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on Mineralogy and Preliminary Concentration Tests of Paz del Rio Iron Ore Sample

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EXAMINATION OF PAZ DEL RIO IRON ORE

INTRODUCTION

On April 10, 1974, the Mineral Resources Research Center (MRRC) of the University of Minnesota received two samples weighing about 50 lb each from the Paz del Rio iron ore deposit in Colombia. At the MRRC the two samples were designated Lots 1 and 2 of Ore 1885-C62. A mineralogical examination and a few exploratory concentration tests were requested by Acerias Paz del Rio, S. A., to see if beneficiation at a reasonable cost was feasible.

MINERALOGICAL EXAMINATION

Polished sections were made on some coarse fragments (+1/2 inch), as well as on a concentrate obtained in a Franz magnetic separator. X-ray diffraction analyses were made on some portions of   screened fractions.

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The two samples submitted for study are of two different types. Macroscopically Lot 1 is an oolitic iron ore, dark brown in color, and relatively hard. Lot 2 is an earthy oolitic type, reddish to yellow, and much softer. The chemical analyses of the two lots are shown in Table 1.

Microscopic examination shows that the oolites vary from ovoidal to subspherical with an average diameter around 1 mm. They were originally made up of chlorites (chamosites?), later partially replaced by siderite and hematite in Lot 1 and by goethite in Lot 2. Very often the core and the shells of the oolites cannot be differentiated. The layered structure is concentric in some cases, in other assymetric, suggesting deformation during diagenesis (Photomicrographs 1 and 2). Some internal radial cracks are visible, often filled with siderite or goethite (Photomicrograph 5). The matrix is composed of angular fragmented quartz, intimately associated with siderite and chloritic material (Photomicrographs 1 and 2). In Lot 2 the quartz is generally coarser (around 80 microns) and more abundant than in Lot 1 (Photomicrographs 3 and 4). Goethite in the matrix fills the interstices between the quartz grains along with some residual chloritic material.

Most of the phosphorus in the ore appears to be present as apatite. However, the percentage of apatite present as discrete particles (around 50 microns) does not account for all the phosphorus in the ore. A part of it, especially in Lot 2 where the CaO content is insufficient to tie up all the phosphorus as apatite, is probably finely disseminated throughout the oolites (as iron phosphate?). The alumina is associated with some iron and silicate to form the chloritic minerals. No aluminum hydroxides, e.g., gibbsite, diaspore or boehmite, were identified. The x-ray diffraction studies of the minus 5-micron fraction did not reveal the presence of clay minerals.

In Lot 1 the main iron minerals are siderite, hematite, and goethite. The ratio of iron carbonate to iron oxides is about 3 to 1. The siderite in the oolites is very fine grained, often arranged in thin concentric layers and intimately associated with chlorite. In the gangue the siderite occurs as thin veinlets and stringers often enrobing the quartz grains, or as relatively well developed crystals (average size around 100 microns). Hematite occurs usually as crystal aggregates arranged roughly in concentric layers in the shells of the oolites. The average size of the crystals is around 50 microns. Some hematite is also present in the matrix as scattered crystals of finer size (average around 20 microns). Goethite is less abundant than hematite and usually replaces the siderite. A black organic compound (coal or graphite?) was observed in Lot 1, usually associated with the siderite present in the matrix.

In Lot 2 siderite and hematite have been practically completely replaced by goethite. In the oolites goethite appears in the cores or as concentric layers associated with altered chloritic material. In the matrix veinlets and stringers can be seen associated with angular quartz grains and altered soft chlorite (Photomicrographs 3 and 4).

CONCENTRATION TESTS

Concentration of the Paz del Rio ore was explored by bench-scale scrubbing, heavy-liquid tests, magnetic concentration, reduction roasting and flotation.

Bench-Scale Scrubbing

The microscopic examinations suggested that the ore might be beneficiated by intensive scrubbing. This was based on the presumption that some of the cementing material of the matrix as well as the softer chloritic minerals were lower in iron and could be removed as a slime fraction. The more resistant minerals like siderite, hematite and goethite could be further beneficiated by gravity separation, high intensity magnetic concentration or flotation.

The scrubbing tests were run on samples of each lot that had been crushed through 10 mesh (Table 2). About 150 grams of each lot was scrubbed in a Waring blender for 5 minutes at 20% solids. The screen analyses and iron distributions indicated in Table 3 show that Lot 1 did not respond well to this technique, mainly because of the nature of the matrix which is composed of finely crystallized siderite. The iron distribution in the scrubbed ore remains fairly constant in all the size fractions. By contrast, in Lot 2 it is apparent that the minus 325-mesh fraction, which represent 22.5% by weight, is substantially lower in iron content than the coarser fractions.

Heavy-Liquid Tests on Scrubbed Ore

The results of heavy liquid separation tests at a specific gravity of 3.3 (Table 4) show that siderite and hematite are liberated from the gangue materials at well below 100 microns. Microscopical examination of the float of the 100/200 mesh fraction shows that most of the 29.81% Fe indicated by the chemical analysis occurs as mixed siderite-quartz, particles and inclusion of hematite in chlorite. X-ray diffraction analysis shows that the ratio of carbonate over oxide is around 3 to 1.

In Lot 2 the oolites are cemented by relatively coarser quartz grains, soft chloritic material and goethite, alteration product of siderite. The matrix was quite severely affected by scrubbing, allowing a better separation by heavy liquid of the denser and stronger oolithic fragments from the quartz. The sink of the 48/100-mesh fraction assays 53% Fe. (Iron content of massive goethite is around 62%.) X-ray diffraction of the sink product shows the presence of some quartz along with chlorite.

High-Intensity Magnetic Concentration Tests

Beneficiation by high intensity dry magnetic separation did not prove to be an efficient way to upgrade the ore. In Lot 1 the tail rejection for the 28/200 mesh size fractions was 2.1% of the crude weight while the iron content in the concentrate for the corresponding size fractions was 43.44%, a gain of a little more than 1% (Table 5). The results were somewhat better for Lot 2. For the same fractions 6% of the weight of the crude was rejected as a tailing averaging 3.56% Fe. The concentrate of the 100/200-mesh fraction was upgraded from 38.76% to 49.78% Fe.

Reduction Roasting Tests

The conversion of iron oxides to magnetite was accomplished by crushing the ore lots through 10 mesh and then heating them at approximately 650° for up to an hour in a reducing atmosphere. A single magnetic separation by Davis tube was then made on each roasted ore lot crushed to minus 100 mesh. The results of the exploratory tests (Table 6) show that a much finer grind must be achieved to obtain concentrates of reasonable grade. The increase of iron in the roasted ore is mainly due to loss of volatile elements and to the departure of oxygen. However, the phosphorus content was substantially lower in the roasted ore, confirming the mineralogical observation that half of the phosphorus in Lot 1 and probably more in Lot 2 is represented by mineral species outside the apatite group, probably intimately associated with iron and alumina.

Flotation Tests

Several bench-scale flotation tests were conducted to produce an iron concentrate. The tests were begun by grinding each ore lot separately in a stainless steel rod mill at 50% solids to determine the amount of time required to grind each lot to approximately 100 percent passing a 270 mesh screen. The grinding tests indicated that Lot 1 and Lot 2 had to be ground for 25 and 17 minutes, respectively, to obtain the desired grind. Three different types of iron flotation were tried:

1. Selective flocculation followed by anionic flotation of siliceous gangue.

2. Desliming followed by anionic flotation of iron oxides.

3. Cationic flotation of siliceous gangue.

Selective Flocculation and Flotation by Anionic Flotation of Siliceous Gangue

The ground pulp from each lot was conditioned in a flotation cell with sodium silicate and Calgon as dispersants. Then iron minerals were selectively flocculated with a causticized tapioca solution. The flocculated sample was allowed to settle for a certain time and the supernatant suspension siphoned off. Water was again added. The pulp was again agitated for a while, allowed to settle and then siphoned. This process was repeated three times. The deslimed pulp was transferred to a conditioner and diluted to 40% solids. The pulp pH was adjusted to 11.0, and then conditioned with causticized tapioca, calcium chloride, and a fatty acid. The conditioned pulp was then transferred to a Fagergren laboratory cell, the pH adjusted to 11.0, and the pulp floated to obtain a rougher froth and a rougher cell product. The results and iron analysis of the products are given in Table 7.

Desliming Followed by Anionic Flotation of Iron Oxides

Desliming of the ground pulp was preceded by dispersing the pulp with sodium silicate and Calgon. The dispersed pulp was allowed to settle for 3 minutes (20-micron quartz equivalent) before being siphoned. Decantation was done four times after each water level adjustment, agitation, and settling. The sand fraction was then conditioned with fatty acid either at room temperature or at 95° C prior to flotation at room temperature or 75° C, respectively. The rougher froth (iron fraction) was cleaned once and the rougher tailing was either left as is or scavenged. The results are given in Table 7.

Cationic Flotation of Siliceous Gangue

The ground ore was deslimed as described in the first procedure. The sand fraction was diluted and some British gum and an amine solution were added to it. Froth products were obtained after the conditioning. The results are shown in Table 7.

SUMMARY AND RECOMMENDATIONS

The two lots of the Paz del Rio ore responded differently to conventional beneficiation procedures due to their different mineralogical compositions. In Lot 1 the ore minerals are siderite, hematite, and goethite. In Lot 2 goethite is the main iron ore mineral. Both samples have an oolitic texture. The mineralogical composition of the oolites is very complex mainly because of the intimate association of chlorite, siderite, hematite, or goethite, with no specific segregation of the ore mineral species in the core or in the shells. In Lot 1 the matrix is composed mainly of quartz and siderite, but in Lot 2 the siderite is replaced by goethite.

The preliminary beneficiation tests were for the most part discouraging. The gravity concentration of Lot 1, which produced about a 48% Fe concentrate at about 65% Fe recovery, could perhaps be viewed as an acceptable preconcentration step when compared with the 54% Fe which would be contained in the pure iron oxide minerals in this ore. This preconcentrate might respond more favorably to flotation than did the crude ore.

The magnetic roasting of both samples resulted in appreciable phosphorus volatilization and magnetic separation gave a much improved concentrate which no doubt could be improved further by magnetically concentrating a finer grind and/or by silica flotation of the remaining gangue. Roasting, however, is usually considered an expensive commercial processing step, and in any event the concentrate would probably have a phosphorus content of 0.2%.

The following tests will be carried out at MRRC with funds remaining for this project and will be reported when they become available:

1. gravity concentration of Lot 1 followed by regrinding and flotation of gravity concentrate,

2. magnetic roasting of both Lots 1 and 2 followed by fine grinding (-400 mesh), magnetic separation, and silica flotation.

TABLE 1. CHEMICAL ANALYSES

	Lot 1	Lot 2
% Fe	41.97	44.21
% Fe^{++}	14.19	0.32
% SiO_2	14.96	17.26
% Al_2O_3	4.42	4.91
% CaO	2.29	0.15
% MgO	0.53	0.13
% P	0.80	0.79
% Mn	0.14	0.20
% LOI	14.06	10.60

TABLE 2. SCREEN ANALYSES OF LOTS 1 AND 2 CRUSHED THROUGH 10 MESH

	Lot 1		Lot 2	
Mesh	Wt %	Cumul	Wt %	Cumul
+20	26.2		9.9	
20/35	26.1	52.3	25.7	35.6
35/48	7.8	60.1	12.1	47.7
48/65	9.6	69.7	15.0	62.7
65/100	6.1	75.8	7.4	70.1
100/150	4.5	80.3	5.0	75.1
150/200	4.0	84.3	4.4	79.5
200/270	1.8	86.1	2.0	81.5
270/325	2.5	88.6	2.8	84.3
325/500	3.2	91.8	3.2	87.5
-500	8.2	100.0	12.5	100.0

TABLE 3 SCRUBBING TEST AND SCREEN ANALYSES

Scrubbing feed: -10 mesh

Scrubbing : 5 minutes at 20% solids in Waring Blender

Lot 1				
Mesh	Wt %	Cumul	Fe %	Fe Dist %
+ 20	25.0		40.44	24.2
20/28	15.6	40.6	43.44	16.2
28/48	18.5	59.1	42.40	18.8
48/100	15.4	74.5	41.04	15.1
100/200	8.6	83.1	41.28	8.5
200/325	4.5	87.6	42.24	4.5
-325	12.4	100.0	42.68	12.7
Total	100.0		41.79	100.0

Lot 2				
Mesh	Wt %	Cumul	Fe %	Fe Dist %
+20	9.4		47.04	10.1
20/28	9.5	18.9	49.32	10.7
28/48	22.3	41.2	49.92	25.3
48/100	22.9	64.1	45.68	23.8
100/200	8.8	72.9	38.76	7.8
200/325	4.6	77.5	36.00	3.8
-325	22.5	100.0	36.32	18.5
	100.0		43.94	100.0

TABLE 4 HEAVY LIQUID SEPARATION DATA AT 3.3 SP.G. OF THE +20, 20/28, 28/48, 48/100, and 100/200 MESH SIZE FRACTIONS OBTAINED BY SCRUBBING

		Lot 1				Lot 2			
Mesh	Product	Wt %	Wt %*	Fe %	Fe Dist %	Wt %	Wt %*	Fe %	Fe Dist %
+ 20	float	65.8	16.4	(38.34)	62.4	34.8	3.3	(42.01)	31.1
	sink	34.2	8.6	44.48	37.6	65.2	6.1	49.72	68.9
		100.0	25.0	40.44	100.0	100.0	9.4	47.04	100.0
20/28	float	49.0	7.6	(38.36)	43.3	19.7	1.9	(39.65)	15.9
	sink	51.0	8.0	48.82	56.7	80.3	7.6	51.68	84.1
		100.0	15.6	43.44	100.0	100.0	9.5	49.32	100.0
28/48	float	45.0	8.3	(35.11)	37.3	14.1	3.1	(30.21)	8.5
	sink	55.0	10.2	48.36	62.7	85.9	19.2	53.16	91.5
		100.0	18.5	42.40	100.0	100.0	22.3	49.92	100.0
48/100	float	45.7	7.0	(33.58)	37.1	20.8	4.8	(17.36)	7.9
	sink	54.3	8.4	47.32	62.9	79.2	18.1	53.12	92.1
		100.0	15.4	41.04	100.0	100.0	22.9	45.68	100.0
100/200	float	37.1	3.2	29.81	26.8	34.8	3.1	13.94	12.5
	sink	62.9	5.4	48.04	73.2	65.2	5.7	52.01	87.5
		100.0	8.6	41.28	100.0	100.0	8.8	38.76	100.0

* Wt % by fraction

( ) calc. value

TABLE 5 HIGH INTENSITY MAGNETIC SEPARATION TESTS (Franz Separator) on the 28/48, 48/100 and 100/200 MESH SIZE FRACTIONS OBTAINED BY SCRUBBING

Lot 1						Lot 2					
Mesh	Product	Wt %	Wt %*	Fe %	Fe dist %	Mesh	Product	Wt %	Wt %*	Fe %	Fe dist %
28/48	conc at 0.4 a.	96.6	17.9	43.52	99.1	28/48	conc at 0.5 a.	95.3	21.3	52.16	99.8
	tail at 0.4 a.	3.4	0.6	(10.60)	0.9		tail at 0.5 a.	4.7	1.0	(4.47)	0.2
	total	100.0	18.5	42.40	100.0		total	100.0	22.3	49.71	100.0
48/100	conc at 0.4 a.	94.3	14.5	42.96	98.7	48/100	conc at 0.5 a.	87.1	19.9	51.96	99.1
	tail at 0.4 a.	5.7	0.9	(9.30)	1.3		tail at 0.5 a.	12.9	3.0	(3.26)	0.9
	total	100.0	15.4	41.04	100.0		total	100.0	22.9	45.68	100.0
100/200	conc at 0.4 a.	92.7	8.0	44.16	99.2	100/200	conc at 0.5 a.	77.0	6.8	49.28	97.9
	tail at 0.4 a.	7.3	0.6	4.66	0.8		tail at 0.5 a.	23.0	2.0	(3.57)	2.1
	total	100.0	8.6	41.28	100.0		total	100.0	8.8	38.76	100.0

* % by Wt fraction

( ) calculated fraction

TABLE 6 . CHEMICAL ANALYSES AND DAVIS TUBE SEPARATION OF THE LOTS AFTER MINUS 100-MESH GRINDS

LOT 1

	Fe %	Phos %
Crude ore	41.97	0.80
Roasted ore	49.82	0.47

	Wt %	Fe %	Phos %	Fe Rec %	Phos Rec %
Davis tube conc	76.6	56.23	0.35	86.5	57.0
Davis tube tail	23.4	(28.84)	(0.85)	13.5	43.0
Head		49.82	0.47		

Feed 37.5% minus 325

( ) calculated value

LOT 2

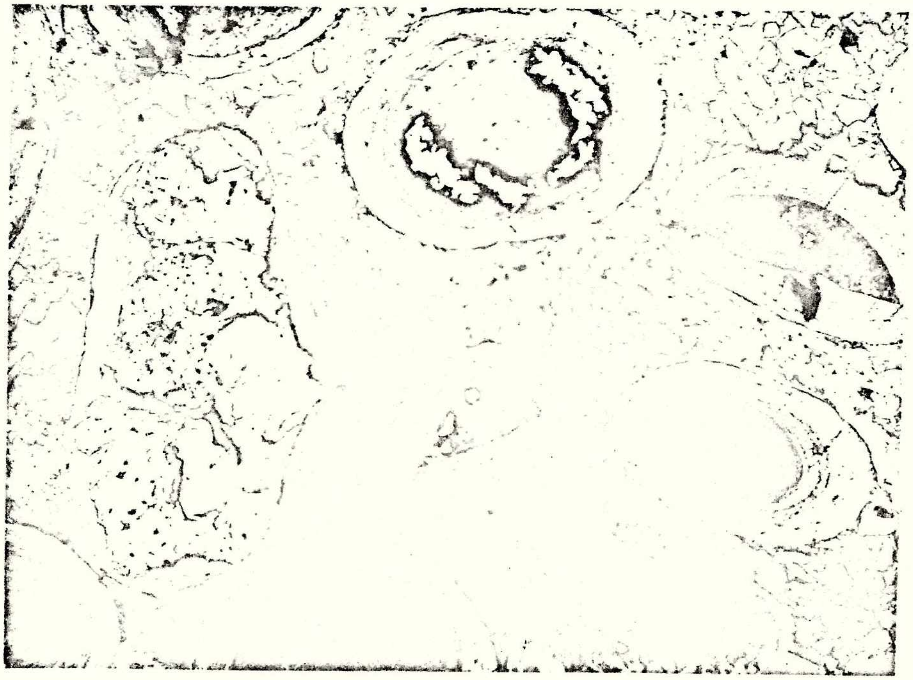
	Fe %	Phos %
Crude ore	44.21	0.79
Roasted ore	53.11	0.45

	Wt %	Fe %	Phos %	Fe Rec %	Phos Rec %
Davis tube conc	69.3	60.83	0.39	79.4	60.0
Davis tube tail	31.7	(34.54)	(0.57)	20.6	40.0
Head		53.11	0.45		

Feed 33.5% minus 325

Table 7. Results of Flotation Tests on Lots 1 and 2

Type of Test	Temp. ° C	Lot No.	Product	% Wt	% Fe	% Fe Dist.
Selective flocculation--anionic silica flotation	room (29° C)	1	Slimes	25.88	41.79	25.76
			Rougher froth (tails)	9.42	35.80	8.02
			Rougher cell product	64.70	42.99	66.22
		2	Slimes	46.64	41.87	44.47
			Rougher froth	3.36	43.47	3.32
			Rougher cell product	50.00	45.86	52.21
Desliming--anionic flotation of iron oxides	room	1	Slimes	34.32	42.19	34.47
			Scavenging tails	12.72	39.63	12.00
			Scavenging conc	25.94	43.31	26.73
			Cleaning tails	7.69	42.35	7.76
			Cleaner conc	19.33	41.39	19.04
	95° C-75° C	1	Organic (?) froth	4.38	32.12	3.34
			Slimes	27.82	43.39	28.54
			Rougher tails	35.92	41.07	34.90
			Cleaning tails	4.05	42.51	4.07
			Cleaner conc	27.83	44.27	29.15
Cationic flotation of siliceous gangue	room	1	Slimes	24.72		
			Rougher froth	3.06		
			Rougher cell product	72.22	42.34	
	room	2	Slimes	49.56		
			Rougher froth	0.45		
			Rougher cell product	49.99	46.35	

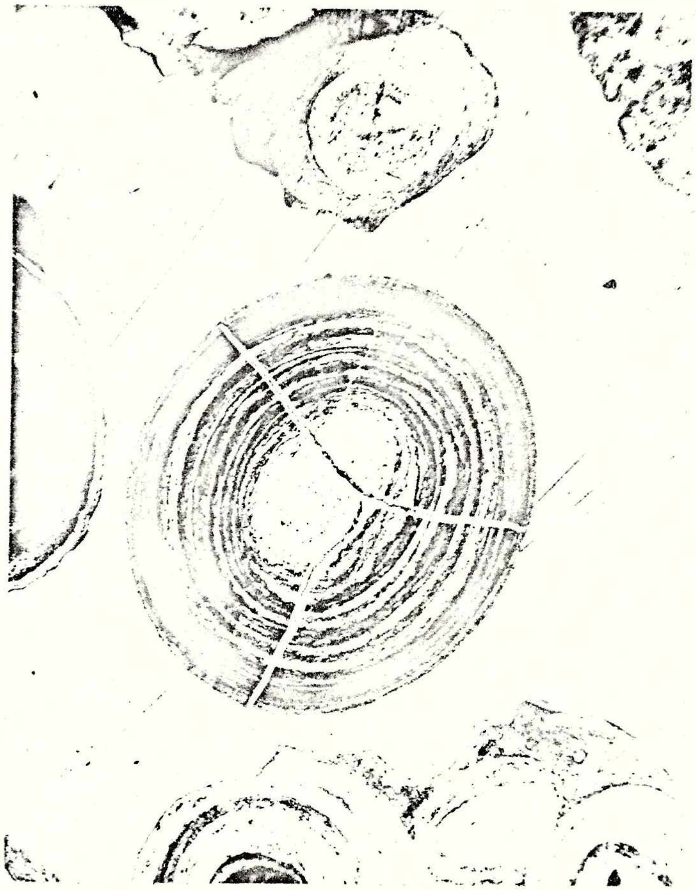


Photomicrograph No. 1. Polished section of a coarse fragment of Lot 1. Magnification 50x. Oolite formed of concentric layers of chlorite, siderite with aggregates of hematite crystals (white). Matrix is made of quartz (grey) and veinlets of siderite (light grey). Note the assymetric fragments of geothite (left, greyish white) enrobed by thin envelopes of siderite.

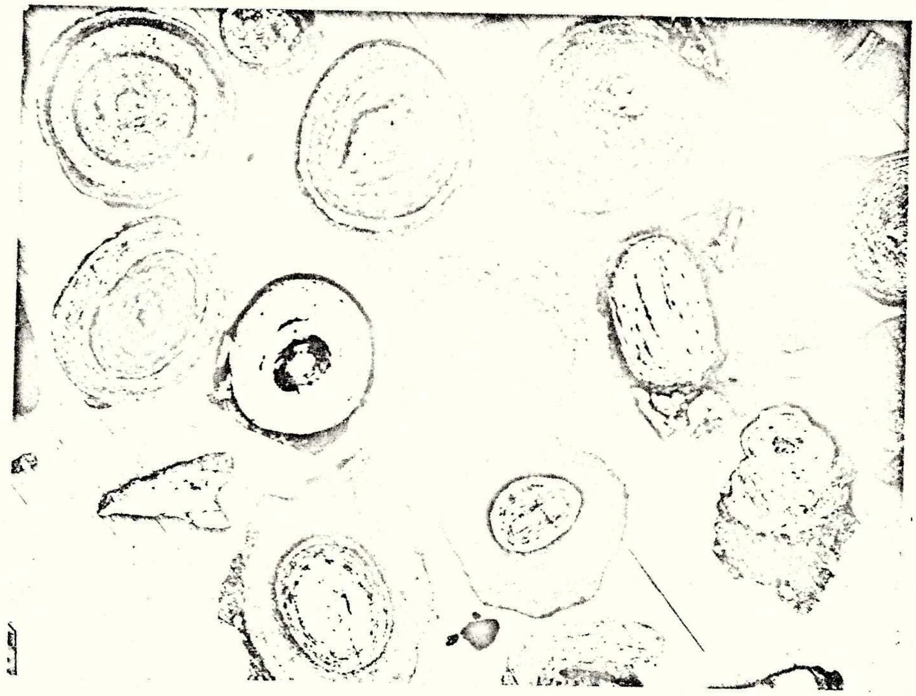
Photomicrograph No. 2. Lot one. Polished section. Magnification 50x. Oolites of thin concentric layers of siderite and chlorite with coarser aggregates of hematite (white). The matrix is mainly composed of siderite (light grey), quartz (dark grey). Very fine crystals of hematite (white dots) are scattered in the cement.

Photomicrograph No. 3. Lot two. Polished section. Magnification 50x. Oolites with concentric layers of goethite (greyish white). Associated with remnants of chloritic material. The black area in the matrix correspond to holes created during the polishing and showing the soft nature of the gangue.

Photomicrograph No. 4. Lot two. Polished section. Magnification 50x. Oolites of goethite (greyish white). The matrix is composed of angular quartz fragments (grey), altered chloritic material (black) and very fine goethite (white cloudy stringers).



Photomicrograph No. 5. Polished section. Magnification 50x. Lot two: oolites liberated by scrubbing. Dense core of goethite surrounded by concentric layer of chlorite and goethite. Radial cracks filled with goethite.



Photomicrograph No. 6. Lot two. Magnification 50x. Polished section of the high intensity magnetic separation concentrate of the 28/48 mesh size fraction. Oolites of goethite (greyish white) and chloritic material (dark grey).