Amended.

COMMONWEALTH OF AUSTRALIA.

DEPARTMENT OF NATIONAL DEVELOPMENT.

BUREAU OF MINERAL RESOURCES
GEOLOGY AND GEOPHYSICS.

RECORDS:

1964/106

RUM JUNGLE PHOSPHATE PROJECT LABORATORY INVESTIGATIONS 1962/64

Compiled by

P.W. Pritchard

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LABORATORY INVESTIGATIONS 1962-64.

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Records 1964/106

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INTRODUCTION

The phosphate rock found at Rum Jungle in 1961 has been examined and tested in various ways by several organisations. The results of this work have been scattered throughout several Bureau of Mineral Resources files and they are compiled in this report so that they will not be overlooked during future assessments of the deposit.

The mineralogical report forming section 4 of part 2 and the beneficiation tests reported in section 1 and 2 of part 4 were supplied by Esso Standard Oil (Australia) Limited; their permission to release this information is greatly appreciated.

PART 1.

CHEMICAL RESULTS

Following the recognition of phosphate rock in the Rum Jungle area at the end of 1961 five samples were submitted to the B.M.R. Canberra laboratory for partial analysis.

Table 1.

ANALYSES OF RUM JUNGLE PHOSPHATE ROCK

Sample	SiO_2 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	P_2O_5 (%)	F (%)	TiO_2 (%)	Ignition loss at 900°C (%)
A. D550, 20'-41'6"	18.68	20.28	23.80	0.50	17.25	0.75	0.90	5.50
B. D550, 46'6"-49'	9.58	15.17	35.06	n.d.	24.60	1.89	0.80	2.70
C. Geolsec.	0.54	4.63	49.26	n.d.	37.40	3.0	<0.05	2.10
D. D555 92'	2.54	5.27	35.34	13.10	14.53	2.86	<0.05	25.58 (CO_2 23.3)
196211	0.04	6.23	50.37	n.d.	38.85	3.10	<0.05	0.45

1. Analyst : S. Baker - B.M.R. Laboratory number 62/1082
2. A, B and D are composited spot samples from diamond and auger drill holes. Sample C is composited from surface samples taken at 26E 12.7N, 25E 14.31N, 25E 15N at Geolsec. Sample 196211 is a surface sample from an apatite vein at Stapleton North.
3. Ignition loss includes organic matter, combined water and CO_2.
4. n.d. means not detected, i.e. <0.2%.

In September 1962 four drums of phosphate rock from Costean 1 at Geolsec. were submitted to the Australian Mineral Development Laboratories for assay and mineralogical determinations. (See the mineralogical report by Trueman and Ayres on this material).

Table 2.

ANALYSES OF RUM JUNGLE PHOSPHATE ROCK
COSTEAN 1. GEOLSEC.

Sample		Analysis as received						
			Acid Soluble					
	P_2O_5 (dry basis) (%)	Moisture (%)	P_2O_5 (%)	Fe_2O_3 (%)	Al_2O_3 (%)	CO_2 (%)	F (%)
484/1 0'-90'	11.85	2.11	11.60	21.0	25.8	0.10	0.70
484/2 265'-305'	12.60	3.37	12.15	17.9	24.8	0.64	0.96
484/3 305'-320'	22.75	0.49	22.65	13.1	6.2	0.03	2.44

Sample	P_2O_5 (dry basis) (%)	Analysis as received Moisture (%)	P_2O_5 (%)	Acid Soluble Fe_2O_3 (%)	Acid Soluble Al_2O_3 (%)	CO_2 (%)	F (%)
484/4 459'-462'6"	37.35	0.42	37.20	3.40	2.65	0.21	5.40

Analysts : M. R. Hanckel, T. Francis, (A.M.D.L. 5.11.62)

In September 1962 duplicate samples of material assayed by A. G. Fricker in the B.M.R. field laboratory at Coomalie Creek were sent to A.M.D.L. for check assays for P_2O_5 and for further information on the iron, aluminium, fluorine and carbonate content of the Rum Jungle material. The results of these assays with Fricker's P_2O_5 assays in brackets for comparison are shown in Table 3.

Table 3.

ANALYSES OF RUM JUNGLE PHOSPHATE ROCK

Sample *	%P_2O_5 (dry basis)	Analysis as received Moisture (%)	P_2O_5 (%)	Acid Soluble Fe_2O_3 (%)	Acid Soluble Al_2O_3 (%)	CO_2 (%)	F (%)
A45,0'-2'	14.00 (14.7)	0.92	13.9	18.0	4.90	0.19	1.26
2'-4'	24.90 (25.3)	0.70	24.70	9.7	5.00	0.30	2.19
A117,20'-24'	11.70 (12.2)	3.78	11.30	7.3	9.4	0.18	1.04
24'-26'	14.60 (14.9)	2.74	14.20	6.9	7.1	0.23	1.41
A1027,32'-42'	10.30 (10.7)	1.79	10.10	6.1	11.7	0.22	1.13
42'-50'	14.50 (15.2)	1.38	14.30	4.80	9.6	0.72	1.32
D550,30'-35'	14.70 (14.5)	1.72	14.45	21.4	8.4	0.25	1.44
35'-40'	16.30 (16.6)	1.22	16.10	25.0	8.2	0.28	1.52
40'-45'	19.90 (18.8)	0.96	19.70	17.7	7.6	0.39	1.87
45'-50'	23.65 (24.3)	0.67	23.45	19.4	6.1	0.50	2.17
D530,35'-38'	15.55 (15.3)	1.38	15.30	33.0	8.3	0.14	1.25
58'-63'	20.85 (21.3)	1.28	20.60	31.6	5.0	0.21	1.84

[illegible]

G/C1, 450'-455'	13.6 (11.0)	1.73	13.35	17.4	9.0	0.14	1.12
459'-460'	33.0 (31.8)	1.86	32.40	5.0	7.3	0.30	2.82
G/C2, 82'-83'	17.95 (18.6)	1.34	17.75	5.2	13.7	0.17	1.59
83'-86'	33.25 (33.8)	0.46	33.10	6.8	4.45	0.27	3.08
A3/C1, 127'-130'	34.70 (35.8)	0.37	34.55	5.5	4.10	0.31	3.21
132'6"-137'	13.45 (13.3)	1.59	13.25	20.3	10.1	0.14	1.26
DG1, 62'3"-67'10"	15.10 (15.4)	0.13	15.10	4.7	2.20	0.22	1.53
82'3"-87'	31.05 (30.9)	0.10	31.00	3.55	1.90	0.41	3.06
* 87'-9'1"	30.70 (31.5)	0.08	30.65	3.00	2.15	0.40	3.04
111'3"-116'	29.30 (29.7)	0.11	29.30	5.1	2.90	0.43	2.88
116'120'9"	25.20 (26.4)	0.10	25.15	3.85	2.85	0.35	2.75
173'6"-178'4"	21.5 (22.4)	0.18	21.50	8.0	5.3	0.25	2.05
222'9"-227'7"	19.85 (19.9)	0.28	19.80	13.6	5.9	0.23	1.86

Analysts : T.Francis and M. R.Hanckel. A.M.D.L. (5. 11. 62)

P_2O_5 analyses in brackets A. G.Fricker. B.M.R. 1962.

* A45 - Auger drill hole

D550 - Territory Enterprises Pty. Ltd. diamond drill hole

DG1 - Bureau of Mineral Resources diamond drill hole

G/C1 or A3/C1 - Bureau of Mineral Resources costeans

Standard Oil Company of New Jersey had the following analyses made in September 1962.

Table 4.

ANALYSES OF RUM JUNGLE PHOSPHATE ROCK(1)

Sample (2)	%P_2O_5	%Fe_2O_3	%Al_2O_3	%CaO	%SiO_2	%F
3 30' from DG1	0.28	3.67	2.98	0.53	90.83	0.007
4 Costean C1	32.16	3.97	1.67	50.77	1.62	3.31
5 Costean C1(3)	31.05	13.10	7.69	35.61	6.38	2.37
6 DDG1, 191'	4.60	5.19	2.49	6.15	80.3	n.d.
7 Buckshee	32.30	5.20	3.05	47.56	3.70	2.99
9 Auger hole 56E 10N 30-40'	0.15	20.76	14.68	2.27	33.66	0.20

1. Analyses for Standard Oil Co. Inc. These are confidential.
2. Samples 3,4,5,7,9 were assayed by Ledoux and Co. New Jersey. Sample 6 was assayed by J.P.R.
3. X-ray powder diffraction pattern shows apatite and wavellite.

The beryllium content of the phosphate rock from the Castlemaine Hill area is given in Table 5.

Table 5

BERYLLIUM IN RUM JUNGLE PHOSPHATE ROCK

Prospect	Number of samples	Average Be content p.p.m.
Easticks	2	10
Geolsec	6	13
Area 3	1	8
Area 4	3	10

Analyses by A. D. Haldane (B.M.R.)

PART 2.

MINERALOGICAL REPORTS

1. HAEMATITE-BEARING APATITE ROCK FROM THE "GEOLSEC" RADIOMETRIC ANOMALY, RUM JUNGLE AREA, NORTHERN TERRITORY

by

W.M.B. Roberts, S. Baker and W.B. Dallwitz

Introduction

Two samples (Nos. 195932 and 195933) of slightly radio-active material from the "Geolsec" radiometric anomaly, Rum Jungle area, were submitted by the Rum Jungle Geochemical Party at the request of the A/Asst. Chief Geologist, for mineralogical and petrographic examination. Sample No. 195932 was of drill cuttings and sample No. 195933 consisted of small rock fragments taken from an outcrop.

Petrographic, X-Ray and Chemical Results

Specimen No. 195933 is a massive, fine-grained, purplish brown rock with pockets and encrustations of a white to pale brown mineral. The brown colouration is due to inclusions of fine-grained iron oxide. Encrustations of this mineral may take the form of groups of small rosettes made up of fine acicular crystals; these crystals were identified as apatite by X-ray powder photography.

A thin section (No. 7670) of Specimen 195933 (Registered Rock No. R9415) was found to consist almost entirely of apatite (90 to 95%) and haematite (5 to 10%). Very small amounts of hydrated iron oxide had been formed from the haematite in places. The apatite is mostly granular (grain size 0.015 mm to 0.25 mm), but some of it has a fibrous-radiating, colloform structure. The grain size of the haematite ranges from dust-sized particles up to about 0.15 mm. Some of the fibrous-radiating apatite is densely packed with dusty haematite. None of the apatite is free from haematite.

A small fragment of the specimen from which the section was cut was found to contain 36.8% P_2O_5.

Parts of samples No. 195932 and No. 195933 were examined on the X-ray spectorgraph, and the radioactivity (0.044% eU_3O_8 for sample No. 195932) was found to be due almost entirely to uranium. Minor traces of thorium are present in about the quantity usually found with uranium in nature.

Attempts to isolate a separate uranium mineral from sample No.195933 were unsuccessful. A pinkish fibrous-radiating mineral, which was separated from the crushed rock, was identified by X-ray diffraction as a mineral of the apatite group. An X-ray powder photograph of crushed material representing the whole rock showed that it was made up almost entirely of the same mineral.

A sample (Field No. F25E/15N/0) taken from the same general area had previously been chemically analysed by S. Baker and was found to contain 39.5% P_2O_5, 50.3% CaO, 3.6% Fe_2O_3, less than 0.5% SiO_2 and no sulphate. Microscopic examination of this material showed that it consists of apatite and minor amounts of haematite, and an X-ray diffraction analysis confirmed that the principal constituent is a mineral of the apatite group.

2. REPORT OF INVESTIGATIONS OF PHOSPHATE ROCK FROM THE GEOLSEC DEPOSIT NORTHERN TERRITORY.

by

N.A. Trueman and D.E. Ayres *

* Australian Mineral Development Laboratories Adelaide.

(See the assays in Table 2 Part 1 of this record.)

General

Two specimens of different appearance were selected from each sample to determine the major constituents, the mineral association and grainsizes of the phosphate minerals. X-ray diffraction methods were required for complete mineral identification because of the fine-grained nature of the material and the probability that phosphate minerals other than apatite were present.

Summary

Apatite is the major phosphate mineral present. Phosphate minerals which occur in minor amount are millisite, a member of the variscite-strengite series, and wavellite. Other minerals, present in varying amounts are quartz, a kaolinite group mineral, sericite or illite, haematite and probable goethite. For simplicity, throughout the report the kaolinite group mineral is termed kaolinite.

As can be seen from the descriptions, there is often considerable variation between the two specimens from each sample. The high grade material has the most even composition.

Table 1

MINERAL CONTENT

RUM JUNGLE PHOSPHATE ROCK - COSTEAN 1

Sample No.	Major mineral.	Minor minerals.
484/1 (0-90')	kaolinite	wavellitem millisite
484/2 (265-305')	millisite	variscite-strengite, kaolinite, wavellite, sericite or illite, quartz.
484/3 (305'-320')	apatite	quartz, kaolinite, millisite.
484/4 (459'-462'6")	apatite	quartz, millisite, sericite or illite.

Formulae of the less common phosphate minerals are:-

millisite - $Na_2O.2CaO.6Al_2O_3.4P_2O_5.15H_2O$

variscite - $AlPO_4.2H_2O$) end members of the series

strengite - $FePO_4.2H_2O$)

wavellite - $Al_3(OH)_3(PO_4)_2.5H_2O$

Reference:-
"Dana's System of Mineralogy", Vol. II, 7th Edit., 1951.

Detailed Descriptions

Sample 484/1 : 0 to 90 feet

Specimen A: This is a reddish-brown, nodular aggregate consisting predominantly of kaolinite and millisite with occasional, angular quartz grains and fine iron oxide (probably goethite). The colour of the specimen is caused by the iron oxide.

The kaolinite and millisite are extremely fine-grained (less than 30 microns in diameter) and can only be differentiated by X-ray diffraction techniques.

Specimen B: This is an extremely fine-grained aggregate of kaolinite, wavellite, millisite and iron oxide (probably goethite). Generally the kaolinite, wavellite and millisite are intimately associated, however, some millisite is also present as rims, less than 20 microns thick, bordering several cavities in the specimen.

All grains are less than 30 microns in diameter.

Sample 484/2 : 265 to 365 feet

Specimen A: This is a porous, fine-grained aggregate of millisite, kaolinite, sericite (or illite), quartz and iron oxide (probably goethite). There are actually two different coloured aggregates present in the specimen. One white, the other red-brown. Microscopically it appears that the only difference between the two is that iron oxide is more abundant in the red-brown aggregate.

Specimen B: This is a rather more porous aggregate than A. White, compact, fine-grained aggregates of variscite-strengite, wavellite and kaolinite are cemented by porous, red-brown material similar to the red-brown material in A.

Sample 484/3 : 305 to 320 feet

Specimen A: This is a brecciated rock consisting predominantly of fine-grained apatite with minor amounts of quartz and haematite. The haematite is extremely fine-grained and occurs interstitially to, or as minute inclusions in, the apatite. Several fractures cutting the rock contain fine-grained, yellow-brown iron oxide (probably goethite) and also brownish apatite.

Apatite grains range from 1 or 2 microns to 360 microns in diameter. Most grains are less than 65 microns in diameter.

Specimen B: This sample consists predominantly of angular quartzite fragments, ranging up to several centimetres in diameter, and occasional apatite fragments, cemented by a fine-grained aggregate of kaolinite, millisite, quartz, apatite and yellow-brown iron oxide (probably goethite).

Apatite grains are less than 65 microns in diameter. Kaolinite and millisite grains are mostly less than 5 microns in diameter and are intimately associated.

Sample 484/4 : 459 to 462 feet

Specimen A: This is a massive, crystalline apatite rock containing a minor amount of anhedral quartz grains. Apatite is the predominant constituent. Numerous minute haematite inclusions colour it red. The apatite grains range from fine to medium in grainsize and the coarsest grains show growth zoning. Aggregates of apatite show concretionary banding and radial crystallisation. The bands are often fractured and, commonly, the fractures are filled by later apatite. Several finer-grained apatite aggregates are rimmed by the banded type. Quartz occurs as scattered anhedral grains or as aggregates of several grains. A fine-grained micaceous mineral, possibly sericite, occurs with some of the fine-grained apatite.

Apatite grains range from several microns to 700 microns in diameter and quartz from about 5 microns to 600 microns. The majority of the haematite grains are 1 or 2 microns in diameter but haematite occurring interstitially ranges up to 150 microns diameter.

Specimen B: This is a brecciated altered apatite rock which was originally fairly similar to specimen A. Quartz is much less abundant but the proportion of micaceous minerals is higher. Apatite is the predominant mineral and has similar features and grainsize to that in A. Fine-grained, white aggregates occurring in fractures in the rock are composed of apatite and millisite.

Bulk Sample

The specimens from this sample vary in composition from brecciated quartzite with minor apatite to a predominantly apatite-rich rock, i.e. from approximately 25 per cent apatite, 75 per cent quartz to 75 per cent apatite, 25 per cent quartz and micaceous minerals. The bulk sample is thus generally similar to sample 484/4.

Apatite grains in the specimens range from 1 or 2 microns to approximately 700 microns in diameter. Both the finer and coarser-grained banded aggregates are brecciated. The fragments are cemented by later apatite and minor amounts of sericite or illite. Most of the apatite contains abundant haematite inclusions which range from less than 1 micron to approximately 100 microns in diameter. Quartz occurs as anhedral grains or granular aggregates ranging from approximately 10 microns to 1.5 millimetres in diameter. Sericite (or illite) is a common constituent of fractures in the brecciated specimens.

3. PETROGRAPHIC EXAMINATION OF RUM JUNGLE APATITE ROCK

by

W.R. Morgan

Herewith are the results of a detailed petrographic examination of four samples of apatite rock collected in the Rum Jungle area, N.T. The specimens examined are:-

R.10792 - D.D.H., 555, at 92 feet.
R.12824 - D.D.H., D.G.1, at 119 feet, 4 inches.
R.12825 - D.D.H., D.G.1, at 31 feet, 5 inches.
R.12826 - D.D.H., D.G.1, at 86 feet, 6 inches.

Generalized descriptions of R.12824 to R.12826 can be found in my earlier report (file 64NT/1, 29th November, 1962). Specimen R.10792 consists mostly of a mosaic of fine apatite crystals together with granules and flakes of hematite; the hematite is enclosed within apatite crystals, and is not interstitial. Small areas of heavily ferruginized argillaceous material are surrounded by the fine apatite. The rock is cut by very irregular veins that are filled with calcite, subordinate chlorite, and small amounts of coarse apatite and opaque iron oxide. In these veins, the opaque iron minerals form crystals discrete from the coarse apatite.

The table shows the grain-sizes and modal analyses of the specimens. The modal analyses should be regarded as inaccurate for two main reasons. Firstly, because of the extremely fine-grained nature of the rocks: the grain-sizes are smaller than the thickness of the rock slice (0.03mm.).

TABLE 1

DISTRIBUTION OF APATITE AND HEMATITE IN RUM JUNGLE PHOSPHATE ROCK.

Sample	Grainsize and B.S. seive apertures.						Modal analyses (%).			
	Apatite				Hematite		Apatite		Hematite	Others
	Coarse		Fine				Coarse	Fine		
	Grain-size mm	BS aperture	Grain-size mm.	BS aperture	Grainsize (mm)	BS aperture				
R10792	0.13	100	0.018- 0.045	<300	0.0001- to 0.027	<300	5	34	20	41
R12824	0.02- 0.1	300- 150	0.004- 0.012	<300	0.0005- 0.007	<300	67	25	3	4
R12825	-	-	0.12- 0.036	<300	0.0005- 0.013	<300	-	38	17	45
R12826	0.06- 0.3	300- 45	0.012	<300	0.005- 0.018	<300	37	31	20	12

Secondly, the effect of the hematite; much of it is too fine-grained to be seen properly during point-counting, resulting in an under-estimation of the amount present. However, what can be seen and counted during modal analysis will be over-estimated because of the "Holmes Effect" (Chayes, 1956). Taking these two effects of hematite into account, it is hard to judge whether the ultimate results are over-estimates or under-estimates.

Other minerals are:- R.10792: calcite, chlorite, subordinate quartz; R.12824: quartz, ferruginous argillaceous material, and some sericite; R.12825: ferruginous slate, silty slate, siltstone, and quartzite; R.12826: quartz and mica.

In all specimens, hematite is enclosed within apatite crystals, and not interstitial to them; this statement applies particularly to the finer-grained apatite, although some hematite is enclosed in the coarser apatite.

Specimen R.12824 is the one most free of hematite. The total amount of apatite present is reduced to 62% if the vugs (represented by holes in the slide) are taken into account. Some of the vugs are filled by quartz, and may be more completely filled in other parts of the rock at this horizon.

Reference

Chayes, F., 1956 : The Holmes Effect and the Lower Limit of Modal Analysis. Miner. Mag., 31, 276-281.

4. RUM JUNGLE PHOSPHATE ORE *

by

J.R. Lohr and S.W. Frazier

Here is an informal report covering a preliminary examination which you requested for the two prospect samples of phosphate ore submitted by R.A. MacDonald of Standard Oil. Their field descriptions and analytical data are cited in their letter of September 20, and reference to this information is made in my report (Table 1).

Prospect Sample 4

This rock specimen, which is described as a greyish red, highly phosphatic siltstone, contains fluorapatite as the only significant phosphatic phase and it also represents the bulk mineral constituent. Next in abundance but minor by weight are the iron oxides - mainly micaceous and fibrous hematite with minor goethite. The detrital mineral group consists of quartz, degraded muscovite, tourmaline (var. dravite), epidote, anatase, zircon, and possibly others of less significance. Collectively, the detritals represent an extremely minor weight fraction.

The apatite species appears to be exclusively fluorapatite, judging from its pale green colour, absence of CO_2, and uniform refractive indices (N_o= 1.628, N_o= 1.631). It is present as subhedral to euhedral stubby

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* Confidential report to Standard Oil Co. of New Jersey from the Tennessee Valley Authority U.S.A.

(See the assays in Table 4 of Part 1 of this record.)

hexagonal prisms terminated by pyramidal forms. Crystal faces are smooth and unscarred. Crystals show zonal growth bands (hopper effect), with inclusions of scalar hematite frequently concentrated along these growth bands, or during interruptions in the cyclic precipitation of the apatite crystals. The unit crystals range from silt sizes to a maximum of about 80 microns. They are randomly oriented and lightly interpenetrated, and it is this intergrowth which appears to be the primary cementation in the rock.

The hematite - goethite phases are found in sizes generally not exceeding about 5 microns. The micaceous to fibrous crystals, and loose agglomerates of these crystals, fill the interstices in the apatite mass but do not appear to have contributed significantly to the cementation.

Quartz is found in the unusual form of rough surfaced, skeletal grains that have been severely pitted by chemical erosion. Of the remaining detritals, muscovite shows chemical weathering to hydromuscovite, as indicated by depressed refractive indices, birefringent halos, and the physical condition of the mica scales (deformed, crimped, curled scales often marked by a network of shrinkage fissures).

Prospect Sample 7

This grab sample, which is described as having a similar texture and chemical composition to sample 4, comprises nearly the same suite of minerals as sample 4.

Fluorapatite is the only significant phosphatic phase, and it is also the bulk mineral species in the specimen. Hematite (and possibly some goethite) is the important iron mineral. Quartz is extremely minor, less than that found in sample 4. On the other hand, muscovite in a partially degraded condition is much more abundant than in sample 4, and accounts for most of the SiO_2 and Al_2O_3 reported in the chemical analyses. Of the remaining detritals, tourmaline (var. dravite) was the only phase of any significance.

Again, the fluorapatite exists in a microscopically coarse crystalline form. The denser texture of this specimen is caused by a greater degree of cementation of the apatite crystals. The unit crystals range from silt sizes upward to about 110 microns, and there has been extensive intergrowth during the final stages of cyclic growth. Also because of this more extensive intergrowth, much of the scalar hematite is occluded in the apatite crystals - notably in bands representing interruptions in the cyclic growth process.

The agglomerates of degraded muscovite, residual hematite, and possibly very minor clay are seen to fill the interstitial voids in the apatitic mass.

Discussion

The coarsely crystalline form of the apatite is quite unlike any of the domestic sedimentary phosphates now being exploited. The uniform coarseness of the crystals, their euhedral form, smooth unscarred surfaces, the hyperfine internal growth band structure, and the nature and location of the inclusions clearly indicate that it formed by recrystallization in situ of the original sedimentary phosphorite, collophane. In both samples, remnants of the precursor phosphate were detected in the form of porous, rounded, fine-grained aggregates of collophane (cryptocrystalline apatite) heavily stained with limonite. This hydrolytic alteration appears to have been caused by fluoride-bearing water, since the apatite is now exclusively fluorapatite, and the primary silica phase shows extensive chemical erosion. Recently, a similar example of this type of recrystallization in situ was observed in a deep-seated francolite deposit from a S.A. prospect.

Laboratory studies of the four component system $CaO-P_2O_5-HF-H_2O$ showed that very dilute fluoride solutions attacked various basic calcium phosphates to produce the equilibrium phase, fluorapatite, in periods of time as short as 24 months. The crystalline apatite obtained in these particular equilibrations possessed the same coarse, euhedral form, habits, and hyperfine growth bands as found in samples 4 and 7.

Chemical Analyses : The phase composition of samples 4 and 7 cannot be reconciled closely with the chemical analyses supplied for material reported to be from the same location. The cause for the discrepancy appears to be related to the foregoing discussion.

Both grab samples are essentially binary mixtures of fluorapatite and hematite, but the CaO/P_2O_5 ratio is too high for such a pure form of fluorapatite, and no other calcium phases were detected in significant amounts.

If by chance the analyses were obtained on specimens which had not undergone extensive recrystallization, the presence of a significant fraction of the original phosphatic material (francolite) and the usually associated rhombic carbonates could account for the high CaO/P_2O_5 ratios reported here. On the other hand, we have confirmed that the hematite phase is below 5% in sample 4 or 7. Disregarding this minor amount of iron oxide, the apatite in either sample 4 or 7 approaches the purity of the high-grade igneous apatite deposits of Templeton, Quebec.

Beneficiation : The amount of iron present would probably discourage using these ores directly as electric furnace burden. Calcining would not be expected to cause any significant change in its phase composition. In sample 4 where the apatite is as microscopically coarse crystals, and the very fine-grained hematite is found as weak agglomerates and as free fines largely unoccluded by the apatite, considerable iron oxide could be recovered in laboratory tests by crushing followed by washing, blunging, or sedimentation.

In sample 7, however, the hematite is largely occluded in the apatite crystals, and little iron oxide could be removed by physical methods.

Material similar to sample 4 might also be processed by a modified flotation by depressing the apatite and removing the iron oxides, muscovite, clays, etc., by desliming. Or, a high-grade hematite concentrate might be removed by magnetic or electrostatic separators, using dry crushed ore.

Wet methods of extraction of the phosphatic phase using acid may not be practical because of a problem involving rates of solution. The rate of solution of the apatite is decreased because of its coarsely crystalline form (low specific surface), while the dissolution rate of the iron oxide is increased because of its very fine particle sizes. The two materials could not be spearated in the laboratory by digestion with various acids, but not all acids were tested. The first attempts certainly were not promising.

It is not possible, therefore, to draw firm conclusions as to how the phosphatic rock might be processed or beneficiated, based on the preliminary examination of these two grab samples. The exceptionally high grade of this unusual rock, however, should encourage considerably more exploratory studies.

Table 1.

SAMPLE LOCALITIES - PHOSPHATIC ROCK - RUM JUNGLE.

SAMPLE NO.	LOCATION	STRATIGRAPHIC POSITION	FIELD DESCRIPTION OF SAMPLE
AUST 1	2.0 miles due east of Batchelor.	Coomalie Dolomite, Lower Proterozoic.	Dolomite, grayish red to bluish gray, rhombic to sucrosic. Contains approximately 15% vein-filling dolomite/calcite.
AUST 2	Geolsec Prospect Castlemaine Hill, approx. 100 feet from DDH G.1 of B.M.R. phosphate group.	Upper unit of Depot Creek Sandstone Member; 25 feet above base of member.	Sandstone, grayish-red, fine to medium grained, hematitic.
AUST 3	Geolsec Prospect, Castlemaine Hill, approx. 30 feet from DDH G.1. of B.M.R. phosphate group.	Base of middle unit of Depot Creek. Sandstone Member.	Sandstone, breccia, grayish red, fine grained to pebble sized. Coarser material pale red to white.
AUST 4	Geolsec Prospect, Castlemaine Hill, in trench near DDH G.1 of phosphate group.	In lower (phosphate) bed of Depot Creek Sandstone Member; 20' ± below top of unit.	Siltstone, sandy, clayey, grayish red, highly phosphatic.
AUST 5	Geolsec Prospect, Castlemaine Hill in trench near DDH G.1 of phosphate group.	In lower (phosphate) bed of Depot Creek Sandstone Member; 46' ± below top of unit.	Siltstone, clayey, light to pale brown, highly weathered, highly phosphatic.
AUST 6	Geolsec Prospect, Castlemaine Hill in B.MR. DDH G.1 of phosphate group.	Core in lower (phosphate) unit 191' below surface.	Ss., v.f. - coarse grained, silty, grayish red, contains abdt. fragments of white ss. up to pebble size.

SAMPLE NO.	LOCATION	STRATIGRAPHIC POSITION	FIELD DESCRIPTION OF SAMPLE
AUST 7	Buckshee Prospect, approximately 1 mile west of TEP's Whites openpit uranium mine.	In lower (phosphate) unit of Depot Creek Sandstone Member.	Siltstone, clayey, grayish red (weathered surface lilac coloured), highly phosphatic.
AUST 8	Buckslee Prospect approximately one mile west of TEP's Whites openpit uranium mine.	In upper? unit of Depot Creek. Sandstone Member.	Hematite, medium, light gray, weathered surface rust coloured.
AUST 9	Auger hole 56/10N/30'-40'	In lower (phosphate) unit of Depot Creek Sandstone.	Reddish brown powder, probably from clayey siltstone.

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[1] These samples were collected when the phosphate deposits were thought to occur in the lower part of the Upper Proterozoic Depot Creek Sandstone shown on the B.M.R. Rum Jungle Special 1" : 1 mile geological map.

PART 3

BOTANICAL TESTS

1. REPORT ON POT TESTS WITH SAMPLES OF RUM JUNGLE PHOSPHATE ROCK.

by

M.J.T. Norman[1]

Samples of two types of phosphate-rich rock from the Rum Jungle area were received from the Bureau of Mineral Resources for assay. The samples were :

1. Rum Jungle No. 1.[2] - Pale red colour
2. Rum Jungle No. 2. - Ochre colour

Methods

The assay method adopted was to compare the responses of grain sorghum plants grown in Tippera clay loam (a phosphorus-deficient soil from Katherine, N.T.) to the two phosphates and to Christmas Island rock phosphate dust and superphosphate at 4 levels of fertilizer. The 17 treatments were:

Rum Jungle No. 1) (1 cwt/ac (equivalent)
Rum Jungle No. 2) (4 cwt/ac
x
Christmas Island Rock) (8 cwt/ac
Superphosphate) (16 cwt/ac

Control .... No phosphate

(With 3 replicates)

Fertilizers were milled to a fine powder and mixed with Tippera clay loam soil. Five-inch enamel pots were filled and each planted with 12 seeds of "Hegari" grain sorghum on October 26, 1962. The pots were placed in the greenhouse and watered 2 - 3 times a day. On December 12, all plants were harvested. The tops were cut off at ground level and the roots washed free of soil. Tops and roots were dried separately at 85°C for 24 hours and weighed. The dried samples were milled and analysed for total phosphorus.

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[1] C.S.I.R.O. Division of Land Research and Regional Survey.

[2] Both samples were taken from Costean 1 at Geolsec. Sample 1 came from 459 to 462.5 feet and sample 2 from 260 to 350 feet.

The total phosphorus content of the fertilizers was :

Rum Jungle 1	-	15.2%
Rum Jungle 2	-	5.8%
Christmas Island	-	15.6%
Superphosphate	-	10.5%

159 mgm of fertilizer was mixed in each pot for each equivalent 1 cwt/ac of fertilizer. Percentage recovery of the total phosphorus mixed in each pot, at the 4 cwt/ac level, was therefore :

Rum Jungle 1	-	1.44%
Rum Jungle 2	-	2.90%
Christmas Island	-	2.57%
Superphosphate	-	9.82%

Thus, compared with Christmas Island rock phosphate, Rum Jungle 1 had a similar total P content but gave a substantially lower recovery by plants; Rum Jungle 2 had a low total P content but gave a recovery of the same order as Christmas Island rock. Percentage recovery from superphosphate was higher than that from all other materials.

Conclusion

All the results, dry matter yield, phosphorus content, phosphorus yield and phosphorus recovery, support the conclusions that the amount of phosphorus in the Rum Jungle samples immediately available to plants was low. Whether the rock is suitable as a base for the manufacture of superphosphate is a matter for industrial chemists and economists to decide, but from this test it would seem that unless the material shows a compensating long-term residual effect of a very high order it is not suitable for direct use as a phosphate fertilizer.

Table 1

POT TESTS.

PHOSPHOROUS CONTENT OF PLANTS

Plant Attribute	Equivalent Fertilizer Level	Rum Jungle Sample 1	Rum Jungle Sample 2	Christmas Island Dust	Super-phos-phate
Phosphorus Content of tops (%P)	1 cwt/ac	0.072	0.049	0.066	0.044
	4 cwt/ac	0.063	0.047	0.064	0.123
	8 cwt/ac	0.061	0.062	0.064	0.238
	16 cwt/ac	0.071	0.064	0.062	0.221
	Mean of all levels	0.067	0.056	0.064	-
	Mean of 1 & 4 cwt/ac	0.067	0.048	0.065	0.087
	Control P content with no fertilizer 0.036				
Phosphorus Content of roots (%P)	1 cwt/ac	0.046	0.040	0.036	0.036
	4 cwt/ac	0.037	0.040	0.039	0.075
	8 cwt/ac	0.040	0.042	0.042	0.101
	16 cwt/ac	0.041	0.040	0.050	0.138
	Mean of all levels	0.041	0.040	0.043	-
	Mean of 1 & 4 cwt/ac	0.041	0.040	0.038	0.058
	Control P content with no fertilizer 0.044				
Mean Phosphorus Content of whole plant (%P)	1 cwt/ac	0.058	0.045	0.051	0.040
	4 cwt/ac	0.050	0.044	0.052	0.100
	8 cwt/ac	0.051	0.051	0.055	0.168
	16 cwt/ac	0.054	0.051	0.057	0.181
	Mean of all levels	0.053	0.048	0.054	-
	Mean of 1 & 4 cwt/ac	0.054	0.044	0.052	0.073
	Control P content with no fertilizer 0.040				

2. REPORT ON POT TEST WITH SAMPLES OF CALCINED RUM JUNGLE PHOSPHATE ROCK

by

M.J.T. Norman[1]

Introduction

Samples of two types of phosphate-rich rock from the Rum Jungle area were received from the B.M.R. in 1963 for biological assay. A pot test with sorghum on Tippera clay loam soil from Katherine, N.T. was carried out, in which the fertillizers were compared, at 4 rates, with superphosphate and Christmas Island rock phosphate dust. The results were disappointing, though with the possibility of long-term residual values, a 4-year field experiment with the two materials was established at Katherine in December, 1963.

Following a report from the British Phosphate Commissioners concerning the beneficial effects of calcining low-grade phosphate rock from Christmas Island, small samples of the Rum Jungle materials were calcined at 800°C for 2 hours and given a further pot test.

Methods

The method adopted was to compare the responses of grain sorghum plants grown in Tippera clay loam (a phosphorus - deficient soil from Katherine, N.T.) to the two calcined phosphates and to superphosphate and Christmas Island rock phosphate dust at 4 levels of fertilizer with 3 replicates.

The 17 treatments were :

Calcined Rum Jungle No. 1) (1 cwt/ac equivalent
Calcined Rum Jungle No. 2 } { 4 cwt/ac "
Christmas Island Rock) (8 cwt/ac "
Superphosphate } { 16 cwt/ac "
Control .... No phosphate

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[1] C.S.I.R.O. Division of Land Research and Regional Survey.

Fertilizers were milled to a fine powder and mixed with Tippera clay loam soil. Five-inch enamel pots were filled and each planted with 12 seeds of Texas 620 grain sorghum on November 26, 1963. The pots were placed in the greenhouse and watered 2-3 times a day. On January 7 (6 weeks later) all plants were harvested. The tops were cut off at groundlevel and the roots washed free of soil. Tops and roots were dried separately at 85°C for 24 hours and weighed. The dried samples have been retained for future phosphorus analysis, but it was thought that in view of the interest attaching to the results an interim report on dry matter yields alone would be desirable.

Results

(See attached table)

The plants responded well to all fertilizers up to the 8 cwt/ac level. Considering tops only, there was an approximately linear response to Rum Jungle Nos. 1 and 2 and to Christmas Island up to the 16 cwt/ac level. The interaction between rates and fertilizers was significant at P - 0.01, owing to a depression in yield at the highest level of superhosphate. Differences between fertilizers and rates in root yield of sorghum were not significant, and hence the comparison of total yield (roots and tops) was similar to that of tops alone.

Table 1.

POT TESTS.

SORGHUM YIELD FROM SUPERPHOSPHATE AND CALCINED ROCK PHOSPHATE

Fertilizer	Equiv. Rate (cwt/ac)	Sorghum yield (gms per pot)		
		Tops	Roots	Total
Super-phosphate	1	2.64	2.35	4.99
	4	3.16	2.14	5.30
	8	6.49	3.22	9.71
	16	5.32	2.67	7.99
	Mean	4.40	2.60	7.00
Calcined Rum Jungle No. 1	1	2.46	2.82	5.28
	4.	4.21	3.04	7.25
	8	4.43	2.88	7.31
	16	5.70	4.09	9.79
	Mean	4.20	3.21	7.41
Calcined Rum Jungle No. 2	1	2.69	2.26	4.95
	4	2.72	2.55	5.27
	8	3.70	3.18	6.88
	16	4.41	2.24	6.65
	Mean	3.38	2.56	5.94
Christmas Island Dust	1	2.69	2.47	5.16
	4	3.80	3.02	6.82
	8	4.39	2.68	7.07
	16	4.61	3.79	8.40
	Mean	3.87	2.99	6.86
Mean of fertilizers	1	2.62	2.47	5.09
	4	3.47	2.69	6.16
	8	4.75	2.99	7.74
	16	5.01	3.20	8.21
	Mean	3.96	2.84	6.80
Control		2.16	1.72	3.88

Table 2.

STATISTICAL ANALYSIS OF THE YIELDS

Main effect of fertilizers	± 0.16 (P 0.001)	N.S. at P = 0.05	± 0.32 (P 0.05)
Main effect of rates	± 0.16 (P 0.001)	N.S. at P = 0.05	± 0.32 (P 0.001)
Interaction, fertilizers X rates	± 0.32 (P 0.01)	N.S. at P = 0.05	± 0.64 (P 0.05)

(Standard errors refer to analysis of all figures excluding control)

Discussion

In comparison with the previous test with uncalcined samples of Rum Jungle phosphate, these results are very promising. The increase in citrate solubility following calcining appears to have increased the availability of phosphate to the plant (under pot conditions, it should be stressed) to a marked degree.

3. REPORT ON POT TESTS WITH SAMPLES OF RUM JUNGLE PHOSPHATE ROCK.

CALCINING TIME-TEMPERATURES VS. UNTREATED ORES.

by

M.J.T. Norman[1]

Introduction

A request was received from the Bureau of Mineral Resources in 1963 for biological assay of two grades (high and low) of phosphate rich rock from Rum Jungle, N.T. Two pot experiments were conducted, using grain sorghum as test plant, on Tippera clay loam (a phosphorus deficient soil from Katherine, N.T.).

In the first experiment, finely ground Rum Jungle phosphates were compared, at four rates of application, with superphosphate and Christmas Island rock phosphate. Rum Jungle phosphate performed very poorly.

Following a report that calcining low grade Christmas Island phosphate ores, to increase their citrate solubility, was recommended to the British Phosphate Commissioners, a second experiment was carried out. Calcined Rum Jungle phosphates were compared with superphosphate and Christmas Island rock phosphate, again at four levels. Calcining increased the effectiveness of the high grade material to approximately that of superphosphate, and the low grade material to that of Christmas Island rock phosphate. When a third experiment was planned to measure directly the effect of calcining Rum Jungle phosphate, it was found that the samples used in the second experiment had been calcined, by error, at 800°C for 2 hours instead of 800°F for ½ hr. Further samples were obtained and half of each calcined at 450°C for 40 mins.[2]

Methods

High and low grade Rum Jungle phosphates, calcined at 800°C for 2 hours, 450°C for 40 minutes and uncalcined, were compared at four rates of application and with a control.

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[1] C.S.I.R.O. Division of Land Research and Regional Survey.

[2] The samples calcined at 800°C for 2 hours were part of the material used in botanical test 1. The material used in this experiment is part of the uncalcined phosphate rock used in field tests started in Katherine in 1963.

The twenty five treatment combinations were :

$$\left.\begin{array}{l}\text{Rum Jungle No.1}\\ \text{Rum Jungle No.2}\end{array}\right\} \times \left\{\begin{array}{l}\text{Calcined } 800^{\circ}\text{C 2 hr}\\ \text{Calcined } 450^{\circ}\text{C 40 min}\\ \text{Uncalcined}\end{array}\right\} \times \left\{\begin{array}{l}\text{1 cwt/ac equivalent}\\ \text{2 cwt/ac equivalent}\\ \text{4 cwt/ac equivalent}\\ \text{8 cwt/ac equivalent}\end{array}\right\}$$

+ Nil.

The design was replicated three times. The experiment was conducted in the greenhouse in Canberra.

The fertilizers, obtained in the form of powder from B.M.R. were mixed with sieved Tippera clay loam and placed in five inch enamel pots. Each pot was planted with seventeen seeds of Texas 620 grain sorghum on 20th February, 1964, and watered 2-3 times per day. On 28th February, the number of plants per pot was reduced to eight. The trial was rerandomized each week.

On 20th March, all plants were harvested at ground level, and the roots washed free from soil. Tops and roots were dried separately at 105°C overnight and weighed. The dried samples have been retained for phosphorus analysis, the results of which are not yet available.

Results

(see attached table)

The plants did not respond to fertilizer as well as in the first two experiments. Total growth per pot was as low as 20% of that recorded previously, possibly due to two weeks shorter growing period and lower glasshouse temperatures. In addition some plants showed evidence of a nutritional disorder, probably due to unsatisfactory water relations.

Approximately linear responses were obtained in top growth to rates up to 8 cwt/ac with all forms of fertilizer, although response to rates of uncalcined phosphate was not significant. There was no interaction between rates and forms of fertilizer.

Calcining considerably improved the effect on grain sorghum growth. The most interesting effect, however, was the increase in yield obtained with higher grade ore calcined at 800°C for 2 hr compared with that calcined at 450°C for 40 min.

The apparent grade x calcining interaction, although failing to reach significance at the 5% level is probably a real effect due to higher phosphorus content of the fresh ore sample obtained for calcining at the lower temperature.

Variability in root yields was much higher than in top yields. Significant differences were obtained between mean rates of fertilizer, but the differences between types of fertilizer failed to reach significance at the 5% level. There were no significant interactions.

Discussion

450°C for 40 mins closely approximates the time/temperature combination of 800°F for ½ hour recommended to the British Phosphate Commission. There is no obvious reason for the increase in yields obtained by calcining at 800°C for 2 hours.

Before having larger quantities calcined for field experimentation, a further pot experiment is planned. This will be designed to evaluate the effects of times and temperatures of calcining, covering the range 400-800°C at 30 mins-2 hours on both high and low grade ores.

Table 1.

POT TESTS.

YIELDS OF OVEN DRY MATERIAL FROM CALCINED PHOSPHATE ROCK POT TESTS.
K384C

Temperature-time	Grade[*]	Roots yield (gms/pot) 1 (cwt/ac)	2 (cwt/ac)	4 (cwt/ac)	8 (cwt/ac)	Means	Tops yield (gms/pot) 1 (cwt/ac)	2 (cwt/ac)	4 (cwt/ac)	8 (cwt/ac)	Means
450°C for 40 min	High Grade	0.853	0.967	1.390	1.260	1.120	0.770	0.857	0.983	1.270	0.970
	Low Grade	0.740	0.767	0.883	1.200	0.898	0.740	0.840	0.907	1.160	0.912
800°C for 2 hr.	High Grade	1.100	0.893	0.897	1.370	1.070	0.947	0.913	1.050	1.400	1.080
	Low Grade	0.853	1.130	1.050	0.917	0.986	0.800	0.843	0.873	1.050	0.891
Uncalcined	High Grade	0.700	0.953	0.903	1.060	0.903	0.690	0.840	0.731	0.823	0.772
	Low Grade	0.693	0.827	0.747	0.900	0.792	0.667	0.777	0.843	0.847	0.783
Means		0.822	0.922	0.977	1.120	-	0.768	0.845	0.899	1.090	-

[*] See the assays in Table 1, Section 1, Part 5

Table 2

STATISTICAL ANALYSIS OF YIELDS.

	Roots		Tops	
Item	Standard error	Probability	Standard error	Probability
Type	± 0.0786	>0.05	± 0.044	0.001
Rate	± 0.064	0.05	± 0.036	0.001
Rates x type	± 0.157	not significant	± 0.089	not significant.

PART 4.

BENEFICIATION TESTS.

1. PRELIMINARY STUDY OF THE BENEFICIATION OF A PHOSPHATE ORE BY PHYSICAL METHODS.

by

The Colorado School of Mines Research Foundation Inc.[1]

Introduction

This project, for the preliminary investigation of the beneficiation characteristics of a phosphate ore (reportedly from Australia), was initiated by Mr. W.E. Wallis, Exploration Manager for the Standard Oil Company of New Jersey, by a letter dated October 9, 1962.

Objective and Scope

The present investigation was limited to a preliminary study to determine whether or not a marketable phosphate mineral concentrate might be recovered from the ore by conventional mineral beneficiation methods.

Summary and Conclusions

The following summarizes the results of test work performed with a composite sample having the chemical analysis 17.1 per cent P_2O_5 and 15.0 per cent Fe_2O_3.

A high grade marketable phosphate product containing less than the permissible amount of iron oxide impurity cannot be recovered economically from any particle size range by conventional beneficiating methods. The reasons for this are :-

(a) Much of the iron is less than five microns in size and is intimately bound within the phosphate mineral crystal. Consequently, any concentration of fluoroapatite particles in sizes larger than 325-mesh still contains too much iron to be an acceptable product.

About 25 percent of the P_2O_5 in the sample is represented in this size fraction.

(b) Approximately 75 per cent of the P_2O_5 and 67 per cent of the Fe_2O_3 are contained in the subsieve or minus 325-mesh primary slimes and clay material. Although the possibility of obtaining a satisfactory product from this size range exists, there are no commercial physical methods that successfully and economically beneficiate this colloid-like material.

Recommendations

Owing to the low value of the final product, the beneficiating methods ordinarly applied to American phosphate deposits must necessarily be low in cost. These methods, unfortunately, are not applicable to the project sample.

If, however, other considerations such as the size of the deposit, the ease of mining, and the extent of the local market are favourable, then further investigation into new and perhaps more costly beneficiating procedures may be in order. Among these possible methods are:

1. The selective chemical leaching of either the desired material or the soluble impurities.

2. The separation and removal of the phosphatic particles by selective flocculation.

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[1] Confidential report to the Standard Oil Co. Inc.

3. The use of flotation for the ultrafine material.

Basic research will be required to determine the suitability of, and to develop the procedure for, any of the methods mentioned.

The principal deterrent to the flotation of slime material has been the enormous quantities of reagent required to effect flotation. The considerable amount of phosphate being discarded with the slimes in the Florida operations has been of some concern, and investigations into the possible recovery of this material by flotation have been made. Some success has been achieved by the use of oiled auxilliary collector minerals, upon which the desired slime material collects and is recovered, but this technique requires further development.

Discussion

Three samples of phosphate ore, marked DN-29(37), DN-29(38) and DN-29(42), were received and these were composited into one sample.* This composite sample, which was designated Composite Sample No.1, was the feed material for all tests reported here.

Details of the procedure used in making the composite sample for the investigative program are given in Exhibit 1 of the Appendix. A partial chemical analysis is given below (Table 1).

Table 1

CHEMICAL ANALYSIS COMPOSITE SAMPLE NO. 1.

P_2O_5 %	Fe_2O_3 %	U_3O_8 %
17.1	15.0	0.018

Particle size and P_2O_5 distribution

A screen assay test was conducted to determine the particle size and P_2O_5 distributions in the project sample. In this test, the minus $\frac{1}{4}$ inch composite material was given an attrition mixing scrub in order to reduce any agglomerates, both clay and mineral, to discrete particles. The complete data for this test are given in Exhibit 2 of the Appendix.

The particle size distribution results, which are also shown graphically in Figure 1, revealed that 77.0 per cent of the material was smaller than 270-mesh and that 73.4 per cent was smaller than 400-mesh (38 microns).

Each mesh range above 270-mesh was not submitted separately for chemical analysis because so little of the total sample was represented by each.

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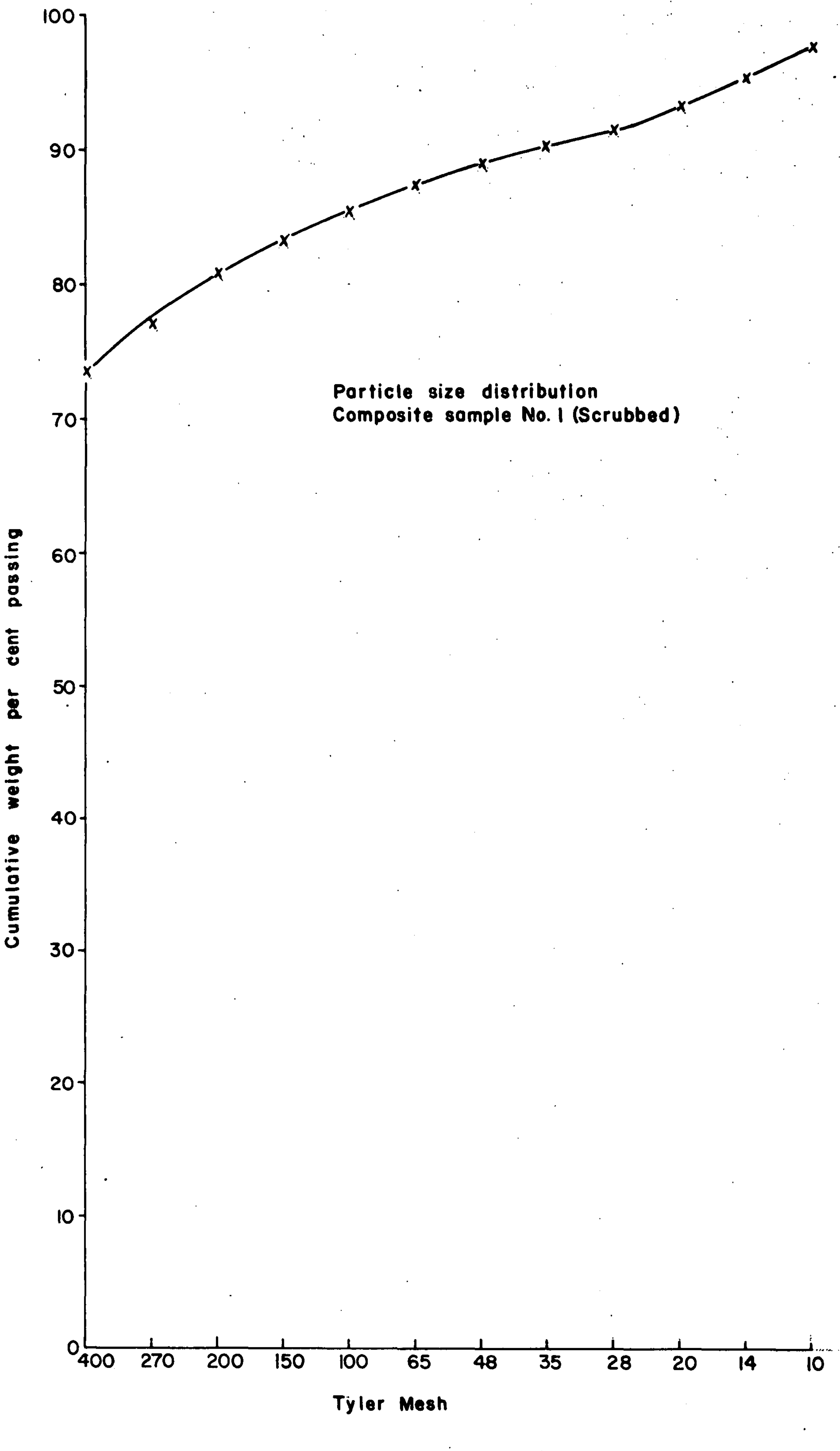
* The samples originally sent to Esso Standard Oil (Aust) Pty Ltd were taken from costean 1 at Geolsec over the following intervals.

Sample 1	280'	- 295'
" 2	295'	- 315'
" 3	315'	- 330'

Fig. 1



Particle size distribution
Composite sample No. 1 (Scrubbed)

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Instead the screen fractions were combined to form-

1. A coarse fraction as represented by the plus 28-mesh material.
2. A minus 28 plus 270-mesh fraction. This is the normal range of phosphate mineral flotation.
3. A minus 270-mesh slime fraction.

The chemical analysis, weight distribution, and P_2O_5 and Fe_2O_3 distribution of these three broad categories are shown in Table 2.

Table 2.

CHEMICAL ANALYSIS; WEIGHT, P_2O_5 AND Fe_2O_3
DISTRIBUTION IN SCRUB SAMPLE 1.

Screen Product (Tyler) Mesh	Weight %	Analysis P_2O_5 %	Analysis Fe_2O_3 %	Distribution P_2O_5 %	Distribution Fe_2O_3 %
Feed (computed)	100.0	17.1	15.1	100.0	100.0
+ 28	8.6	15.9	21.6	8.0	12.3
-28 +270	14.4	11.5	13.9	9.7	13.2
-270	77.0	18.3	14.6	82.3	74.5

The important facts revealed by the screen assay test were that the major portion, 82.3 per cent, of the P_2O_5 was present in the ultrafine (minus 270-mesh) material and that the Fe_2O_3 distribution closely followed that of the P_2O_5. This indicated close physical association of iron with the phosphate mineralization.

Material degradation

It was reasoned that the scrub used to prepare the sample for the screen assay may have been too severe, and that material degradation from this treatment may have been responsible for much of the subsieve material.

To determine whether or not this were possible, a test was performed in which the sample was scrubbed in stages. The material was given first a light blunging in a rotary-type scrubber to soften and loosen the primary slimes and then a series of one-minute attrition scrubs. The minus 270-mesh material was removed after each scrub and only the plus 270-mesh subjected to further treatment.

Exhibit 3 of the Appendix presents the details of this test, and Figure 2 is a graphical representation of the test results.

The 44.2 per cent minus 270-mesh shown at zero time in Figure 2 was the amount of fine material removed by the rotary blunge. The amount of slime production was fairly rapid for the first three attrition scrubs and then decreased to a uniform rate of approximately 1.5 per cent for each minute of scrub. It was deduced from this that the rotary blunge and the three minutes of attrition scrub effectively removed the primary slime material in the sample, and that further scrubbing served only to reduce particle size by autogenous grinding.

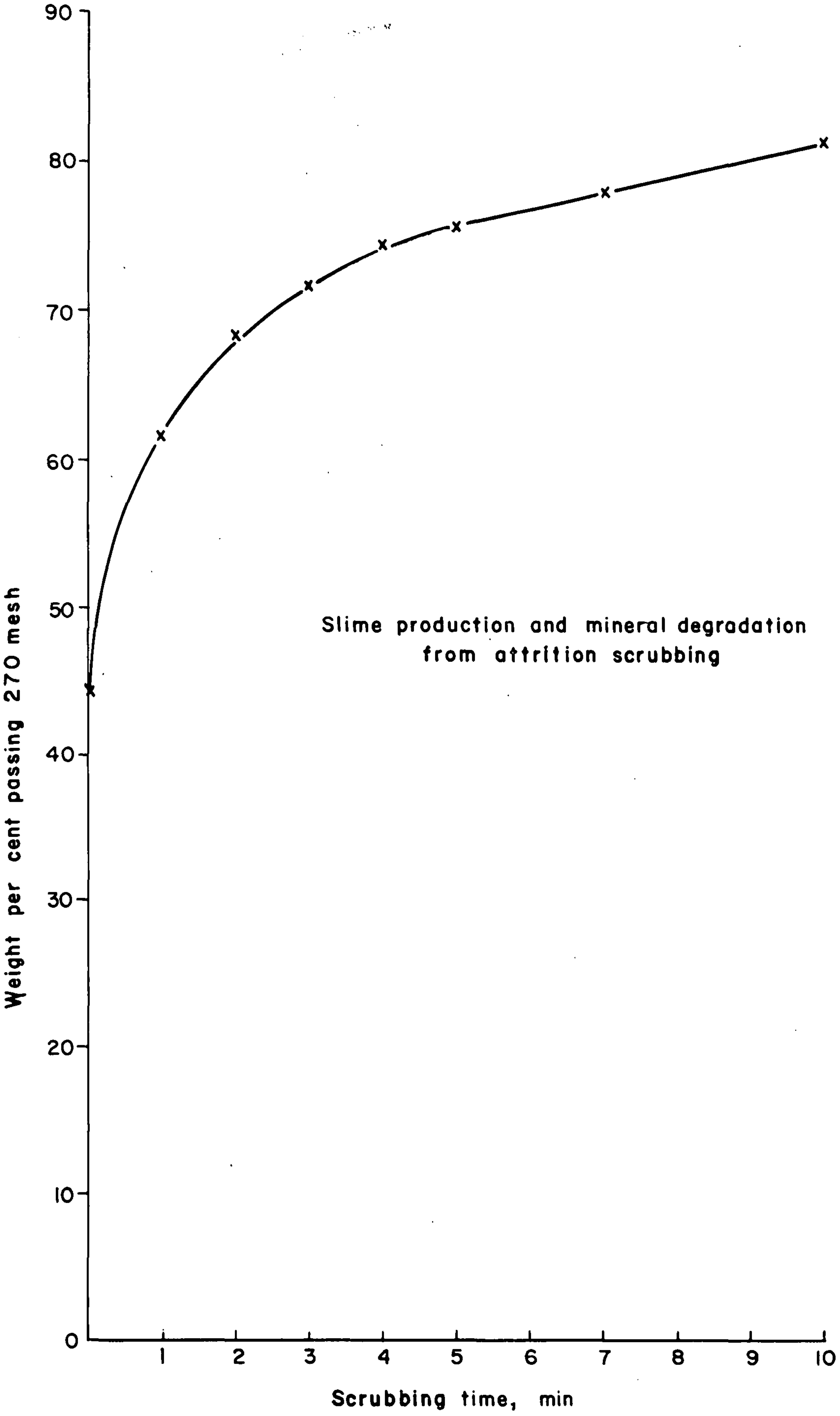
Figure 2.

Although only 71.8 per cent of the sample weight was less than 270-mesh after the three-minute scrub, versus 77.0 per cent in Screen Assay Test No.1, the difference was insufficient to make any significant change in the screen assay results. Even at reasonably mild scrubs, at least 75 per cent of the P_2O_5 may be expected in the ultrafine sizes.

Floatation test

Although most of the P_2O_5 was in the fraction which was outside the range normally considered for flotation, a flotation test was performed with the minus 28 plus 325-mesh fraction to determine the type of product that

Fig.2



Slime production and mineral degradation from attrition scrubbing

NT/A84

Bureau of Mineral Resources, Geology & Geophysics *To accompany Record 1964/106* might be achieved by this beneficiation method.

First attempts at flotation were made with samples which had been scrubbed only mildly in order that more material would be in the flotation size range. These were unsuccessful, however, because considerable amounts of slimes were produced during the reagent conditioning step, and even large quantities of reagent were ineffective in coating the apatite particles.

The final procedure used to obtain the feed for the flotation test is outlined in Scrub Test No.2, Exhibit 2 of the Appendix. Briefly, this procedure included stage crushing the original material in minus 14-mesh, then blunging this in a rotary scrubber, and finally scrubbing in a laboratory Fagergren flotation machine. The latter scrub was considerably milder than that obtained with a Wemco attrition scrubber, and the final products resulting from the treatment above are shown in Table 5.

Table 3.

CHEMICAL ANALYSIS; WEIGHT, P_2O_5 AND Fe_2O_3 DISTRIBUTION IN SCRUB SAMPLE 2.

Screen Product (Tyler) Mesh	Weight %	Chemical Analysis (computed) P_2O_5 %	Chemical Analysis (computed) Fe_2O_3 %	Distribution P_2O_5 %	Distribution Fe_2O_3 %
Feed	100.0	16.8	15.0	100.0	100.0
- 14 + 28	9.6	17.7	20.2	10.1	13.0
- 28 +325	19.8	13.6	15.1	16.1	20.0
-325	70.6	17.5	14.2	73.8	67.0

The feed preparation procedure resulted in more material weight and more P_2O_5 in the intermediate size range (compare with Screen Assay Test No.1) but much was still in the form of hard clay balls.

The minus 28 plus 325-mesh material was conditioned for four minutes with the following reagent quantities:

Table 4.

FLOTATION TEST - CONDITIONING REAGENTS

Reagent	Pounds per Ton of Flotation Feed
Sodium Hydroxide	0.33
Acintol D29LR (a partially distilled tall oil)	1.33
Fuel Oil No.2	1.48

A considerable excess of collector reagents was added purposely to allow for the consumption by the slimes produced during conditioning.

The concentrate obtained from the rougher flotation was given four cleanings. The tailings from the first three cleaning operations were somewhat similar in appearance and therefore were combined for chemical analysis.

The table below summarizes the results obtained from the flotation test. Complete flotation test data are given in Exhibit 4 of the Appendix.

Table 5.

RESULTS OF FLOTATION TEST

Product	Weight	Chemical Analysis		Per Cent Distribution	
		P_2O_5	Fe_2O_3	P_2O_5	Fe_2O_3
Feed (computed)	100.0	13.6	15.1	100.0	100.0
Final concentrate	10.8	34.2	9.44	27.2(1)	6.8
4th cleaner tailing	1.9	24.6	16.2	3.4	2.0
3rd cleaner tailing	4.8	11.6	10.2	19.3	15.3
2nd cleaner tailing	7.2				
1st cleaner tailing	10.6				
Rougher tailing	64.7	10.5	17.7	50.1	75.9

(1) Only 4.4 per cent from original ore.

A concentration of the P_2O_5 and some rejection of the iron minerals was accomplished by flotation. Whereas the feed material contained approximately equal quantities of P_2O_5 and Fe_2O_3 the final concentrate product P_2O_5 content was almost four times that of the Fe_2O_3.

Although the concentrate product had a satisfactory P_2O_5 analysis, the 9.44 per cent Fe_2O_3 content was much too high for this material to be accepted commercially.

The P_2O_5 recovery represented by the flotation concentrate was only 4.37 per cent of the P_2O_5 in the original sample.

From the test results it was concluded that the flotation method of mineral beneficiation was not applicable to the project sample material.

Gravity concentration

A gravity concentration test was performed with the minus 14 plus 28-mesh material to determine the type of product obtainable from this size range. In view of the data obtained from prior test work the results from this test would be of academic interest only.

The sink-float procedure was used in this test to separate minerals of different specific gravity. The sample was treated first with a liquid having the specific gravity 2.88. The particles having a specific gravity higher than 2.88 were collected in a sink product, while those floating at this density were further treated with a liquid of lower specific gravity, 2.67;

thus, three products were obtained. The lower liquid gravity was just sufficient to float quartz. Test details are given in Exhibit 5 of the Appendix. Test results are reproduced below.

Table 6

RESULTS OF GRAVITY CONCENTRATION TEST

Product	Weight %	Chemical Analysis P_2O_5 %	Chemical Analysis Fe_2O_3 %	Per Cent Distribution P_2O_5 %	Per Cent Distribution Fe_2O_3 %
Feed (computed)	100.0	17.7	20.2	100.0	100.0
Float at Sp. Gr. 2.67	29.6	10.4	10.7	17.4	15.7
Float at 2.88, Sink at 2.67	34.8	14.5	29.4	28.5	50.7
Sink at Sp. Gr. 2.88	35.6	26.9	19.1	54.1	33.6

These results again demonstrated the intimate association of phosphorus and iron and the impracticability of producing a satisfactory concentrate by conventional phosphate beneficiation techniques.

Petrographic examination of the flotation concentrate.

To determine the reason for the high iron content of the flotation concentrate, this material was sized into the four fine fractions, minus 100 plus 150, minus 150 plus 200, minus 200 plus 270, and minus 270 plus 325-mesh, and examined under the petrographic microscope. The primary phosphate mineral, fluoroapatite, occurred as stout subhedral forms in each of these fractions. There was also some light to dark-brown structureless material that was identified tentatively as clay. This was expected because the material had not been given the vigorous scrub necessary to reduce clay lumps.

A few grains of fibrous wavellite were observed in each fraction, and the small amount of quartz present was most abundant in the plus 150-mesh fraction and decreased to essentially none in the minus 270 plus 325-mesh range.

Scales of micaceous hematite occurred in zonal-like arrangements within the fluoroapatite crystals. These iron particles ranged in size from 0.5 to 5 microns, and appeared to have been incorporated into the fluoroapatite during crystal growth. The extremely fine size of the iron mineral, together with the manner in which it is associated with the fluoroapatite, indicated that the possibility of recovering a suitable low iron phosphate product by physical methods was very remote.

APPENDIX

Exhibit 1.

Sample Description and Preparation

Sponsor's Designation of Sample	DN-29 (37), DN-29 (38), DN-29 (42)
Date Received at Foundation	November 9, 1962.
Sample Weight	Approx. 100 lbs.
Container	Three 45-lb oil drums. The samples were further enclosed in double durlap sacks.
Sample Description	DN-29 (38) - Pale yellow-brown fines and clayey agglomerates up to 2" in size. DN-29 (37) - Similar to the sample above, but more reddish in colour. Also more coarse material, and some pieces about 3". DN-29 (42) - Similar to (37) above except there was less of the coarse material. There were also 2 or 3 hard rock pieces.
Method of Preparation	After crushing the coarse pieces to $\frac{3}{4}$", each of the 3 individual samples was mixed thoroughly and split once. Then $\frac{1}{2}$ of each individual sample was taken and combined to form a composite sample which was designated Composite Sample No.1. The composite sample was further crushed to $\frac{1}{4}$", mixed thoroughly, and small samples split out for the test work. The moisture content of the Composite Sample was 9.3%.

Table 1.

CHEMICAL ANALYSES COMPOSITE SAMPLE NO.1.

P_2O_5 %	Fe_2O_3 %	U_3O_8 %
17.1	15.0	0.018

Exhibit 2

Screen Assay Test

Screen Assay Test No.1

Sample	Approx. 670 grams of Composite Sample No. 1 (-$\frac{1}{4}$").
Procedure	The sample was pulped to 40% solids and scrubbed for 5 min in a laboratory Wemco attrition scrubber. The sample was deslimed by screening at 270M. Since the +270M fraction appeared to still contain clay particles, this fraction was given an additional 4 min scrub before wet screening to produce the size fractions listed below. The individual fractions represented so little material that they were not analyzed separately but combined into three fractions; a +28M coarse fraction, a size range (-28+270) suitable for flotation, and a -270M size.

Results:

Table 2

RESULTS OF SCREEN ASSAY TEST

Screen Product (Tylor) Mesh	Weight % Direct	Weight % Cumulative	Chemical Analysis P_2O_5	Chemical Analysis Fe_2O_3	Per Cent Distribution P_2O_5	Per Cent Distribution Fe_2O_3
Head (computed)	100.0		17.1	15.1	100.0	100.0
+ 10	2.5	2.5	15.9	21.6	8.0	12.3
- 10 + 14	2.2	4.7				
- 14 + 20	2.3	7.0				
- 20 + 28	1.6	8.6				
- 28 + 35	1.2	9.8	11.5	13.9	9.7	13.2
- 35 + 48	1.3	11.1				
- 48 + 65	1.7	12.8				
- 65 +100	1.8	14.6				
-100 +150	2.3	16.9				
-150 +200	2.3	19.2				
-200 +270	3.8	23.0				
-270	77.0		18.3	14.6	82.3	74.5

Note: The -270 +400M fraction was determined later and found to be 3.6% of the total sample weight.

Exhibit 3

Scrub Tests

Scrub Test No. 1

Purpose : Because Screen Assay Test No. 1 has shown that most of the sample was less than 270M, this test was performed to determine the effect of the attrition scrub on sample degradation.

Sample : Approx. 670 grams of Composite Sample No. 1 (-$\frac{1}{4}$"=.

Procedure : The sample was given a gentle scrub by blunging at 20% solids in a rotary scrubber. Following this, the pulf was screened at 270M. The +270M fraction was then pulped to 50% solids and scrubbed in a laboratory Wemco attrition scrubber for 1 min. The scrubbed product was again screened at 270M. The attrition scrub procedure was repeated for a total of five 1-min. scrubs, a 2-min. scrub, and a 3-min scrub.

Results:

Table 3

RESULTS OF SCRUB TESTS NO.1.

		Scrub time (min.)		Weight	
Screen product mesh (Tyler)	Scrub Type	Direct	Cumulative	Direct	Cumulative
-270	Blunge	15	-	44.2	44.2
-270	Attrition	1	1	17.3	61.5
-270	Attrition	1	2	6.9	68.4
-270	Attrition	1	3	3.4	71.8
-270	Attrition	1	4	2.5	74.3
-270	Attrition	1	5	1.5	75.8
-270	Attrition	2	7	2.3	78.1
-270	Attrition	3	10	3.3	81.4
+270				18.6	

Scrub Test No.2

Purpose :	To provide material for Flotation Test No.1
Sample :	Approx. 1.380 grams of Composite Sample No.1 That had been stage crushed to pass 14M.
Procedure :	The sample was blunged for 15 min in the rotary scrubber and then screened at 325M. The +325M fraction was transferred to a laboratory Fagergren flotation cell and given a series of 2-min. scrubs, each followed by screening at 325M. Total scrubbing time was 10 min.

The +325M material was screened at 28M, and the -28 +325M fraction again scrubbed in the flotation cell. Scrubbing was discontinued even though it was evident that additional scrubs would produce more -325M material.

The type of scrubbing action obtained with the flotation cell was considerably milder than that with the Wemco attrition.

Results :

Table 4

RESULTS OF SCRUB TEST NO.2.

Sample (Screen product in Tyler mesh)	Weight %	Chemical analysis (Computed)		Distribution	
		P_2O_5 %	Fe_2O_3 %	P_2O_5 %	Fe_2O_3 %
Feed	100.0	16.8	15.0	100.0	100.0
- 14 + 28	9.6	17.7	20.2	10.1	13.0
- 28 +325	19.8	13.6	15.1	16.1	20.0
-325	70.6	17.5	14.2	73.8	67.0

Exhibit 4

Flotation Test

Flotation Test No.1.

Purpose :	To test the amenability of the phosphate mineral to the flotation process.
Sample :	The -28 + 325M fraction of Composite Sample No.1 obtained from Scrub Test No.2.

Test Conditions :

Table 5.

FLOTATION TEST PROCEDURE

Process	Time min.	% Solids	pH Start	Reagents - Pounds Per Ton of Ore		
				NaOH	Acintol D29LR	Fuel Oil - No. 2 Oil.
Conditioning	4	60		0.33	1.33	1.48
Rougher flotation	3	15	8.5			

Note: The rougher concentrate was given 4 cleanings.

Results :

Table 6.

RESULTS OF FLOTATION TEST.

Product	Weight %	Chemical Analysis		Per Cent Distribution	
		P_2O_5 %	Fe_2O_3 %	P_2O_5	Fe_2O_3
Feed(computed)	100.0	13.6	15.1	100.0	100.0
Final concentrate.	10.8	34.2	9.44	27.2	6.8
4th cleaner tailing	1.9	24.6	16.2	3.4	2.0
3rd cleaner tailing	4.8	11.6	10.2	19.3	15.3
2nd cleaner tailing	7.2				
1st cleaner tailing	10.6				
Rougher tailing	64.7	10.5	17.7	50.1	75.9

Remarks: An excess of reagent was used purposely to help counteract the deleterious effect of the slimes which were produced during conditioning.
The rougher tailing was again reagentized after the 1st float, but very little new material was floated. However, when this tail was rescrubbed, the slimes removed, and then reagentized, about 37 grams of material was floated. This product contained a considerable amount of quartz, so it was recombined with the rougher tailing.
The first 3 cleaner tailings were somewhat similar in appearance (each contained considerable quartz) so they were combined into 1 sample for chemical analysis.

Exhibit 5.

Gravity Concentration Test

Gravity Concentration Test No.1.

Purpose : To determine whether a high phosphate material can be produced by gravity concentration.

Sample : The -14 + 28M fraction obtained from Scrub Test No.2.

Procedure : The sample was treated by the heavy liquid procedure for separating minerals of different specific gravities.
Using first a liquid having the specific gravity 2.88, the material which was heavier than this specific gravity was recovered as a product, while the material which floated was further separated with a liquid medium having a specific gravity of 2.67.

Acetylene tetrabromide, diluted as required with carbon tetrachloride, was the heavy liquid used.

Results :

Table 7.

RESULTS OF GRAVITY CONCENTRATION TEST

Product	Weight %	Chemical Analysis		Per Cent Distribution	
		P_2O_5 %	Fe_2O_3 %	P_2O_5	Fe_2O_3
Head (computed)	100.0	17.7	20.2	100.0	100.0
Float as sp. gr. 2.67 2.67	29.6	10.4	10.7	17.4	15.7
Float at 2.88, sink at 2.67	34.8	14.5	29.4	28.5	50.7
Sink at 2.88	35.6	26.9	19.1	54.1	33.6

Observations : The float at specific gravity 2.67 consisted of quartz principally.

2. THE USE OF RUM JUNGLE PHOSPHATE ROCK TO PRODUCE A NITROGENOUS FERTILISER.

In May 1964 Esso Standard Oil (Australia) Ltd reported that a split of the 100 lb. sample sent to the Colorado School of Mines Research Foundation Inc. by the Standard Oil Co Inc. was given to two companies who used a secret comminution process to obtain a nitrogenous phosphatic product giving the analysis in Table 1.

Table 1.

ASSAY OF NITROGENOUS PHOSPHATE PRODUCT.

Item	Percentage
Moisture	8.86
Moisture 26. 4. 64	8.98
Total nitrogen	14.31
Nitrate nitrogen	7.57
Ammoniacal nitrogen	6.05
P_2O_5	21.40
Citrate unsoluble P_2O_5	0.06
Water soluble P_2O_5	2.40
Iron Fe	1.23
" Fe_2O_3	1.76
Alumina Al	4.27
" Al_2O_3	8.06
Fluorine F	0.59
Siliceous material	0.10
Acid soluble calcium Ca	6.56
" " " CaO	9.2

Assayed by Shuey & Co. U.S.A.

The Esso people in America made the following comment-

> "The mosture content of this product was 8.86%. An unusually low critical relative humidity (20%) indicates that the fertiliser is very hygroscopic although the sample was received dry and free flowing, and remained that way on overnight exposure to atmospheric moisture. The high moisture content and low critical relative humidity suggest the formation of stable salt hydrates. The fertiliser was 56% water soluble. The water soluble P_2O_5 accounted for 11.2% of the total P_2O_5. A 10% slurry of this product had a pH of 4.77."

PART 5

TESTS IN PROGRESS

1. LABORATORY TESTS.

Samples of calcined material from Costean 1 at Geolsec were sent to Dr. Norman of the C.S.I.R.O. in February 1964 for further pot tests. Some of the results from these tests are reported in Sections 2 and 3 of Part 3.

Table 1.

ASSAYS OF RUM JUNGLE PHOSPHATE SAMPLES FOR POT AND FIELD TESTS.[1]

Sample	Locality	%P_2O_5	Ignition Loss %
1.	Geolsec Costean 1.	22.5	
1.(ignited)[2]	315'-330'	24.8	8.7
2.	Geolsec Costean 1.	33.8	
2.(ignited)[2]	459'-460'	34.2	3.0

[1] Assayed by S. Baker (B.M.R.)

[2] Calcined for 40 minutes at 450°C.

The samples were crushed at the Mount Wells Stamp Battery in the Northern Territory and are taken from the material sent to the C.S.I.R.O. experimental station at Katherine in 1963 for field tests.

2. FIELD TESTS IN THE NORTHERN TERRITORY

Uncalcined crushed samples of the material described in Table 1 were sent to the C.S.I.R.O. Experimental Station at Katherine and to Mr. J. Turnour of Coomalie Creek for field tests.

The testing to be carried out at Katherine is outlined in the following programme.

C.S.I.R.O. KATHERINE RESEARCH STATION

PRE-SCHEDULE

K.367 - RUM JUNGLE PHOSPHATE ASSEMENT.

by

M.J.T. Norman

INTRODUCTION : In 1963, at the request of B.M.R., L.R.R.S. conducted 6-week pot tests with sorghum on two samples of Rum Jungle phosphate rock.[1] Responses were poor. Again at the request of B.M.R., L.R.R.S. is undertaking a field trial to measure the residual value of these materials.

OBJECTIVE : To compare the initial and residual values of two samples of Rum Jungle phosphate rock with those of Christmas Island rock phosphate dust on a permanent pasture of birdwood grass.

TREATMENTS :

Initial applications 1963/64			Annual applications (from 1963/64 on)
A. Rum Jungle No.1	4 cwt/ac	X	(1 cwt/ac super (S) (Nil (C)
B. " "	8 cwt/ac		
C. " "	12 cwt/ac		
D. Rum Jungle No.2	4 cwt/ac		
E. " "	8 cwt/ac		
F. " "	12 cwt/ac		
G. Christmas Island	4 cwt/ac		
H. " "	8 cwt/ac		
I. " "	12 cwt/ac		
J. Control Nil			

DESIGN + AREA : H.5 (West). The 10 initial treatments on main plots 35 x 30 ft. Randomized block with 3 replicates.
Total area 210 x 300 ft.

MANAGEMENT :

(a) At end of first year, forage harvest in April(1964).

(b) In subsequent years, forage harvest in February and April.

(c) Apply super treatments in late dry season each year.

(d) Beginning November, 1963, top dress whole experiment by drill with 3 cwt/ac. sulphate of ammonia annually.

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[1] Two samples of phosphate rock were collected from Costean 1 Geolsec. see Part 5 Table1of this record.

SAMPLING : (a) In first year, sample in April only,

(b) In subsequent years, sample in February and again in April. Both immediately before forage harvesting.

(c) At sampling, cut 3 random 1 x 1 yd. quadrats per plot and bulk together. Weigh samples, take 200 gm. chopped sample and dry. Reweigh. Mill dried sub-samples for subsequent N and P analysis.

3. BENEFICIATION TESTS

Two samples have been collected from Costean 1 Geolsec. for beneficiation tests in France by Cerphos.

Table 2.

PHOSPHATE SAMPLES FOR BENEFICIATION TESTS BY CERPHOS.

Sample	Site	Semiquantitative P_2O_5 Analysis.
1.	Costean 1 Geolsec. 459'-460'	28
2.	Costean 1 Geolsec. 315'-330'	12

CONCLUSION.

At present the high iron and aluminium content of the Rum Jungle phosphate rock, its fine grainsize and its consequent tendency to form a large proportion of slimes when crushed, make it unsuitable for use as raw material for conventional methods of superphosphate manufacture.

Considerable hope exists that the current C.S.I.R.O. experiments will prove it to be suitable for use as calcined ground rock phosphate whose value as a fertiliser will equal that of superphosphate.