispersed in the matrix which is mainly Ca (apatite).

5.5.6 Microprobe examination (Operating Conditions p. 204)

Microprobe analysis studies for the Frodingham ironstone made it possible to characterise the amount of impurities and the intergrowing of the useful and gangue components. In this work interest was in the phosphorus distribution and the study of the elements associated with the phosphorus and occurring with it in the ironstone. The number of samples investigated using this method was 40 samples.

By comparing the result from electroprobe analyses and X-ray examination it was possible to deduce the kind of phosphate minerals present and the minerals intergrowing or associated with it.

Using Microprobe analysis techniques, the above was done for samples from different beds throughout the core and this technique showed the structure and the grain size of iron oxides and the gangue minerals and the intergrowth between them may be seen in the examples given below.

Figure 5.16: Microprobe electron micrographs of Frodingham ironstone sample No. B.100-1, Fe, Si, Al, P and Ca X-ray distribution map.
The X-ray distribution shows that the ooliths are ellipsoidal in shape; the length of the major axis varies from 224 μm to 384 μm. The matrix is of Ca rich material. The Fe X-ray distribution map shows a high concentration of Fe in the ooliths and is associated with small amounts of low concentration Si, Al, P and Ca except for the grain in the bottom left of the map where its concentration is much higher.

In addition, there is a rim of high Fe concentration associated with Al and Si surrounding a shell fragment in the matrix (see extreme top centre and right of map).

The Si X-ray distribution shows a low concentration of Si in the ooliths associated with low concentrations of Al, P, Ca and Fe. It is also seen associated with Fe in the rim of the shell fragment in the matrix.

The Al X-ray distribution shows the same distribution as Si.

The matrix is of Ca rich materials which cement the ooliths and are not associated with the other elements.

Ca occurs as a subangular grain in an oolith (top of the micrograph). It has partially replaced the Fe in this oolith. Ca is also dispersed in low concentration in the ooliths.

In addition there is a shell fragment of high concentration of Ca in the matrix.
Figure 5.16  Microprobe Electron
Micrographs of Frodingham Ironstone
Sample No. B 100-1, x125

a - X-ray distribution map for Iron
b - X-ray distribution map for Silicon
c - X-ray distribution map for Aluminium
d - X-ray distribution map for Phosphorus
e - X-ray distribution map for Calcium
P is dispersed in the ooliths in low concentration and is associated with Al, Si, Fe and Ca.

Fe is shown in:

a - ooliths associated with Si, Al, P and Ca. This was shown from X-ray studies to be chamosite.

b - as a rim surrounding the shell fragment, associated with Si and Al, again shown to be chamosite.

Si shows:

a - dispersed in the ooliths associated with Fe, Al, P and Ca, and as previously proved to be chamosite.

b - in the matrix as fine particles and as a rim surrounding the shell fragment; in both cases associated with Al and Fe. Proved to be chamosite.

Al shows in:

a - ooliths associated with Si, Fe, Ca and P (chamosite)

b - the matrix associated with Si and Fe (chamosite)

Ca shows as matrix rich in Ca, and as shell fragment and also a partial replacement for Fe in an oolith; proved from X-ray studies to be calcite.

P is shown disseminated in the ooliths and associated with Fe, Si, Al and Ca. X-ray studies proved these to be apatite.

Figure 5.17: Microprobe electron micrographs of Frodingham ironstone sample No. B.9, Fe, Si, Al, P and Ca. X-ray distribution map.
In the upper part of the figure there are Fe ooliths which are ellipsoidal in shape; the length of the major axis varies from 150 μm to 350 μm.

Also there are two subrounded grains of Si for which the length of the major axis varies from 50 μm to 100 μm. In addition, there is a small amount of low concentration Si within the ooliths.

The matrix is mainly Ca in the upper part and a mixture of Fe, Si, Al and Ca in the lower part where the Fe and Si concentration appears to be higher than that of Al and Ca.

The Fe X-ray distribution shows a high concentration of Fe in the ooliths and in the lower part of the matrix, with a higher concentration in the oolith than in the matrix of the bottom half.

The Si occurs as subrounded grains in the upper part of the micrograph. Within the matrix, it occurs in high concentration in the lower parts. In addition it is seen within one oolith in low concentration (middle left hand of the micrograph).

Al is shown within the matrix in low concentration and is associated with Si, Fe and Ca. It is also shown within the ooliths in small amounts of low concentration associated with Fe and a small amount of low concentration Si.

In the upper part of the micrograph the matrix contains a high concentration of Ca which is not associated with the
Figure 5.17  Microprobe Electron
Micrographs of Frodingham Ironstone
Sample No. B 9, x100

a - X-ray distribution map for Iron
b - X-ray distribution map for Silicon
c - X-ray distribution map for Aluminium
d - X-ray distribution map for Phosphorus
e - X-ray distribution map for Calcium
other elements. In the lower part the matrix contains less Ca which is associated with Fe, Si and Al.

P occurs in the ooliths and within the matrix in very small amounts of low concentration.

Fe is shown:

a - in high concentration within the ooliths associated with small amounts of low concentration of Si, Al, P and Ca. From X-ray and petrographic studies these proved to be hematite.

b - in high concentration associated with Si, Al and Ca in the matrix. From X-ray studies this was likely to be chamosite.

Si shows:

a - as subrounded grains of high concentration not associated with other elements. Proved from X-ray studies to be quartz.

b - as small amounts of low concentration in the ooliths associated with Al and Fe. From X-ray studies this was shown to be clay minerals.

c - as high concentration in the lower part associated with Al, Fe and Ca (chamosite) and other clay minerals.

Al is shown:

a - as small amount of low concentration in the ooliths associated with Fe and Si; as previously proved to be clay minerals.
b - in the lower part within the matrix associated with Si, Fe and Ca; as previously proved to be chamosite and other clay minerals.

Ca is shown:

a - in high concentration in the matrix. From X-ray studies proved to be calcite.

b - within the matrix associated with Si, Al and Fe. Proved to be calcite.

P is shown as a very little amount of low concentration in the ooliths associated with small amounts of low concentration Ca, Al and Si, and high concentrations of Fe. From X-ray studies this was likely to be apatite.

In the upper part the matrix is mainly Ca in high concentration and in the lower part it is a mixture of high concentrations of Fe and Si with low concentration of Al and Ca. From X-ray studies this was proved to be calcite in the upper part and a mixture of chamosite, calcite and clay minerals in the lower parts.

Figure 5.18: Microprobe electron micrographs of Frodingham ironstone sample No. B.49, Fe, Si, P and Ca, X-ray distribution map.

Ooliths are ellipsoidal in shape; the length of the major axis varies from 250 \( \mu m \) to 450 \( \mu m \). In several ooliths the
nuclei are of an element different than the surroundings. These subsequently appear as subrounded grains in the cores of the ooliths.

The matrix is Ca rich. The Fe X-ray distribution map shows high concentration of Fe in the ooliths. It also shows that Fe concentration is independent of the concentration of Si, Ca or P.

The Si X-ray distribution shows high concentrations of Si associated with Fe in the rim surrounding the ooliths. The Si is also seen in high concentration in the matrix associated with Fe in the right hand side of the micrograph.

P occurs as high concentration of anhedral grains associated with Ca in the ooliths; the length of the major axis varies from 10 µm to 70 µm. It is also seen disseminated in the ooliths in low concentration.

Ca occurs as a matrix of high concentration and is not associated with the other elements. Also there are anhedral Ca grains associated with P in the ooliths.

Fe is shown:

a - in high concentration within the ooliths associated with small amounts of Si, P and Ca. From X-ray studies proved these to be hematite.

b - in some areas of the matrix associated with Si. From X-ray studies this was shown to be composed of clay minerals.
Figure 5.18  Microprobe Electron

Micrographs of Frodingham Ironstone

Sample No. B 49, x100

a - X-ray distribution map for Iron
b - X-ray distribution map for Silicon
c - X-ray distribution map for Phosphorus
d - X-ray distribution map for Calcium
Si is shown in:

a - a rim of high concentration surrounding the ooliths and associated with Fe. These were proved to be clay minerals.

b - in some areas of the matrix associated with Fe (clay minerals).

P is shown in one of the ooliths as a subrounded grain in the centre and also as anhedral grains in other ooliths. In all cases it was associated with Ca. X-ray studies proved these to be apatite.

Ca is shown in:

a - the matrix not associated with the other elements. From X-ray studies proved this to be calcite.

b - the oolith's associated with P (apatite)

c - the ooliths as anhedral grains not associated with other element (calcite).

**Figure 5.19**

This figure illustrates part of the same area shown in Figure 5.18 but at a magnification of x250 compared with x100 in Figure 5.18. The main feature which has been highlighted is the elliptical oolith sited bottom centre in the previous figure.

The matrix is of Ca rich material and is not associated with the other elements. Fe X-ray distribution shows high concentration of Fe in the oolith associated with small amount of Si, P and Ca.
Figure 5.19  Microprobe Electron

Micrographs of Frodingham Ironstone

Sample No. 49, x250

a - X-ray distribution map for Iron
b - X-ray distribution map for Silicon
c - X-ray distribution map for Phosphorus
d - X-ray distribution map for Calcium
The Si X-ray distribution shows a relatively higher concentration of Si associated with Fe in a rim surrounding the oolith. The Si is also seen disseminated in the oolith in lower concentration. In the core of the oolith is a subrounded grain containing moderately high concentration of P and Ca associated with each other; the length of the major axis is about 72 μm. Also there are anhedral grains of high concentration of P and Ca associated with each other in the oolith; the length of their major axes varies from 8 μm to 28 μm.

Fe shows in the Fe rich oolith associated with Si, P and Ca (shown to be hematite).

Si shows:

a - as a rim surrounding the oolith associated with Fe.
   Proved from X-ray studies to be clay minerals.

b - in the oolith associated with Fe, P and Ca (clay minerals).

P as indicated above appears as anhedral grains in the oolith (apatite).

Ca shows in:

a - the matrix not associated with other elements (calcite).

b - in the oolith associated with P (apatite).
5.5.7 Summary of Results

The Frodingham ironstone bed is situated in North Lincolnshire - east of Scunthorpe. It occurs in the lower Jurassic bed of the South Humberside area.

From the logging of the cores in Figures 5.8 and 5.9 it is clear that calcite forms the dominant matrix mineral in most of the samples, and is also present as numerous shell fragments. Siderite occurs in dark grey lenses of Siderite mudstone. Limonite forms ooliths and may be common in the matrix. Chamosite occurs in a massive form as chamosite mudstone, and as a dominant matrix mineral. The detailed logs have been simplified into four lithologies as shown below:

A. Limonite ooliths with a calcite-dominant matrix,

B. chamosite mudstone,

C. limonite ooliths having a matrix dominated by chamosite,

D. limonite ooliths and calcite shells, more or less replaced by siderite, are set in calcite matrix.

The lithology shows that most of the cores are made up of type C and type D.

The average chemical analyses for the total thickness of the ironstone are as follows:
<table>
<thead>
<tr>
<th></th>
<th>Core Y112</th>
<th></th>
<th>Y113</th>
<th></th>
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</thead>
<tbody>
<tr>
<td>Fe</td>
<td>25.6%</td>
<td></td>
<td>24.6%</td>
<td></td>
</tr>
<tr>
<td>Lime</td>
<td>20.5%</td>
<td></td>
<td>21.2%</td>
<td></td>
</tr>
<tr>
<td>SiO₂</td>
<td>7.88%</td>
<td></td>
<td>8.58%</td>
<td></td>
</tr>
<tr>
<td>Sulphur</td>
<td>0.38%</td>
<td></td>
<td>0.43%</td>
<td></td>
</tr>
<tr>
<td>Phosphorus</td>
<td>0.33%</td>
<td></td>
<td>0.32%</td>
<td></td>
</tr>
<tr>
<td>Loss on ignition</td>
<td>24.25%</td>
<td></td>
<td>24.15%</td>
<td></td>
</tr>
</tbody>
</table>

**Vertical chemical variation within the ironstone**

<table>
<thead>
<tr>
<th></th>
<th>Core Y112</th>
<th></th>
<th>Y113</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>15.9-43.3%</td>
<td></td>
<td>15.7-39.8%</td>
<td></td>
</tr>
<tr>
<td>Lime</td>
<td>6-36.3%</td>
<td></td>
<td>6.2-35.9%</td>
<td></td>
</tr>
<tr>
<td>Sulphur</td>
<td>0.037-2.3%</td>
<td></td>
<td>0.019-4.1%</td>
<td></td>
</tr>
<tr>
<td>Phosphorus</td>
<td>0.05-1.11%</td>
<td></td>
<td>0.11-1.1%</td>
<td></td>
</tr>
</tbody>
</table>

X-ray analyses revealed the presence of calcite, chamosite, apatite, quartz, hematite, siderite, chlorite and clay minerals. Wedd (55), Whitehead et al (35), and Davies and Dixie (36) mentioned that the phosphate mineral in Frodingham ironstone was collophane, Hallimond et al (56) suggested it was titanophosphates. But this study proved for the first time that it is apatite, by using new techniques (X-ray diffraction and scanning electron microscope) for the examination of this ore.

* The total number of X-ray samples examined were 18 in number & Co Kα & CuKα radiation were used on each sample (Section 5.54 & fig. 5.12)
6.0 Dephosphorisation of Iron Ores

Modern high speed basic oxygen steelmaking plants are
inconvenienced by the presence of phosphorus in the hot
metal to be refined. Although lime blowing and double
slag techniques can overcome the chemical problems of
phosphorus removal it would be advantageous if this
impurity was removed from the ore before it was charged
to the blast furnace. In fact, the Japanese ore buyers in
Australia have put a maximum of 0.07% P in the specification
for ore supplies in order to eliminate the double slag proces-
sing of hot metal. Many methods have been suggested for this
elimination including: Chemical processing (38,39,40,41,42,43),
Roasting followed by leaching with dilute acid or alkali (44,45),
Flotation (46) and Oil agglomeration methods (47,48,49).

6.1 Chemical Processing

6.1.1 Nitric Acid

Lucia and Losada (43) treated an oolitic magnetite ore
concentrate containing 0.3 to 0.5% P with (35-75 g/litre)
nitric acid using a 25% pulp density. They found that up
to 95% of the phosphorus present as apatite was dissolved
at ambient temperatures with only 1.5% iron loss in solution.
They presented the process from a purely technical point of
view, but it seems that the major cost is the acid supply
and maintenance because this acid can cause severe corrosion
to the steel structure.

6.1.2 Sodium Carbonate

This process of Mueller et al (40) consisted of washing an
iron oxide concentrate with a 10% sodium carbonate solution
at slightly elevated temperatures (up to 70°C) followed by washing the solids with water. Using a Brazilian hematite, the phosphorus content was reduced from 0.16 to 0.05% and sulphur from 0.052 to 0.015%. It would seem that the high temperature of operation would cause too large an energy and water input to make this process economic, although for a high value metal such as copper it is an economic proposition.

6.1.3 Flox Process

Reeve (39) studied the possibility of concentrating the low grade ores used at Appleby-Frodingham to raise their iron content and reject as much as possible of their gangue content. He described a cyclic chemical process for treating these ores, using hydrochloric acid and volatilising the iron as ferric chloride at 300-350°C. All other major constituents, including sulphur and phosphorus, remained in the solids. The ferric chloride so produced was hydrolysed and converted at 550°C to pure ferric oxide, with the regeneration of HCl gas for recycling. The process described was most conveniently carried out on finely crushed ore, fluidized in a stream of reactant gases. The process only just failed to be economic due to the high cost of the make up hydrochloric acid and high temperature of reaction although producing very pure oxide.

SEE ADDENDUM (p.205)

6.2 Roasting and Leaching with Dilute Acid or Alkali (Amdephos)

There are in the Pilbara region in the north west of Western Australia large tonnages of iron ore with a phosphorus concentration of about 0.15%.
For the ores studied by the Amdel Iron Ore Mining Company, optical and microchemical methods failed to detect phosphate minerals in polished sections of the ore, which consisted of grains of martite (Fe₂O₃) scattered through very fine-grained goethite, with some cavities containing clay. By electronprobe microanalysis, however, one grain of calcium phosphate mineral less than 10 μm surrounded by clay was detected in one specimen, and examination of another specimen revealed that some patches of goethite contained small amounts of phosphorus estimated to be between 0.1% and 0.5% and rarely up to 1%. This was not concentrated in definite mineral grains but was dispersed throughout the goethite, and was probably adsorbed by hydrated iron oxide from phosphate bearing solutions. It was not uniformly distributed and much of the goethite showed no phosphorus above background interference. Phosphorus was not detected in any grains of martite or hematite.

In the process developed on a laboratory scale known as "Amdephos", heat treatment (1200-1250°C) caused the P to segregate without the use of any additives and it was then extracted by leaching with dilute acid (0.5 N H₂SO₄) or alkali (0.5 N NaOH) at 90°C. The phosphorus content was reduced from 0.15% P to 0.035% P.

A cost study was made for a plant treating 1 x 10⁶ tonne/a pellets and it indicated that the major cost was for acid supply. Hence, with acid regeneration total treatment cost became 50 C (20p) per tonne of product. This cost for
Amdephos process was most likely to be economic without the inclusion of costs for the roasting process. They suggested that this process could be partially or wholly debited to another operation, e.g. the firing of pellets which is necessary for their induration, as well as for phosphorus solubilisation. In the case of a goethitic lump ore for which heating results in a considerable concentration of the iron content, the cost of firing could be shared between upgrading and dephosphorisation.

A more detailed discussion of the Amdephos process has been given by Gooden et al (44).

6.3 Flotation
Bahr (46) studied the dephosphorisation of the Mancorvo iron ore deposit in Portugal by flotation. Tests were carried out with a sample of hematite ore ground to 95% below 80 μm. The contents of a sample which was deslimed in a hydrocyclone at approximately 10 μm, were 45.7% Fe and 0.46% P. A two stage flotation process was used and the yield from the first was phosphate mineral and from the second hematite. The P yield was 73% and Fe yield in the P concentrate of 11%. The phosphorus content in the Fe concentrate was 0.2% P. It was noticed that the incomplete flotation of the phosphate minerals could have been due to extremely fine intergrowths of the phosphate minerals with hematite. This process did not produce a sufficiently low phosphorus content in the final product to be feasible.
6.4 Beneficiation of Phosphoric Iron Ore by Oil Phase Agglomeration Methods (47, 48)

The Snake River iron ore deposit, in the northern Yukon, Canada, was estimated to contain some 30,000 million tonnes of ore; with chemical analysis as follows:

\[
\begin{array}{ccccccc}
\text{Fe} & \text{SiO}_2 & \text{P} & \text{CaO} & \text{Al}_2\text{O}_3 & \text{TiO}_2 & \text{S} \\
\text{wt}\% & 44 & 30 & 0.34 & 3.0 & 1.4 & 0.10 & 0.02 \\
\end{array}
\]

The ore body consisted mainly of microcrystalline hematite, and chalcedonic quartz in several varieties of aggregation. The hematite was most commonly in the form of irregularly bonded interlocking crystals ranging in size from 5 to 200 \(\mu\text{m}\) and averaging 30 \(\mu\text{m}\). The phosphorus-containing minerals were apatite and collophane. The gangue minerals were finely disseminated and occurred in particle size down to 4 \(\mu\text{m}\). Selective agglomeration of the phosphorus minerals was carried out in the ball mill by the addition of fatty acids, such as oleic and linoleic, during the initial wet fine grinding to -400 mesh (-38 \(\mu\text{m}\)) at a pulp density of 70%. The ground ore was then further treated to selectively agglomerate the iron minerals which were separated from the remaining gangue minerals. Successful beneficiation was achieved on both the crude iron ore and jig concentrate (54.6% Fe and 0.39% P) with some concentrates assaying 65.9% Fe and less than 0.02% P.
7.0 Possible Application of Dephosphorisation Techniques to Bahariya, Aswan and Frodingham Ores

7.1 Bahariya Ore

This ore consists mainly of hematite and goethite with pockets of ochre and manganese oxides. The gangue minerals are halite, gypsum, barytes, quartz and calcite, with the following chemical analysis:

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<table>
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<tbody>
<tr>
<td>Fe content is between</td>
<td>49.8 to 63.2%</td>
</tr>
<tr>
<td>Manganese oxides</td>
<td>0.16 - 1.61%</td>
</tr>
<tr>
<td>Sodium Chloride</td>
<td>Trace - 6.61%</td>
</tr>
<tr>
<td>Silica</td>
<td>0.7 - 2.1%</td>
</tr>
<tr>
<td>Phosphorus</td>
<td>0.14 - 0.34%</td>
</tr>
</tbody>
</table>

The chemical and reduction processing of this ore is complicated by the presence of halite which causes more difficulties in the smelting than does the phosphorus content which is in the lower medium range.

From the microprobe and scanning electron microscopic examination the phosphorus is below detection concentrations mainly because of the very fine particle sizes (range ~5 μm) and the even distribution throughout the ore. Removal of such material requires very fine grinding similar to or finer than the Yukon ore to expose one of the facets of the gangue mineral to enable it to be contacted by a solvent during a leaching operation. There could be an advantage to be gained from the presence of chloride ions in a leaching process if they are in sufficient quantity to reduce the pH
of the water solvent to act as a hydrochloric acid solution. Such a process as acid process could then be considered but leaching with alkalis, e.g. sodium carbonate, would be too expensive due to the loss of alkali with the chloride present, but the presence of chloride suggests acid process as the possible beneficiation technique. However, for the main ores of this type another leaching solvent may be more appropriate than water, e.g. acid or alkali dependent upon the gangue content.

The great advantage of the processes in the case of Bahariya iron ore would be the simultaneous removal of sodium chloride from the ore.

It should be noted that the Bahariya iron ore is a Bilbao type (1), i.e. it is similar to:

- Cumberland in Great Britain;
- Bilbao in Spain;
- Erzberg, Austria;
- Ouenza, Bou kadra and other deposits in Algeria;
- Djerissa, Douaria and Tamera in Tunisia.

The Bahariya iron ore is presented in this thesis as an example of Bilbao type.

7.2 Aswan Iron Ore

From the chemical analyses of the Aswan Cores it is seen that the first core contained 42-55% Fe and 0.1-2.9% P, and the second analyses 41-57% Fe and 0.35-2.3% P. X-ray
examination showed the iron present as hematite with variable quantities of quartz and apatite but only a small amount of clay minerals. Scanning electron micrographs (Figs. 4.13, 4.14 and 4.17-4.21) showed the iron bearing ooliths measuring 75-750 μm in length with quartz crystals of sizes in range 40 to 220 μm although the majority are less than 100 μm. The apatite crystals were much smaller, all -80 μm and mostly <60 μm. From the micrographs in Figs. 4.13-4.15, 4.18, 4.19 and 4.21 it can be deduced that apatite occurs in the oolitic iron ore

(i) in the hematite matrix
(ii) in the hematite matrix surrounding the ooliths and the quartz grains
(iii) filling in the pores in the oolithic surfaces
(iv) as matrix cementing the ooliths with the hematite grains.

In the sandstone and clay beds the chemical analysis showed only 1-8% Fe and P 0.04-0.14%. The Microscan showed that quartz crystals up to 90 μm size were found in a chamositic matrix with small apatite crystals <25 μm in the chamosite cementing the quartz grains.

It would be seen that iron oxides and chamosite adsorb apatite crystals. Cook and McElhinny (10) suggested that most of the phosphorus is associated with the iron in an adsorbed form. Barbour (50) studied the distribution of phosphorus in the iron ore deposits of Itabira, Minas Gerais, Brazil. This study indicated that iron hydroxides were able to fix phosphorus.
Stout (51), Toth (52) and Colman (53) suggested that the fixation of phosphorus by hydrated oxides of iron and aluminium was a simple replacement or change of phosphate by hydroxyl ions. The results of Emerson and Widmer (54) from electron microprobe investigations for a sample collected from sedimentary core taken in Greifensee, Switzerland, showed a strong correlation between iron and phosphorus. These results are further evidence to support the idea that phosphorus is associated with the iron in an adsorbed form.

Figures 4.13, 4.14 and 4.17-4.21 show that most of the apatite occurs in the matrix or as matrix (Fig. 4.21). The amount of apatite which occurs in the ooliths is very small (about 5%).

Table 7.1 shows the size distribution of quartz, apatite and clay mineral grains in the ooliths and in the matrix for oolitic iron ore samples which were taken to represent the two cores in the Aswan area. It also shows that the liberation size for the apatite grains is about 20 μm but in many cases apatite grains make up aggregates up to 150 μm (Fig. 4.14) or 80 μm (Fig. 4.15). In this case it is suggested that the size liberation be ~80 μm. It was mentioned earlier that Bahr (46) had used flotation to separate the phosphate mineral from the very finely ground Portuguese Moncorvo iron ore (95% <80 μm). A 73% P yield was achieved. Poor results were due to the extremely fine intergrowth of the phosphate with hematite.
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<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>As10</td>
<td>50-100</td>
<td>Very fine</td>
<td>30</td>
<td>75-250</td>
<td>60-220</td>
<td>20-60</td>
<td>Fine</td>
<td></td>
</tr>
<tr>
<td>As9</td>
<td>60</td>
<td></td>
<td>20</td>
<td></td>
<td>25-150</td>
<td>25-65 aggregate to 150</td>
<td>H.Mat.</td>
<td></td>
</tr>
<tr>
<td>As25</td>
<td>10</td>
<td>120-160</td>
<td>5-25</td>
<td>750</td>
<td>80-300</td>
<td>10</td>
<td>Chamosite Mat.</td>
<td></td>
</tr>
<tr>
<td>As6</td>
<td>10</td>
<td>30-50</td>
<td>5-20</td>
<td>90-290</td>
<td>40-160</td>
<td>Irregular patches</td>
<td>H.Mat.</td>
<td></td>
</tr>
<tr>
<td>As9-2</td>
<td>10</td>
<td>30-50</td>
<td>5-20</td>
<td>90-290</td>
<td>80</td>
<td>20-60</td>
<td>H.Mat.</td>
<td>Little</td>
</tr>
<tr>
<td>As11</td>
<td>10</td>
<td>30-50</td>
<td>5-20</td>
<td>90-290</td>
<td>5-60</td>
<td>As matrix</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
From the Aswan ore results it can be seen that there are no very small grains of apatite intergrown with the hematite and only in one case, Fig. 4.21, are there hematite grains (4-60 μm) small enough to interfere with the apatite. Thus, it would seem that flotation concentration could be applied to the removal of apatite from Aswan ore crushed and ground to <80 μm, with an expectation of 90% P removal.

In the earlier classification of iron ores it was indicated that the Aswan ore was similar to ores from Lung-yen (Chahar Province) in China and Sierra Grande (Rio Negro, Patagonia in Argentina).

7.3 Frodingham Ironstone

The results of the electron microprobe and scanning electron microscope investigation for the distribution of phosphorus in the Frodingham ironstone are shown below:

1. Apatite occurs as very fine grains (<5 μm) and intergrown with the hematite grains in the hematite ooliths (100-450 μm).

2. It is also found as very fine grains (again <5 μm) intergrown with chamosite grains in the chamosite ooliths (Figure 5.15 and 5.16).
3. Apatite occurs as matrix of fine grains (as above) intergrown with chamosite and calcite grains (Figure 5.13), and in the lower part of Figure 5.17. It is also seen intergrown with chamosite grains only.

4. Occurring as anhedral and subrounded grains in the ooliths (8-28 μm).

It would seem from this study that the apatite is associated with the hematite and chamosite in an adsorbed form, and the apatite occurs in fine grains which are located in the pores in the ooliths. It was also found that the apatite was in the chamosite and hematite ooliths or in the chamosite matrix more than it occurred in calcite matrix, as shown in Figures 5.17, 5.18 and 5.19.

Berner (57) and Serruya (58) noticed that the concentration of dissolved reactive phosphate was significantly lower in sediments containing high weight percentages of calcium carbonate than in those of lower calcium carbonate percentages. The present results confirmed that the phosphorus was more likely to be present in a chamosite matrix than in a calcite matrix.

Electron microprobe results on phosphorus by Emerson and Widmer (54) for a sample collected from sedimentary cores in Greifensee, Switzerland, of P-rich particle showed a strong correlation between iron and phosphorus. They showed superimposed iron and phosphorus electron microprobe line diagrams for the same sediment particle.
Emerson and Widmer (54) results were identical to the results of our investigation on Frodingham ironstone for one oolith by electron microprobe analysis. The present investigation which was carried out on ooliths from different samples showed superimposed phosphorus and iron electron microprobe line diagrams (Figure 7.1). This result would appear to support the theory that phosphorus or apatite is associated with iron oxides in an adsorbed form. It was also confirmed that the major phosphorus mineral in the Frodingham ironstone was apatite.

In an attempt to remove phosphorus from Frodingham ironstone some preliminary investigations have been carried out previously on wet washing methods but the results were not sufficiently promising (33).

Tests were made on Frodingham ironstone by kiln roasting followed by magnetic separation. Reeve (33) found that iron recoveries as high as 93% were attained with percentage iron in the tailings below 6% and the iron percentage in the concentrate was 37% on average. No phosphorus analysis was given in Reeves (33) results but it seemed that the phosphorus percentage was increasing in the concentrate because the phosphorus was intermeshed in fine grains by hematite or chamosite which was removed in the concentrate, while the amount of phosphorus which occurred in the calcite matrix was negligible as in Figure 5.19 and the none magnetic phase was mainly calcite. The liberation size he used for the feed for magnetic separation was ~800 μm which was much too large to achieve the liberation of the phosphorus bearing grains (5-72 μm).
FIG. 7.1
SUPERIMPOSED IRON & PHOSPHORUS ELECTRON MICRO-
PROBE LINE DIAGRAMS FOR THE SAME OOLITH
As mentioned before in the section 6.1.3, Reeve (39) treated Frodingham ironstone using hydrochloric acid and volatilisation of the iron as ferric chloride at 300-350°C, while all other gangue minerals including phosphorus minerals remained in the solids. The ferric chloride so produced was hydrolysed and converted at 550°C to produce pure ferric oxide.

Dephosphorisation studies on the calcareous iron ores of the Big Seam from the Birmingham district of Alabama (45) included a reduction roasting operation followed by magnetic separation. The iron ore analysis was 36% Fe, 13% CaO and 0.33% P. From the data given neither reduction roasting nor magnetic separation of the reduced ore ground to minus 38 μm effectively achieved liberation of the phosphorus and subsequent rejection from the iron concentrate. An additional test was made to determine the effect of grinding much finer than 38 μm. In this case the reduced ore was ground to a product containing less than 13 percent by weight of material coarser than 20 microns. Magnetic separation of this material yielded a concentrate with improved phosphorus rejection but recovery of iron was significantly reduced. This magnetic concentrate analysed 61.1% Fe, 3% CaO, 0.19% P and 8.3% insoluble. The authors suggested that some additional treatment such as acid leaching would be required for more effective reduction of the phosphorus content. The additional treatment (acid leaching) showed that the acid requirements for effective reduction of the phosphorus content were more than the
stoichiometric requirement for the CaO reaction. It also showed that essentially all of the CaO must be made soluble before the acids reacted with the phosphorus bearing compound. Test results showed that the reduction roasting followed by fine grinding, magnetic separation, acid leaching, filtration and washing processes yield iron concentrates containing 62% Fe and 0.04% phosphorus, with iron recoveries of 82 to 90% and phosphorus extraction of 90 to 93%. However, acid requirements were high.

It is suggested that two processes are possible for the dephosphorization Frodingham ironstone:

1. Flox process (Section 6.1.3)

2. Reduction Roasting followed by fine grinding, magnetic separation, acid leaching, filtration and washing.

A detailed economic analysis is required for both processes to assess the feasibility of these processes but it would seem that they must both be uneconomic under present day iron ore costs.

Frodingham ironstone is a Minette, marine sedimentary type of ore and is similar to the following ores:

Lorraine Basin, France,
Luxembourg,
Salzgitter type Sidiments,
Federal Republic of Germany,
and hence it would be reasonable to assume the results presented may be applicable to the dephosphorisation of these and similar ores.
8.0 CONCLUSIONS

The techniques developed in this research were used to investigate the physical state of phosphorus found in Bahariya, Aswan and Frodingham iron ores. These are similar to many iron ore deposits throughout the world, and because of this, the benefits to be derived from ore dephosphorisation are self evident. The techniques used in this study were mainly geological:- Lithology, Mineralogy, Microscopy, X-ray analysis, Electron micro-probe analysis and Scanning electron microscope.

8.1 Bahariya Ore

It was shown that this ore:

1. is brown or yellowish brown in colour and consists mainly of hematite and goethite. The gangue minerals are halite, gypsum, barytes, quartz and calcite; with an iron content between 49.8 to 63.2% and phosphorus 0.14 to 0.34%.

2. The phosphorus occurs as very fine particles of apatite (-5 μm) and is distributed throughout the ore.

3. Removal of the phosphorus would require fine grinding (-40 μm) to expose the phosphate mineral so that it can be attacked by a solvent as for acid leaching.

8.2 Aswan Iron Ore

1. This is an oolitic iron ore of enriched Minette type, occurring as bands in the middle group of the Nubian series. The iron ore bands are at the surface or close thereby with iron contents between 41-57% and
phosphorus 0.1-2.9%. X-ray examination showed hematite with variable quantities of quartz, apatite and small amount of clay minerals. Scanning electron micrographs show hematite ooliths (75-750 μm), quartz crystals (40-220 μm), and the apatite crystals were -80 μm.

2. In the oolithic iron ore beds apatite occurs in many phases but usually concentrated in one or two of them never throughout the ore cross sections examined. Thus, it appeared in the hematite matrix; in the hematite matrix surrounding the ooliths and the quartz grains; filling in the pores in the oolithic surfaces; and as matrix cementing the ooliths with the hematite grains.

3. In the sandstone and clay beds, the chemical analysis showed 1-8% Fe and 0.04-0.14% P; quartz crystals up to 90 μm size were found in a chamositic matrix with apatite crystals <25 μm.

4. The distribution of the apatite was mainly in the matrix or as matrix; while the amount of apatite distribution in the ooliths is very small (about 5%).

5. The liberation size for the apatite grains is about 20 μm but in many cases apatite grains made up aggregates of 150 μm or 80 μm. From this study, it is suggested that the liberation size would be -80 μm.

6. Flotation concentration could be applied to the removal of apatite from Aswan ore crushed and ground to -80 μm.
8.3 Frodingham Ironstone

1. Occurs in the lower Jurassic bed of South Humberside.

2. Detailed logs have been simplified into four lithologies A, B, C and D. The lithology shows that most of the cores are made up of type C and type D.

3. The average iron content is 25.1% and the phosphorus 0.32%. X-ray analysis showed the presence of calcite, chamosite, apatite, quartz, hematite, siderite, chlorite and clay minerals.

4. Apatite is found associated with several minerals but only as very fine particles (<5 μm) intergrown with the hematite grains in the hematite ooliths; as very fine grains intergrown with chamosite in chamosite ooliths; as the matrix of fine grains intergrown with chamosite and calcite; intergrown with chamosite grains only in the matrix; as anhedral and subrounded particles (8-28 μm) in the ooliths.

5. Apatite is associated with the hematite and chamosite in an adsorbed form. It is found in the chamosite and hematite ooliths or in the chamosite matrix more than it occurs in the calcite matrix.

6. Based on the findings from this research, it is suggested that dephosphorisation of Frodingham ironstone by the Flox process or Reduction roasting followed by fine grinding, magnetic separation, acid leaching should be feasible.
9.0 SUGGESTIONS FOR FUTURE WORK

The specialised techniques used in this work have been shown to be useful in the examination of phosphorus bearing minerals in iron ores. These could be involved in studying other classes of iron ores to discover their phosphorus content and distribution.

The suggested methods of dephosphorisation for the three ores investigated need to be proven by mineral dressing and leaching tests. In this way it should be possible to make an economic assessment of dephosphorisation for the Aswan, Bahariya and Frodingham ores.

9.1 Economics

It is possible that in the work described above dephosphorisation processes were said to be uneconomic on very limited data. However, the economics of an ore dephosphorisation method need to be worked out for the whole iron and steel production route. For example, on the ore beneficiation costs it is unlikely that any suggested process could be economic from the increased value of the ore product. If, however, the extra costs at the B.F. (increased coke rate) and increased slag forming additions and process time extension in the steelplant were used in the accounting system, then a much more favourable economic picture would emerge. Thus, an important extension to the dephosphorisation work would be this overall process evaluation.
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