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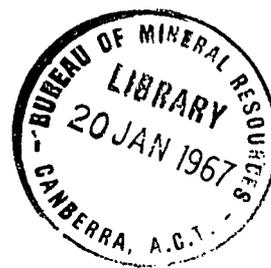
DEPARTMENT OF NATIONAL DEVELOPMENT  
BUREAU OF MINERAL RESOURCES  
GEOLOGY AND GEOPHYSICS

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UPGRADING WESTERN AUSTRALIAN MANGANESE ORES

(A.M.D.L. Report No. 373)

by

L. Bollen and D.C. Madigan

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\* of the Australian Mineral Development Laboratories, Adelaide.

## Introduction

In the course of producing metallurgical grade manganese ore to buyers specifications a number of producers in Western Australia have accumulated dumps of undersize material which is also lower in grade than the usual specification.

Samples of dumps at Woodie Woodie and Peak Hill were supplied by the Western Australian Mines Department and beneficiation tests were carried out on this material.

The report has been included in the Bureau's Record series so that it may enjoy the wider distribution offered by the "open file" system. It has not been altered in any way.

2/1/6

AMDL Report 373  
September, 1964

UPGRADING OF WESTERN AUSTRALIAN MANGANESE ORES

by

L. Bollen and D. C. Madigan

to

THE BUREAU OF MINERAL RESOURCES

Investigated by: Metallurgy Section

Officer in Charge: P. K. Hosking

L. Wallace Coffey, Director

THE AUSTRALIAN MINERAL DEVELOPMENT LABORATORIES

Adelaide South Australia

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## 1. INTRODUCTION

Australian consumption<sup>1</sup> of metallurgical grade manganese ore (48% Mn) in 1962 was estimated at approximately 44,000 tons. Approximately one third of this was supplied from domestic sources.

Production in Australia of metallurgical grade manganese ore fell from 85,800 tons in 1961 to 71,300 tons in 1962. In 1962 some 63,000 tons of high grade ore were exported, mainly to Japan.

Some Australian producers, to fulfil market requirements, screen out low-grade fines. The Bureau of Mineral Resources requested that beneficiation tests be carried out on samples from two dumps of such reject material. The samples tested were from Peak Hill and Woodie Woodie.

## 2. SAMPLE FROM PEAK HILL DUMP

### 2.1 Summary

One 44-gallon drum of minus 1½-inch material was received. The material assayed 30.0 per cent Mn and 19.2 per cent Fe. The ore consisted of pyrolusite with a gangue consisting of goethite, clay and quartz.

A sizing analysis of the ore showed that, although washing and screening methods would remove some low-grade material, the resultant concentrate was not of acceptable grade.

Gaseous reduction at 800°C of a sample of minus ½-inch plus 10-mesh material resulted in a loss in weight of 14.4 per cent. After grinding the reduced material to minus 300 mesh with subsequent magnetic separation, a non-magnetic fraction assaying 49.0 per cent Mn and 9.6 per cent Fe was obtained. This concentrate contained 87.4 per cent of the manganese in the fraction treated. The manganese to iron ratio is 5.1 compared with the ratio generally required of at least 6. Blending with manganese ores of low iron content would be a method of making this concentrate acceptable.

Heavy liquid separations conducted on reduced material crushed to minus 10 mesh showed that some silica could be rejected by gravity methods. In the minus 100- plus 200-mesh fraction 61.6 per cent of the silica was rejected whilst coarser fractions rejected less. If gravity and magnetic separations were applied to the reduced ore, the manganese concentrate produced would still carry a penalty because of the amount of silica present. The presence of other penalty elements could not be ascertained owing to the lack of funds.

Flotation tests were unsuccessful because the floatabilities of the pyrolusite and goethite were similar. Flotation manganese concentrates containing large amounts of iron were obtained in all tests.

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1. Commonwealth of Australia, Bureau of Mineral Resources, "The Australian Mineral Industry - 1962 Review".

A leaching test was made on the original ground ore with sulphur dioxide, and leaching tests were made on reduced ore with sulphuric acid, nitric acid and ammoniacal ammonium carbonate with the results shown in Table 1.

TABLE 1: RESULTS OF LEACHING TESTS

Reagent	Extraction, %		Mn: Fe Ratio in Leach Liquor
	Mn	Fe	
SO <sub>2</sub>	98.2	5.81	26.8
H <sub>2</sub> SO <sub>4</sub>	100	10.6	14.9
HNO <sub>3</sub>	98.3	8.04	19.2
NH <sub>3</sub> -(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>	9.2	0.07	206

Leaching with sulphur dioxide is recommended for further study.

## 2.2 Material Examined

One 44-gallon drum of ore was received. The ore was approximately minus 1 $\frac{1}{2}$  inches in sizing and contained a very high percentage of minus 10-mesh material. A mineralogical examination (see Appendix A) was carried out on lump ore and on a representative sample of ore crushed to minus 10 mesh.

The ore was assayed with the following results:

		<u>%</u>
Manganese	Mn	30.0
Iron	Fe	19.2
Phosphorus	P <sub>2</sub> O <sub>5</sub>	0.12
Acid insoluble		11.1
Lime	CaO	0.085
Magnesia	MgO	0.03
Alumina	Al <sub>2</sub> O <sub>3</sub>	3.9

### 2.3 Equipment

The following equipment was used:

Screen, Warill:(a)	20 in. dia
Magnetic separator, Stearns:	disc, "pick-up" type
Crushing rolls, Star Machinery Ltd:	9 in. dia x 6 in. wide
Gas-fired furnace fitted with continuous weighing mechanism	
Davis tube magnetic separator	
Rod mill, laboratory:	10.5 in. long x 7 <sup>5</sup> / <sub>16</sub> in. dia
Flotation cell, Fagergren:	2500 ml capacity
Laboratory sieves:	BSS

### 2.4 Experimental Procedure and Results

#### 2.4.1 Sizing Analysis of the Ore

A 500-lb sample of the dump material, as received, was screened into fractions which were crushed and assayed. Results are given in Table 2.

TABLE 2: SIZING ANALYSIS OF ORE AS RECEIVED

Size Fraction	Weight %	Assays		Distribution	
		Mn %	Fe %	Mn %	Fe %
+ 1 in.	22.1	35.3	18.2	26.1	20.5
- 1 + 1/2 in.	12.7	37.1	16.4	15.8	10.6
- 1/2 in. + 10 mesh	40.5	32.1	19.7	43.6	40.5
- 10 + 72 "	15.5	21.5	24.6	11.2	19.3
- 72 + 200 "	5.1	11.4	20.1	2.0	5.2
- 200 "	4.1	9.4	18.9	1.3	3.9
Feed (calc)	100.0	29.8	19.7	100.0	100.0
Feed (assay)	-	30.0	19.2	-	-

(a) Reference to specific equipment in this report is made to facilitate understanding and does not imply endorsement of such equipment by The Australian Mineral Development Laboratories.

### 2.4.2 Dry Magnetic Separation Tests

Dry magnetic separation tests were carried out on ore crushed to minus 10 mesh and minus 52 mesh. Test conditions are given in Table 3 and results in Tables 4 and 5.

TABLE 3: OPERATING CONDITIONS OF STEARNS DRY MAGNETIC SEPARATOR

Conditions	Ore Crushed to	
	- 10 mesh	- 52 mesh
Coil current, amp	1.1	1.1
Belt speed: ft/min	30	30
Pole gaps: front inch	$\frac{3}{16}$	$\frac{3}{32}$
back inch	$\frac{1}{8}$	$\frac{1}{16}$

TABLE 4: DRY MAGNETIC SEPARATION OF ORE CRUSHED TO MINUS 10 MESH

Product	Weight %	Assays		Distribution	
		Mn, %	Fe, %	Mn, %	Fe, %
Magnetic concentrate 1	13.2	25.8	26.9	11.5	18.6
Magnetic concentrate 2	28.4	34.4	18.7	32.8	27.8
Tailing	58.4	28.4	17.6	55.7	53.6
Feed (calc)	100.0	29.8	19.1	100.0	100.0
Feed (assay)	-	30.0	19.2	-	-

TABLE 5: DRY MAGNETIC SEPARATION OF ORE CRUSHED TO MINUS 52 MESH

Product	Weight %	Assays		Distribution	
		Mn, %	Fe, %	Mn, %	Fe, %
Magnetic concentrate 1	25.4	29.8	22.4	24.7	28.6
Magnetic concentrate 2	8.2	31.1	21.0	8.2	8.6
Tailing	66.4	30.8	18.8	67.1	62.8
Feed (calc)	100.0	30.6	19.9	100.0	100.0
Feed (assay)	-	30.0	19.2	-	-

#### 2.4.3 Gaseous Reduction with Subsequent Magnetic Separation

A sample of the minus  $\frac{1}{2}$ -inch plus 10-mesh fraction of the original ore was given a magnetising roast. A 1500 g charge was heated for 4 hours at 800°C in a gas fired furnace with a continuous weighing mechanism. A mixture of CO-CO<sub>2</sub> was passed through the charge for 1 hour after constant weight had been obtained. The charge was cooled using carbon monoxide and nitrogen. No further reduction in weight was recorded during the cooling period.

The sample lost weight as follows:

	%
Loss in weight at 105°C	2.0
- ditto - 800°C (heated in air)	10.8
" 800°C (heated in reducing atmosphere)	14.4

The reduced sample was crushed to minus 10 mesh. Portions of the minus 10-mesh material were crushed to minus 36 mesh and minus 100 mesh. A Stearns pick-up type magnetic separator was used to separate the minus 10-mesh and minus 36-mesh material. Operating conditions are given in Table 6 and results are given in Tables 7 and 8.

TABLE 6: OPERATING CONDITIONS OF STEARNS DRY  
MAGNETIC SEPARATOR

Conditions			Reduced Material Crushed to	
			- 10 mesh	- 36 mesh
Coil current,	amp	0.1	0.15	
Belt speed,	ft/min	12	15	
Pole gaps:	front, inch	0.2	0.2	
	back, inch	0.15	0.15	
Field strength:	front, gauss	200	nd <sup>(a)</sup>	
	back, gauss	280	nd	

(a) nd - not determined.

TABLE 7: DRY MAGNETIC SEPARATION OF REDUCED  
MATERIAL CRUSHED TO MINUS 10 MESH

Product	Weight %	Assays		Distribution	
		Mg, %	Fe, %	Mg, %	Fe, %
Magnetic concentrate 1	17.2	11.5	50.7	5.4	35.0
Magnetic concentrate 2	15.5	19.7	44.0	8.3	27.4
Tailing	67.3	47.1	13.9	86.3	37.6
Feed (calc)	100.0	36.8	24.9	100.0	100.0
Feed (assay) <sup>(a)</sup>	-	37.5	23.0	-	-

(a) Reduced material (calculated).

TABLE 8: DRY MAGNETIC SEPARATION OF REDUCED MATERIAL CRUSHED TO MINUS 36 MESH

Product	Weight %	Assays		Distribution		
		Mn, %	Fe, %	Mn, %	Fe, %	
Magnetic concentrate 1	41.2	44.2	17.7	42.1	21.4	77.3
Magnetic concentrate 2	3.0					
Tailing	55.8		49.9	9.8	78.6	22.7
Feed (calc)	100.0		35.5	24.1	100.0	100.0
Feed (assay) <sup>(a)</sup>	-		37.5	23.0	-	-

(a) Reduced material (calculated).

The results shown in Tables 7 and 8 indicated that increased iron rejection might be obtained by finer grinding and subsequent magnetic separation. Hence wet magnetic separation tests using a Davis tube were conducted on samples of reduced material ground to minus 100 mesh and minus 300 mesh. Operating conditions of the Davis tube are given in Table 9 and results of the separation are given in Tables 10 and 11.

TABLE 9: OPERATING CONDITIONS OF THE DAVIS TUBE

Coil current	amp	3.0
Water flow-rate	ml/min	800
Tube movement	cycles/min.	80

An examination, by x-ray diffraction, of the magnetic tailing reported in Table 11 showed hematite as the main phase of the iron oxide. No magnetite or goethite was detected.

TABLE 10: WET MAGNETIC SEPARATION OF REDUCED MATERIAL CRUSHED TO MINUS 100 MESH

Product	Weight %	Assays		Distribution	
		Mn, %	Fe, %	Mn, %	Fe, %
Magnetic concentrate	36.3	15.2	44.5	14.7	72.6
Tailing	63.7	50.4	9.6	85.3	27.4
Feed (calc)	100.0	37.6	22.3	100.0	100.0
Feed (assay)(a)	-	37.5	23.0	-	-

(a) Reduced material (calculated).

TABLE 11: MAGNETIC SEPARATION OF REDUCED MATERIAL GROUND TO MINUS 300 MESH

Fraction	Weight %	Assays		Distribution	
		Mn, %	Fe, % <sup>(a)</sup>	Mn, %	Fe, %
Magnetic concentrate	30.8	11.7	47.8	9.7	67.0
Magnetic middling <sup>(b)</sup>	3.5	30.7	26.8	2.9	4.3
Magnetic tailing	65.7	49.0	9.6	87.4	28.7
Feed (calc)	100.0	36.9	22.0	100.0	100.0

(a) Acid soluble Fe.

(b) A middling fraction was made because the end point of the separation was not clearly defined. The magnetic fraction in the Davis tube continually rejected or "shed" material. This rejected material has been termed middling.

#### 2.4.4 Heavy-Liquid Separations of Reduced Ore

A portion of the material which had undergone gaseous reduction (see Section 2.4.3) was crushed to minus 10 mesh. A sizing analysis of this material is given in Table 12.

TABLE 12: SIZING ANALYSIS OF REDUCED MATERIAL  
CRUSHED TO MINUS 10 MESH

Size Fraction				Weight %	Assays(a)		Distribution	
Microns		Mesh, BSS			Mn, %	SiO <sub>2</sub>	Mn, %	SiO <sub>2</sub> , %
-1670	+420	- 10	+ 36	75.9	37.9	7.1	79.0	68.6
- 420	+150	- 36	+100	15.4	33.9	9.0	14.4	17.7
- 150	+ 75	-100	+200	4.6	28.1	11.6	3.5	6.8
- 75		-200		4.1	26.7	13.2	3.1	6.9
			Feed (calc)	100.0	36.4	7.8	100.0	100.0

(a) Calculated from Table 13.

The size fractions, excluding the minus 200-mesh fraction, were separated in heavy liquid (bromoforn) having a specific gravity of 2.85 to 2.90. Results are given in Table 13.

TABLE 13: RESULTS OF HEAVY LIQUID SEPARATIONS

Fractions		Weight %	Assays		Distribution	
Mesh BSS	Heavy Liquid Fraction		Mn, %	SiO <sub>2</sub> , %	Mn, %	SiO <sub>2</sub> , %
- 10 + 36	Heavy fraction	95.5	39.6	5.1	99.7	68.6
	Light fraction	4.5	2.2	49.3	0.3	31.4
	Feed (calc)	100.0	37.9	7.1	100.0	100.0
- 36 +100	Heavy fraction	88.6	36.5	5.35	95.6	53.0
	Light fraction	11.4	14.0	37.0	4.4	47.0
	Feed (calc)	100.0	33.9	9.0	100.0	100.0
- 100 +200	Heavy fraction	84.0	31.3	5.3	93.6	38.4
	Light fraction	16.0	11.2	44.6	6.4	61.6
	Feed (calc)	100.0	28.1	11.6	100.0	100.0

#### 2.4.5 Flotation Tests

The dump material crushed to minus 10 mesh was ground in a rod mill under the following conditions:

Time of grinding,	min	15
Pulp density,	% solids	60
Ore charge,	g	1000
Rod mill size,		10 <sup>1</sup> / <sub>8</sub> x 7 <sup>5</sup> / <sub>16</sub> in. dia
Number of rods,	1 in. dia	15
Rod mill,	rpm	67

The ground pulp had the sizing shown in Table 14.

TABLE 14: SIZING OF GROUND PULP

Size Fraction		Weight	
Microns	Mesh, BSS	%	
+ 300	+ 52	0.7	
- 300 + 210	- 52 + 72	9.0	
- 210 + 150	- 72 + 100	23.6	
- 150 + 105	- 100 + 150	21.3	
- 105 + 75	- 150 + 200	8.0	
- 75	- 200	37.4	
		100.0	

Prior to flotation, the ground pulp was deslimed by decantation three times at 18 microns (nominal size for quartz) in a bucket using 2 lb per ton of sodium silicate (Type N41).

Four flotation tests were carried out. Anionic flotation reagents, Tests 1, 2, 3 and a cationic reagent, Test 4, were used. Emulsions of the two anionic reagent combinations (Test 1, 2 and 3) were as follows:

	Parts by Weight	
	Type A	Type B
Pamak 4	5.0	5.0
Fuel oil	10.0	3.25
Triton X	0.5	0.25
Naphthenic acid	1.0	0.5
	16.5	9.0

Adelaide tap water and the natural pH were used in all tests, which were conducted at 30 per cent solids and 25<sup>0</sup>C. A Fagergren flotation cell of 2.5 litres capacity was used.

Other conditions were as follows:

- Test 1: The ground deslimed pulp was conditioned at 50 per cent solids for 15 minutes with 1.65 lb per ton of Reagent Type A. Flotation time, 7 minutes.
- Test 2: As for Test 1, using 8.25 lb per ton of Reagent Type A. Flotation time, 12 minutes.
- Test 3: Stage additions of 0.5 lb per ton Reagent Type A for two stages, Type B in the following four stages.  
The pulp was conditioned 5 minutes in the flotation cell, with a flotation time of 4 minutes for each stage. A final stage with 1 lb per ton of Reagent Type B was conditioned for 20 minutes.
- Test 4: A cationic reagent, Duomac T, was stage added (0.36, 0.22, 0.27, 0.27, 0.40 lb/ton) with conditioning times of 5, 5, 5, 5, and 20 minutes. Flotation time was 5 minutes after each stage. Aerofroth 65 (0.01 lb/ton) was added to first stage only.

The results of the flotation tests are given in Table 15.

TABLE 15: RESULTS OF FLOTATION TESTS

Test No.	Product	Weight %	Assays		Distribution		
			Mn, %	Fe, %	Mn, %	Fe, %	
1	Rougher conc	22.4	32.0	21.8	23.3	26.3	
	Rougher tailing	70.0	31.6	17.4	71.9	65.5	
	Slime	7.6	19.4	20.1	4.8	8.2	
	Feed (calc)	100.0	30.7	18.6	100.0	100.0	
2	Rougher conc	89.3	32.0	18.2	95.0	89.5	
	Rougher tailing	6.7	11.7	15.7	2.6	5.8	
	Slime	4.0	18.0	21.5	2.4	4.7	
	Feed (calc)	100.0	30.1	18.2	100.0	100.0	
3	Rougher conc 1	23.2	21.8	26.7	17.6	19.7	20.6
	- ditto - 2	28.0					
	" 3	18.0					
	" 4	13.7					
	" 5	5.6					
	" 6	2.5					
	Rougher tailing	2.6	11.2	15.9	1.0	2.2	
	Slime	6.4	18.8	20.8	4.1	7.1	
	Feed (calc)	100.0	29.6	18.7	100.0	100.0	
	4	Rougher conc 1	2.1	6.6	22.4	19.9	5.0
- ditto - 2		2.2					
" 3		2.3					
" 4		14.7	15.1	19.5	18.1	10.0	14.5
" 5		0.4					
Rougher tailing		72.0	33.1	18.8	81.0	71.7	
Slime		6.3	18.8	20.7	4.0	6.9	
Feed (calc)		100.0	29.5	18.9	100.0	100.0	
Feed (assay)		-	30.0	19.2	-	-	

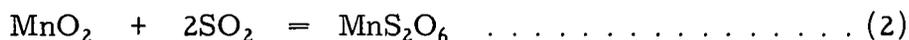
#### 2.4.6 Leaching Tests

Leaching with Sulphur Dioxide. A suspension of 50 g of the original ore (ground to minus 72 mesh) in 150 ml of water was leached by passing a mixture of air and SO<sub>2</sub> containing 6 per cent of SO<sub>2</sub> by volume. The gas mixture was passed for 4 hours at the rate of 167 l per hour. The mixture was stirred magnetically to keep the sample in suspension during leaching. No heat was supplied during leaching, but the heat of reaction

caused an appreciable rise in temperature. After leaching, the mixture was filtered. The filter was washed and filtrate and washings were combined and analysed for manganese and iron. The residue was dried and weighed.

The weight of the dry residue was 23.39 g, i. e. 53.2 per cent of the sample dissolved during leaching. The extraction of manganese was 98.2 per cent, of iron 5.81 per cent. The ratio of Mn to Fe in the leach liquor was 26.8.

Leaching with  $\text{SO}_2$  may produce both sulphate (1) and dithionate (2)



The theoretical quantity of  $\text{SO}_2$  required for a 50-g sample (=15.15 g Mn) is 17.7 g for reaction(1) and 35.4 g for reaction (2). In practice both reactions usually take place, the ratio of sulphate to dithionate depending on the conditions. Hence, the theoretical  $\text{SO}_2$  requirement should be between 17.7 and 35.4 g. The quantity of  $\text{SO}_2$  passed was approximately 105 g, or 3 to 6 times the theoretical quantity required.

Reduction of Ore. Direct leaching of manganese dioxide without preliminary reduction is possible with  $\text{SO}_2$ , but with other leaching reagents which, unlike  $\text{SO}_2$ , are not themselves reducing agents, reduction of the ore before leaching is necessary. The aim is to reduce  $\text{MnO}_2$  to  $\text{MnO}$  and  $\text{Fe}_2\text{O}_3$  to  $\text{Fe}_3\text{O}_4$ , but not to  $\text{FeO}$  or  $\text{Fe}$ , otherwise iron may be extracted with manganese. Reduction conditions were therefore selected that would reduce  $\text{Fe}_2\text{O}_3$  to  $\text{Fe}_3\text{O}_4$ , but not to a lower state of oxidation.

The sample was pelletised before reduction. Pelletisation was effected by dry grinding the sample to minus 72 mesh and working it into a thick paste with water. The paste was spread out on trays, cut into rectangular pellets about  $\frac{3}{4}$  by  $\frac{1}{2}$  by  $\frac{1}{4}$  inch and allowed to dry in air until the pellets were hard enough to be removed from the trays. The pellets were then dried in an air oven at  $80^\circ\text{C}$ .

About 1 kg of the pelletised sample was packed into a stainless steel reduction tube. The tube was set vertically in a gas fired furnace. Reducing gas ( $\text{CO}_2 + \text{CO}$ ) was passed into the bottom of the tube. The temperature was measured by a thermocouple placed in the middle of the sample and was recorded on a chart. The reduction tube rested on a lever connected to a balance, so that variations in weight could be observed.

Reduction was carried out at  $750^\circ\text{C}$  with a gas mixture containing 80 per cent  $\text{CO}_2$  and 20 per cent  $\text{CO}$  by volume and passed at the rate of 2 litres per minute. The reduction was continued until there was no further loss of weight (10 hours), but 95 per cent of the weight loss took place within 6 hours. The total loss in weight was 15.9 per cent. The loss in weight on heating the dried pellets in air for 2 hours at  $800^\circ\text{C}$  was 10.3 per cent so that loss due to reduction was 5.6 per cent. The reduced sample was broken up by means of a roller to pass 72 mesh. The original and reduced materials were assayed for iron and manganese with results as follows:

	<u>Mn, %</u>	<u>Fe, %</u>
Original material	30.3	19.1
Reduced material	36.0	22.9

Leaching of Reduced Ore with H<sub>2</sub>SO<sub>4</sub>. Twenty-five g of reduced ore plus 88.5 ml of H<sub>2</sub>SO<sub>4</sub> solution containing 205.3 g of H<sub>2</sub>SO<sub>4</sub> per litre were stirred for 2 hours at 24.5°C. The mixture was then filtered, the filter was washed with distilled water, and filtrate and washings were diluted to 250 ml. The diluted solution was stood for 24 hours, when a buff coloured precipitate appeared, presumably basic ferric sulphate. This precipitate was removed by filtration on a dry filter, and the clear solution, which had a pH of 2.4, was analysed for manganese and iron.

An excess of H<sub>2</sub>SO<sub>4</sub> of 13.1 per cent of the theoretical quantity required to react with all the manganese present was used. The dried residue weighed 12.85 g, i. e. 48.6 per cent of the sample was dissolved by leaching. The extraction of manganese into the leach liquor was 100 per cent, and of iron 10.6 per cent. The ratio of Mn to Fe in the leach liquor was 14.9.

Leaching of Reduced Ore with HNO<sub>3</sub>. Concentrated HNO<sub>3</sub> (46 ml of 70% acid, SG 1.42) was added slowly with stirring to a mixture of 50 g of reduced sample and 50 ml of water. Considerable reaction occurred, with the liberation of some oxides of nitrogen. When the initial reaction had subsided, the mixture was stirred under reflux for 1 hour at 95°C, then cooled and filtered. The filter was washed with distilled water, and filtrate and washings were diluted to 500 ml. This solution, which had a pH of 1.5, was analysed for manganese and iron.

The HNO<sub>3</sub> used was 10 per cent in excess of the theoretical quantity required to react with all the manganese. The dried residue weighed 26.38 g, i. e. 47.2 per cent of the sample dissolved during leaching. The extraction of manganese was 98.3 per cent, and of iron 8.04 per cent. The ratio of Mn to Fe in the leach liquor was 19.2.

Ammoniacal Leaching of Reduced Ore. The following mixture:

- 62.5 g of reduced sample
- 59.0 g of ammonium carbonate powder
- 222 ml of concentrated NH<sub>3</sub> (15.2N)

was stirred for 1 hour at 25°C and filtered. The filter was washed with a little ammonia solution, and filtrate and washings were combined and analysed for manganese and iron.

The weight of dried residue was 60.22 g, i. e. 3.65 per cent of the sample dissolved during leaching. The extraction of manganese was 9.2 per cent and of iron 0.07 per cent. The ratio of Mn to Fe in the leach liquor was 206.

## 2.5 Discussion

The geology of the Peak Hill deposits has been reported by de la Hunty.<sup>1</sup>

The sample received is understood to be representative of a dump of 100,000 tons of a previous reject material. The ore assayed 30.0 per cent manganese and 19.2 per cent iron, and consisted largely of pyrolusite and goethite. Quartz gangue occurs as grains up to 0.3 mm across and other gangue occurs as irresolvable material (clay and possibly silica) in the goethite (see Appendix A).

A sizing analysis of the ore, Table 2, showed that, although washing and screening methods would remove some low grade material, the resultant concentrate would not be of acceptable grade.

Dry magnetic separation tests, conducted on the ore ground to minus 10 mesh and to minus 52 mesh (see Tables 4 and 5), showed that the ore could not be satisfactorily upgraded by this method.

Gaseous reduction at 800°C of a sample of the minus 1/2-inch plus 10-mesh fraction of the ore produced a loss in weight of 14.4 per cent. Magnetic separation to remove the magnetite so formed produced non-magnetic fractions containing a considerable amount of iron. Magnetic separations conducted on material crushed to minus 10, minus 36, minus 100 and minus 300 mesh produced non-magnetic fractions assaying 13.9, 9.8, 9.6 and 9.6 per cent iron respectively (see Tables 7, 8, 10 and 11). An examination of the non-magnetic fraction (see Table 11) by x-ray diffraction showed that hematite was the main phase of the iron oxide. No magnetite or original goethite was detected. As this material had been gaseously reduced, the presence of hematite in the sample must have been due to either:

1. incomplete gaseous reduction, or
2. re-oxidation of the reduced material.

From Table 11 it can be seen that magnetic separation of the reduced material produced a non-magnetic fraction which assayed 49.0 per cent manganese and 9.6 per cent iron. This fraction contained 87.4 per cent of the manganese and 28.7 per cent of the iron. The Mn to Fe ratio is therefore 5.1, whilst the ratio generally required is greater than 6. Manganese ores are at present purchased on the basis of 48 per cent manganese and may be rejected if lower than 46 per cent manganese.

As stated previously, gaseous reduction was conducted on the minus 1/2-inch plus 10-mesh fraction of the ore as received. This reduced material was crushed to minus 10 mesh and sized fractions were separated in heavy liquid of SG 2.85 to determine if silica could be rejected by gravity means. In the minus 100- plus 200-mesh fraction (see Table 13) 61.6 per cent of the silica was rejected as a light fraction. Coarser fractions rejected less silica. All heavy fractions obtained assayed in excess of 5 per cent silica. Manganese ores sometimes carry penalties if they contain in excess of 4 per cent silica.

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1. de la HUNTY, L. E., "The Geology of the Manganese Deposits of Western Australia", Geological Survey of Western Australia, Bulletin No. 116, 1963.

Flotation tests conducted on ore ground to 37.4 per cent minus 200 mesh, (see Table 15) showed that anionic reagents (Tests 1, 2 and 3) were the most successful in floating the manganese mineral (pyrolusite). Unfortunately, the goethite floated equally well. Varying reagent additions, conditioning times and stage additions, although separating different amounts of material, gave products with similar unacceptable ratios of manganese to iron. Because the floatability of the pyrolusite and the goethite are similar, these two minerals were not separated by flotation.

Ammoniacal leaching of reduced Peak Hill ore was not successful, but leaching of reduced ore with  $H_2SO_4$  and  $HNO_3$ , and leaching of the original ore with  $SO_2$  were all equally successful, giving virtually complete extraction of manganese.

## 2.6. Conclusions

No cheap method of beneficiation was found to produce a concentrate which assayed in excess of 46 per cent manganese and which had a Mn to Fe ratio greater than 6.

Gaseous reduction at  $800^{\circ}C$  with subsequent magnetic separation produced a non-magnetic fraction which assayed 49.0 per cent Mn and 9.6 per cent Fe. This fraction contained 87.4 per cent of the manganese in the fraction treated.

Leaching of the original ore with  $SO_2$  and of the reduced ore with  $H_2SO_4$  and  $HNO_3$  were equally successful.

Leaching with  $SO_2$  is recommended for further study, since:

- a. it does not require preliminary reduction of the ore, and
- b. it extracts less iron than the other acid leaching systems.

## 3. SAMPLE FROM WOODIE WOODIE DUMP

### 3.1 Summary

Two 44-gallon drums of low grade manganese ore were received. The material assayed 32.7 per cent Mn and 6.3 per cent Fe. The ore was approximately minus 1 inch and consisted of manganese oxides, goethite, quartz, clay and carbonates.

A sizing analysis of the ore as received showed that screening on a  $1/2$ -inch aperture screen produced an oversize fraction assaying 45.5 per cent Mn and 4.8 per cent Fe. The fraction contained 38.5 per cent of the manganese in the original ore.

A magnetic separation test conducted on ore which had been gaseously reduced at  $750^{\circ}C$  produced a manganese concentrate of 40.8 per cent Mn and 5.0 per cent Fe. This concentrate contained 79.6 per cent of the manganese in the original ore. If gravity separation had been

applied to the ore prior to gaseous reduction, the grade of this concentrate would have been increased.

Preliminary flotation tests produced low grade concentrates contaminated by iron oxide.

A leaching test was made on the original ground ore with sulphur dioxide, and leaching tests were made on reduced ore with sulphuric acid, nitric acid and ammoniacal ammonium carbonate with the results shown in Table 16.

TABLE 16: LEACHING TESTS ON ORIGINAL GROUND ORE AND REDUCED ORE

Reagent	Extraction		Mn:Fe Ratio in Leach Liquor
	Mn, %	Fe, %	
SO <sub>2</sub>	14.5	0.0	-
H <sub>2</sub> SO <sub>4</sub> (10.9% excess)	88.0	4.9	80.0
H <sub>2</sub> SO <sub>4</sub> (40 % excess)	96.1	35.1	12.7
HNO <sub>3</sub> (10.6% excess)	88.2	0.18	490
HNO <sub>3</sub> (40 % excess)	96.6	28.8	15.1
NH <sub>3</sub> -(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>	31.4	-	-

No cheap method of beneficiation was found to produce a concentrate which assayed in excess of 46 per cent manganese and which had a Mn to Fe ratio greater than 6. If further work is warranted on this ore it is suggested that flotation, and leaching with HNO<sub>3</sub> should be examined more fully.

### 3.2 Material Examined

Two 44-gallon drums of ore were received. The ore was approximately minus 1 inch in sizing. A mineralogical examination (see Appendix B) was carried out on ore crushed to minus  $\frac{3}{8}$  inch and minus 10 mesh.

The ore assayed:

32.7 per cent Mn  
6.3 per cent Fe

### 3.3 Equipment

The following equipment was used:

Screen, Warill:	20 in. dia
Magnetic separator, Davis tube	
Rod mill, laboratory:	10 $\frac{1}{2}$ in. long x 7 $\frac{5}{16}$ in. dia
Flotation cell, Fagergren:	2.5 litre capacity
Gas fired furnace with continuous weighing mechanism	
Laboratory sieves:	BSS

### 3.4 Experimental Procedure and Results

#### 3.4.1 Sizing Analysis of the Ore

Approximately 500 lb of the ore as received was screened into fractions. The fractions were assayed, with results shown in Table 17.

TABLE 17: SIZING ANALYSIS OF THE ORE

Fraction	Weight %	Assays		Distribution			
		Mn, %	Fe, %	Mn, %	Fe, %		
- 1	+ 1 in. + $\frac{1}{2}$ in.	1.7 26.7	28.4	45.5	4.8	38.5	20.7
- $\frac{1}{2}$ in.	+ 10 mesh	44.5					
- 10	+ 52 mesh	15.2	20.2	8.4	9.1	19.5	
- 52 mesh		11.9	12.2	7.9	4.3	14.4	
Feed (calc)	100.0		33.6	6.6	100.0	100.0	
Feed (assay)	-		32.7	6.3	-	-	

#### 3.4.2 Magnetic Separation of Ore after Gaseous Reduction

A wet magnetic separation test, using a Davis tube, was carried out on ore which had been gaseously reduced with a mixture of CO-CO<sub>2</sub> (see Section 3.4.4). The reduced ore ground to minus 72 mesh was separated in the Davis tube operated under the conditions given in Table 18. Results are shown in Table 19.

TABLE 18: OPERATING CONDITIONS FOR DAVIS TUBE

Coil current:	amp	3.1
Water flow-rate:	ml/min	1100
Tube movement:	cycles/min	80

TABLE 19: MAGNETIC SEPARATION OF REDUCED ORE

Fraction	Weight %	Assays		Distribution	
		Mn, %	Fe, %	Mn, %	Fe, %
Magnetic concentrate	20.2	29.6	20.4	15.4	50.5
Magnetic middling <sup>(a)</sup>	4.5	43.2	6.3	5.0	3.5
Magnetic tailing	75.3	40.8	5.0	79.6	46.0
Reduced feed (calc)	100.0	38.7	8.2	100.0	100.0
Reduced feed (assay)	-	38.2	8.5	-	-

(a) A middling fraction was made because the end point of the separation was not definite. Material from the magnetic fraction in the Davis tube was continually rejected or "shed", and this material has been termed middling.

### 3.4.3 Flotation Tests

The dump material crushed to minus 10 mesh was ground in a rod mill under the following conditions:

Time of grinding, min	15
Pulp density, % solids	60
Ore charge, g	1000
Rod mill size,	10 <sup>1</sup> / <sub>2</sub> in. long x 7 <sup>5</sup> / <sub>16</sub> in. dia
Number of rods, 1 in. dia	15
Rod mill, rpm	67

The ground pulp had the sizing shown in Table 20.

TABLE 20: SIZING OF GROUND PULP

Size Fraction				Weight
Microns		Mesh, BSS		%
	+ 300		+ 52	0.1
- 300	+ 210	- 52	+ 72	0.4
- 210	+ 150	- 72	+ 100	2.4
- 150	+ 105	- 100	+ 150	16.8
- 105	+ 75	- 150	+ 200	10.8
- 75		- 200		69.5
				100.0

An emulsion of an anionic flotation reagent mixture was used in the two preliminary tests. This mixture consisted of:

	<u>Parts by Weight</u>
Pamak 4	5.0
Fuel oil	10.0
Triton X	0.5
Naphthenic acid	1.0
	16.5

Other test conditions were as follows:

Test 1. Two reagent additions of 4 and 2 lb per ton were added. The pulp was conditioned for 5 minutes after each addition. Flotation times for the two stages were 13 and 6 minutes respectively.

Test 2. The ground pulp was deslimed twice by decantation (at 18 microns quartz equivalent) using  $\text{Na}_2\text{SiO}_3$  (10 lb/ton) for dispersion. Five stage additions of 1 lb per ton of flotation reagent were added, conditioned for 3 minutes and floated for 4 minutes. A sixth addition of 2 lb per ton was conditioned for 6 minutes and floated for 6 minutes.

Adelaide mains water and the natural pH were used for both tests, which were carried out at 23°C. A Fagergren flotation cell of 2.5 litres capacity, having an impeller speed of 1600 rpm, was used.

The results are given in Table 21.

TABLE 21: RESULTS OF PRELIMINARY FLOTATION TESTS

Test No.	Fraction	Weight %	Assay Mn, %	Distribution Mn, %
1	Rougher concentrate 1	51.0	28.6	45.2
	Rougher concentrate 2	31.7	38.7	38.0
	Tailing	17.3	31.3	16.8
	Feed (calc)	100.0	32.3	100.0
2	Slime	15.8	20.8	9.8
	Rougher concentrate 1	13.7	26.5 <sup>(a)</sup>	10.8
	- ditto - 2			
	" 3	10.8	38.6	13.5
	" 4	14.5	40.8	17.6
	" 5	14.2	42.6	18.0
	" 6	24.9	37.8	28.0
	Tailing	6.1	13.0	2.3
Feed (calc)	100.0	33.6	100.0	
Feed (assay)	-	32.7	-	

(a) This fraction also assayed 4.55 per cent total Fe.

#### 3.4.4 Leaching Tests

Leaching with Sulphur Dioxide. Leaching with SO<sub>2</sub> was done as described in Section 2.4.6. Heat of reaction was very slight. The weight of dry residue was 44.9 g, i.e. 10.2 per cent of the sample dissolved during leaching. The extraction of manganese was 14.5 per cent. No iron was extracted.

Reduction of Ore. About 1 kg of the sample (minus 72 mesh) was pelletised and reduced as described in Section 2.4.6. The reduction time was 6 hours. The total loss in weight was 15.15 per cent. The loss in weight on heating the unreduced pellets in air for 2 hours at 800°C was 9.48 per cent, so that loss due to reduction was 5.67 per cent. The original and reduced materials were assayed for iron and manganese with results as follows:

	<u>Mn, %</u>	<u>Fe, %</u>
Original material	32.7	6.3
Reduced material	38.2	8.5

Leaching of Reduced Ore with H<sub>2</sub>SO<sub>4</sub>. Leaching with H<sub>2</sub>SO<sub>4</sub> was carried out as described in Section 2.4.6. Two tests were made:

- a. With an excess of H<sub>2</sub>SO<sub>4</sub> of 10.9 per cent of the theoretical quantity required to react with all the manganese present, a manganese extraction of 88.0 per cent and an iron extraction of 4.9 per cent were obtained. Heat of reaction caused the temperature of the reaction mixture to rise to about 60°C. The pH of the leach liquor was 3.8, the ratio of Mn to Fe in the leach liquor was 80.0, and the amount of sample dissolved by leaching was 36.9 per cent.
- b. With a H<sub>2</sub>SO<sub>4</sub> excess of 40 per cent the following results were obtained:

Manganese extraction	96.1 %
Iron extraction	35.1 %
pH of leach liquor	2.5
Ratio Mn:Fe in leach liquor	12.7
Loss in weight on leaching	63.6 %

Leaching of Reduced Ore with HNO<sub>3</sub>. Leaching with HNO<sub>3</sub> was carried out as described in Section 2.4.6. Two tests were made, with results as follows:

	a.	b.
Excess of HNO <sub>3</sub>	10.6 %	40 %
Mn extraction	88.2 %	96.6 %
Fe extraction	0.18%	28.8 %
pH of leach liquor	3.8	1.5
Ratio Mn:Fe in leach liquor	490	15.1
Loss in weight on leaching	46.4 %	64.4 %

Heat of reaction raised the temperature of the reaction mixture to about 90°C in test (a) and to about 112°C in test (b). Filtration after leaching was very slow in test (b).

Ammoniacal Leaching of Reduced Ore. The following mixture:

reduced sample	50 g
ammonium carbonate	33 g
strong NH <sub>3</sub> solution	(15.2N) 124 ml

was stirred for 4 hours and filtered. The molar ratio of NH<sub>3</sub> to CO<sub>2</sub> in the mixture was 6, and the ammonium carbonate used was in excess by 20 per cent of that theoretically required to react with all the manganese. Manganese extraction was 31.4 per cent.

### 3.5 Discussion

This manganese ore consisted of manganese oxides, goethite, quartz, clay and carbonates (see Appendix B). The ore assayed 32.7 per cent Mn and 6.3 per cent Fe. The manganese oxides present are pyrolusite, ramsdellite and an unknown manganese oxide. The majority of the manganese minerals is not free at minus 10 mesh. Composites of manganese oxides and coarse goethite are rare, the goethite being generally associated with the gangue minerals. Portion of the quartz, clay and carbonates were liberated in ore ground to minus 10 mesh.

A sizing analysis of the ore as received (see Table 17) showed that the coarser fractions were higher in manganese and lower in iron than the original ore. Screening of the ore on a  $\frac{1}{2}$  inch aperture screen produced an oversize fraction assaying 45.5 per cent Mn and 4.8 per cent Fe. This fraction contained 38.5 per cent of the manganese in the whole ore. The Mn to Fe ratio indicates that this material should be satisfactory for the production of ferromanganese. However, because the manganese content is below 46 per cent, some producers of ferromanganese may deem the material unsatisfactory. Because of insufficient funds the presence of penalty constituents such as  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{CaO}$ ,  $\text{MgO}$ , and phosphorus was not determined. Size fractions finer than  $\frac{1}{2}$  inch were of much lower grade and contained undesirable amounts of iron.

A magnetic separation test (see Table 19) conducted on a sample of ore which had been gaseously reduced at  $750^\circ\text{C}$  by  $\text{CO-CO}_2$  showed that 50.5 per cent of the iron could be removed as a magnetic concentrate. A manganese concentrate which contained 79.6 per cent of the total manganese was obtained. The manganese concentrate assayed 40.8 per cent Mn and 5.0 per cent Fe (hence Mn to Fe ratio is 8.2). For sale purposes a ratio of Mn to Fe in excess of 6 is generally required before material is acceptable for production of ferromanganese. The concentrate assayed only 40.8 per cent Mn and therefore such a material may be unacceptable to the present consumers of manganese ores.

If gravity separation had been applied to the ore prior to agglomeration - gaseous reduction and magnetic separation, a final manganese concentrate of higher grade would have been obtained.

Dry magnetic separation tests were not conducted on the original ore because such tests on the Peak Hill ore gave unsatisfactory results. Both ores are of a similar nature and contain iron minerals which are intimately associated with the manganese minerals and hence are not readily separable from them.

Preliminary flotation tests (see Table 21) on ore ground to 69.5 per cent minus 200 mesh were carried out. The stage addition of 8 pounds of an anionic flotation reagent, Test 2, produced a flotation tailing of only 13.0 per cent manganese. The combined rougher concentrates 1 and 2 assayed 4.55 per cent Fe and it can therefore be concluded that the goethite floated with the manganese minerals and hence was the cause why the rougher concentrates did not exceed 42.6 per cent manganese. The Mn to Fe ratio

in the flotation concentrates could be readily altered by prescreening fines from the ore prior to grinding and flotation. The production, however, of flotation concentrates containing more than 46 per cent manganese could only be achieved with a low recovery of the manganese.

Direct leaching of the ground ore with  $\text{SO}_2$  was not successful. This is in marked contrast to the behaviour of Peak Hill ore and the reason for the difference is not known. It should however be pointed out that, owing to lack of funds, only one leaching test with  $\text{SO}_2$  could be made.

Ammoniacal leaching was more successful than in the case of Peak Hill ore, but the extraction of manganese was too low (31.4%) to warrant further investigation of this process.

Leaching of reduced ore with  $\text{H}_2\text{SO}_4$  and with  $\text{HNO}_3$  was less successful than in the case of the Peak Hill sample. A manganese extraction of 88 per cent was obtained by using an excess of acid of 10 per cent; with an excess of acid of 40 per cent, the manganese extraction rose to 96 per cent. The high recovery obtained by using a large excess of acid is however probably outweighed by the disadvantages of such a process, namely the difficulty of filtration after leaching, and the large amount of gangue minerals dissolved. Of the leaching agents examined  $\text{HNO}_3$  appears to be the most satisfactory, since at the same level of excess acid it extracted as much manganese as  $\text{H}_2\text{SO}_4$  did, but less iron.

### 3.6 Conclusion

No cheap method of beneficiation was found to produce a concentrate which assayed in excess of 46 per cent manganese and which had an Mn to Fe ratio greater than 6.

If further work is warranted on this project it is recommended that flotation and leaching with  $\text{HNO}_3$  be examined more fully.

## APPENDIX A

### MINERALOGICAL EXAMINATION OF PEAK HILL MANGANESE ORE

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ML2510: PS7594-7597. A number of small lumps of Peak Hill dump material was received; several were briquetted in an endeavour to obtain a better idea of the nature of the material.

The lumps which appear dense, massive and of dark steel-grey colour in the hand-specimen consist of massive, ultra-fine-grained pyrolusite with colloform structure. Patches, veinlets and aggregates of slightly coarser pyrolusite occur within the masses, usually with fibrous-radiating to microgranular habit. Occasional small patches, and veins up to 2-3 mm wide, of goethite with fibrous to colloform or earthy habit are included in this material.

The dark-grey material with "sintered" or "brecciated" appearance in the hand specimen consists of very cellular, fine-grained pyrolusite; many of the cavities, which are from 2 mm to 0.1 mm or less in diameter, are filled with earthy goethite.

The reddish earthy to dark-brown oolitic material in the hand specimen consists essentially of goethite in various forms with little or no manganese. The goethite is colloform, earthy, and oolitic to pisolitic. It is thought that fine-grained clay minerals and possibly silica are common in this type of material.

The fourth type of sample consists in the hand specimen of well-defined oolitic goethite with irregular patches of manganese material. These patches are up to 2 cm wide and can be several centimetres long. Microscopic examination shows the sample to consist of goethite ooliths, some with cores of lepidocrocite (?), and irregular, colloform-banded, very fine-grained pyrolusite. The pyrolusite has evidently been deposited after the goethite, since pyrolusite occurs interstitially between the goethite ooliths. The patches of pyrolusite generally contain goethite as lenses, layers and patches of various sizes from 0.005 mm upwards. It is thought that this material will be difficult to treat for that reason.

The minus 10-mesh sample ML2512 (PS7609) appears to be quite well liberated, due probably to the toughness of the fine-grained pyrolusite and the friable nature of the goethite. In fact most of the manganese occurs as free grains of pyrolusite (usually the ultra-fine-grained variety), except for a few grains evidently derived from intimately intergrown pyrolusite-goethite ore. Gangue occurs as occasional grains of quartz up to 0.3 mm across and as irresolvable material in the goethite.

It is thought at this stage that, whilst some of the composites could be freed by finer grinding, the gain may not be great. It is also felt that there may be a good chance that the bulk of the gangue will be associated with the goethite and in the fines. Restricted grinding and sizing may produce a good fraction.

Investigation and Report by: H. W. Fander

Officer in Charge, Mineralogy Section: H. W. Fander

## APPENDIX B

### MINERALOGICAL EXAMINATION OF WOODIE WOODIE MANGANESE ORE

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#### Report MP276/64

Minus  $\frac{3}{8}$ -inch to Plus 10-mesh. These fragments consist predominantly of fine-grained manganese oxide minerals — probably pyrolusite with minor ramsdellite and an unknown oxide - and gangue constituents. Quartz gangue is usually composite with or included in the manganese mineral fragments. Clay and carbonate minerals occur as aggregates ranging in diameter from approximately 1.5 mm to several centimetres. Clay minerals also appear to coat manganese-rich particles.

Minus 10-mesh. The predominant minerals are the manganese oxides, pyrolusite (?) is present as coarse- or fine-grained aggregates some showing a radiating structure. Fine-grained pyrolusite aggregates are mostly massive but a few particles were observed which consisted of fine, prismatic pyrolusite (?) in quartz (?) gangue. Quartz also occurs in other pyrolusite-rich particles. Several coarse, prismatic aggregates of ramsdellite and coarse-grained aggregates of the unknown oxide are present in the sample. As in the coarser fraction of this ore, fine-grained aggregates of clay minerals are present. Goethite occurs as colloform aggregates associated with other gangue but only rarely associated with the manganese minerals. Quartz grains are a minimum of 0.005 mm in diameter in some pyrolusite but generally are greater than 0.01 mm.

Ramsdellite was identified by x-ray diffraction methods. The unknown manganese oxide was not named in the ASTM index, but is near pyrolusite in composition.

#### BENEFICIATION

The majority of the manganese minerals are not free but are associated with gangue constituents. Rarely are they composite with coarse goethite. Coarser clay or carbonate aggregates which occur could probably be separated by heavy liquid separation of the minus 10-mesh material. However, finer grinding would be required for complete separation of the manganese and gangue minerals.

X-ray Diffraction: N. A. Trueman

Investigation and Report by: D. E. Ayres

Officer in Charge, Mineralogy Section: H. W. Fander