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THE EVALUATION OF NORTH REYNELLA SHALE FOR CERAMIC WARE MANUFACTURE

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CONTENTS

			Page
1.	INTRODUCTION		1
2.	SUMMARY		1
3.	MATERIALS EXAMINED		2
4.	EXPERIMENTAL PROCEDURE		2
5.	RESULTS		2
	5.1	Description of the Raw Materials - Their Slaking and Grinding Behaviour	2
	5.1.1	Top Quarry Stockpile Material (No. 1548)	2
	5.1.2	Selected White Material from Bottom Quarry (No. 1549)	3
	5.1.3	Selected Yellow Material from Bottom Quarry (No. 1550)	3
	5.1.4	Selected Purple Material from Bottom Quarry (No. 1551)	3
	5.2	Pressing Behaviour of the Materials	3
	5.3	Mixing and Extrusion Behaviour of the Materials	3
	5.3.1	Composite Top Quarry Sample (No. 1548)	3
	5.3.1.1	Not De-Aired	3
	5.3.1.2	De-Aired	4
	5.3.2	Selected White Material from Bottom Quarry (No. 1549)	4
	5.3.2.1	Not De-Aired	4
	5.3.2.2	De-Aired	4
	5.3.3	Composite Blend of Bottom Quarry Material (No. 1556)	4
	5.3.3.1	Not De-Aired	4
	5.3.3.2	De-Aired	4
	5.3.4	Composite Blend of Material from Top and Bottom Quarries (No. 1557)	5
	5.3.4.1	Not De-Aired	5
	5.3.4.2	De-Aired	5
	5.4	Drying Behaviour of Specimens	5
	5.4.1	Pressed Briquettes	5
	5.4.2	Extruded Ware	5
	5.4.2.1	Composite Top Quarry Material (No. 1548)	5
	5.4.2.2	Selected White Material from Bottom Quarry (No. 1549)	5
	5.4.2.3	Blend of Bottom Quarry Material (No. 1556)	5
	5.4.2.4	Blend of Top and Bottom Quarry Materials (No. 1557)	6
	5.5	Firing Behaviour	6
	5.6	Transverse Strength (Modulus of Rupture)	14
	5.7	Efflorescence	16
6.	CONCLUSIONS AND RECOMMENDATIONS		16

1. INTRODUCTION

The construction of an oil refinery at Halletts Cove together with ancillary business premises and dwellings in the Halletts Cove-Morphett Vale-Christies Beach areas, some 15-16 miles south of Adelaide, South Australia, is likely to create an increasing demand for good quality building materials. At present there is no large heavy clayware plant south of Adelaide and consequently the establishment of a modern works would be both a profitable venture and an asset to the community.

Within the past 12 months the Rosewall Sand and Quarrying Co. Ltd. has discovered a deposit of highly weathered shale at North Reynella some 12 miles south of Adelaide on the Southern Highway. This deposit is located in Section 500, Hundred of Noarlunga, County of Adelaide. The Company owns a 30-acre site and has opened up two quarries on an adjoining 50 acres over which they have a 15-year lease for the removal of material.

In order to encourage the development of the deposit the Department of Mines sponsored this project for an investigation into the suitability of the material for the manufacture of heavy clayware products.

The investigation recorded in this report deals specifically with the 50-acre leased site. The two quarry workings referred to in the text as the top and bottom quarries were inspected and sampled in company with Mr. G. Olliver, geologist of the Department of Mines.

2. SUMMARY

A comprehensive investigation into the pressing and extrusion behaviour of North Reynella shale has been undertaken.

The results are promising but indicate that a variety of different materials exists in the deposit.

Good bricks could probably be made from the material by either the pressing or de-aired extrusion method.

The green and fired strength of ware was found to be not very high.

Considerable variation in the fired colour of specimens as well as flashing due probably to soluble salts was observed.

The production of sound ware is likely to require a firing temperature somewhat higher than is prevalent in local practice, but the best white material from the bottom quarry cannot be regarded as refractory.

The deposit could be a large one as the Company owns a 30-acre site and also has a 15-year lease for the removal of material from an adjoining 50-acre site from which the samples under examination were taken. However, it is recommended that the whole 80 acres be bored in a systematic grid pattern to prove the quantity, quality and consistency of the material available before any large scale developmental work is undertaken, or a modern heavy clayware plant is erected.

3. MATERIALS EXAMINED

The following materials were examined:-

Top Quarry - Stockpile

One composite sample - (No. CM1548; D/M.A337/61)

Bottom Quarry

Selected white material	(No. CM1549;	D/M.A338/61)
" yellow "	(No. CM1550;	D/M.A339/61)
" purple "	(No. CM1551;	D/M.A340/61)

4. EXPERIMENTAL PROCEDURE

Small pieces of material were selected from each sample and submitted to the slaking test.

Each sample was then ground to pass an 18-mesh screen (BSS).

Scout tests were made on the pressing and extrusion behaviour of each material separately and also on appropriately prepared blends.

The results obtained from these tests were used as a guide in the preparation of the following batches for more detailed investigation:

Top Quarry Material (No. 1548)

Selected White Bottom Quarry Material (No. 1549)

Bottom Quarry Blend (No. CM1556) consisting of -

2 parts of selected white	material	(No. CM. 1549)
1 part of " yellow	"	(No. CM. 1550)
1 part of " purple	"	(No. CM. 1551)

Blend of Top and Bottom Quarry Materials (No. CM. 1557) in the proportions of -

4 parts of top quarry material	(No. CM. 1548)
2 parts of bottom quarry white material	(No. CM. 1549)
1 part of " " yellow "	(No. CM. 1550)
1 part of " " purple "	(No. CM. 1551)

These batches were examined for their pressing and extrusion behaviour followed by drying, firing, transverse strength and efflorescence tests on prepared specimens.

5. RESULTS

5.1 Description of the Raw Materials - Their Slaking and Grinding Behaviour

All material was ground to pass an 18-mesh screen (BSS).

5.1.1 Top Quarry Stockpile Material (No. 1548)

This consisted of large pieces of yellow and light-cream to white material of a highly weathered or decomposed nature and a small amount of brown surface material.

Some of the white material slaked down in 45 minutes, but other pieces, although very water absorbent, did not slake. The yellow material absorbed water slowly but did not slake.

The material ground fairly easily but tended to flake on passing through the grinding rolls.

The colour after grinding was cream.

5.1.2 Selected White Material from Bottom Quarry (No. 1549)

This material consisted of hand picked pieces of best white soft shale from the bottom quarry.

Some pieces slaked down in 45 minutes, whereas others did not slake at all but became very soft.

It ground easily. After grinding the colour was off-white.

5.1.3 Selected Yellow Material from Bottom Quarry (No. 1550)

The sample consisted of hand picked pieces of yellow stained soft shale. Some of the pieces contained ferruginous quartz bands.

Representative pieces slaked down in 30 minutes whilst others absorbed water without softening.

The material ground easily. After grinding it was mustard colour.

5.1.4 Selected Purple Material from Bottom Quarry (No. 1551)

This sample consisted of hand picked pieces of purple stained soft shale containing hard veins of ironstone.

Some pieces slaked down in 30 minutes but hard pieces absorbed water without slaking or softening.

The material ground easily apart from hard pieces containing ironstone. The colour after grinding was pale purple.

5.2 Pressing Behaviour of the Materials

The parent materials and blends were scout pressed at different moisture contents. The optimum moisture content for each material was found to be 10.5 per cent.

Accordingly batches of each material and blends were pressed at this moisture content.

They pressed quite well but laminations developed in the briquettes if they were "bumped" in the press.

5.3 Mixing and Extrusion Behaviour of the Materials

All raw materials mixed readily with water when prepared for extrusion.

The individual materials and blends were scouted for extrusion moisture content and then large batches were prepared for full scale extrusion tests.

All de-aired extrusion work was conducted at 28 inches vacuum.

5.3.1 Composite Top Quarry Sample (No. 1548)

A batch was prepared with a moisture content of 21.7 per cent.

5.3.1.1 Not De-Aired

When not de-aired the extruded columns were weak to handle and some of them developed dog-ear cracks. However, the oil lubricated column from the $1\frac{1}{2}$ x 1-inch briquette die and the plain ribbon tile column did not develop dog-ear cracks.

The speed of extrusion was high and the power consumption low.

5.3.1.2 De-Aired

When the material was extruded de-aired it became tough, plastic and clean to handle.

A variety of shapes was produced including special briquettes, roofing tiles and sewer pipes.

Speed of extrusion was high and the power consumption low.

5.3.2 Selected White Material from Bottom Quarry (No. 1549)

A batch was prepared for extrusion at a moisture content of 21.7 per cent.

5.3.2.1 Not De-Aired

When extruded not de-aired the material was weak and formed dog-ear cracks on the columns.

5.3.2.2 De-Aired

When extruded de-aired it was tough, strong, plastic and clean to handle. Only briquettes were formed as the material was under examination for its potential use as a refractory.

Speed of extrusion was high and power consumption low.

5.3.3 Composite Blend of Bottom Quarry Material (No. 1556)

2 parts	of white	material	(No. 1549)
1 part	of yellow	"	(No. 1550)
1 part	of purple	"	(No. 1551)

A batch was prepared for extrusion at a moisture content of 20.5 per cent.

5.3.3.1 Not De-Aired

When extruded not de-aired the material was very short and weak.

The columns developed severe dog-ear cracks during extrusion.

Speed of extrusion was high and power consumption low.

5.3.3.2 De-Aired

When de-aired the blend was found to be stiff and clean to handle but rather short and not as strong as the composite sample (No. CM. 1548) from the top quarry. However, a plain ribbon (tile) column could be extruded and pressed into Marseilles pattern tiles which were rather weak to handle in the plastic state.

Speed of extrusion was high and power consumption low.

5.3.4 Composite Blend of Material from Top and Bottom Quarries (No. 1557)

A batch of this blend, composed of -

4 parts	of top quarry material	(No. 1548)
2 parts	of white bottom quarry material	(No. 1549)
1 part	of yellow " " "	(No. 1550)
1 part	of purple " " "	(No. 1551)

was prepared for extrusion at a moisture content of 21.0 per cent.

5.3.4.1 Not De-Aired

When extruded not de-aired the material was weak and short. Columns developed dog-ear cracks on extrusion.

Extrusion speed was high and power consumption low.

5.3.4.2 De-Aired

When extruded de-aired the blend was tough, strong, plastic and clean to handle. However, some extruded columns tended to form notches on the edges.

Sockets could be formed on pipes.

The speed of extrusion was high and the power consumption low.

5.4 Drying Behaviour of Specimens

5.4.1 Pressed Briquettes

No faults developed in pressed briquettes when separate samples were dried in two different ovens, one of which was maintained at 105-110°C and the other at 40°C (i.e., heat wave conditions). Drying shrinkage was nil.

5.4.2 Extruded Ware

5.4.2.1 Composite Top Quarry Material (No. 1548)

No faults developed in ware made from this material when dried in the two ovens.

The linear drying shrinkage was 6.0 per cent.

5.4.2.2 Selected White Material from Bottom Quarry (No. 1549)

No oven drying tests were conducted on specimens made from this material as the number and type were very limited. The linear drying shrinkage was 5 per cent.

5.4.2.3 Blend of Bottom Quarry Material (No. 1556)

No faults developed when specimens made from this material were dried in the two ovens. The linear drying shrinkage was 4.0 per cent.

5.4.2.4 Blend of Top and Bottom Quarry Materials (No. 1557)

Solid and perforated briquettes and pipes made from this material developed no faults when dried in the two ovens. However, tiles tended to warp.

The linear drying shrinkage was 5 per cent.

5.5 Firing Behaviour

When dry the pressed and de-aired extruded briquettes were fired over the temperature range 800-1200^{0}C. Specimens made from the white material were also fired to 1250 and 1300^{0}C.

The general appearance of the briquettes was noted and their linear shrinkage was measured.

The results are shown in Tables 1 to 4.

TABLE 1A: LINEAR SHRINKAGE AND GENERAL APPEARANCE AFTER FIRING
Top Quarry - Stockpile Sample (No. 1548)
Pressed Briquettes
Linear Drying Shrinkage Nil

Temp ^{0}C	Linear Shrinkage Firing %	Total %	Appearance after Firing
800(AE)	0	0	Soft and chalky, poor ring and rather weak. Fine mottled salmon pink colour.
850(AD)	0	0	As above
900(AC)	0	0	Slightly stronger and better ring than above
950(AB)	0.5	0.5	As above
1000(A)	1.5	1.5	Better ring, otherwise similar to above.
1050(B)	2.3	2.3	As above
1100(C)	3.0	3.0	Strong and better ring. Slightly darker colour.
1150(D)	5.0	5.0	*Hard and good ring. Light mottled brown colour. Suggested optimum firing temperature.
1200(E)	5.8	5.8	Hard and very good ring. Low porosity. Slightly darker colour.

* Suggested optimum firing temperature 1150^{0}C.

TABLE 1B: LINEAR SHRINKAGE AND GENERAL APPEARANCE AFTER FIRING
Top Quarry - Stockpile Sample (No. 1548)
De-Aired Extruded Briquettes
Linear Drying Shrinkage 6 per cent

Temp	Linear Shrinkage		Appearance after Firing
	Firing	Total	
°C	%	%	
800 (AE)	0	6	Not very hard but good ring. Salmon pink colour but lighter than the pressed ones.
850 (AD)	0	6	Slightly harder. Good ring. Slightly lighter colour than above.
900 (AC)	0.5	6.5	Slightly harder. Good ring. Same colour as above.
950 (AB)	0.5	6.5	Slightly harder. Good ring. Same colour as above, but slightly darker flashing due to salt scum.
1000 (A)	1.0	7.0	*Harder, otherwise same as above.
1050 (B)	2.5	8.5	Harder. Darker due to salt scum.
1100 (C)	3.75	9.75	Very sound ring. Light red brown colour with darker flashing due to salt scum.
1150 (D)	5.4	11.4	Darker than above.
1200 (E)	6.2	12.2	Hard. Medium brown colour

* Suggested optimum firing temperature of 1025°C

TABLE 2A: LINEAR SHRINKAGE AND GENERAL APPEARANCE AFTER FIRING
Bottom Quarry - White Material (No. 1549)
Pressed Briquettes
Linear Drying Shrinkage Nil

Temp °C	Linear Shrinkage Firing %	Linear Shrinkage Total %	Appearance after Firing
800 (AE)	0	0	Soft and chalky. Poor ring. Off-white colour.
850 (AD)	0	0	As above.
900 (AC)	0	0	Good ring. Becoming harder.
950 (AB)	0	0	As above.
1000 (A)	0.5	0.5	As above.
1050 (B)	1.0	1.0	As above.
1100 (C)	1.6	1.6	*Hard with good ring. Cream colour.
1150 (D)	2.7	2.7	Good ring and hard. Cream colour.
1200 (E)	4.0	4.0	As above.
1250 (F)	6.0	6.0	Hard. Darker cream colour. Vitrified. Small pockets of incipient fusion.
1300 (G)	6.8	6.8	Hard. Grey colour. Small pockets showing more pronounced fusion.

* Suggested optimum firing temperature of 1100°C.

TABLE 2B: LINEAR SHRINKAGE AND GENERAL APPEARANCE AFTER FIRING
Bottom Quarry - White Material (No. 1549)
De-Aired Extruded Briquettes
Linear Drying Shrinkage 5 per cent

Temp	Linear Shrinkage		Appearance after Firing
	Firing	Total	
°C	%	%	
800 (AE)	0	5	Soft and chalky. Off-white colour.
850 (AD)	0	5	" " " " "
900 (AC)	0	5	" " " " "
950 (AB)	0	5	Becoming harder. Moderate ring. Off-white colour.
1000 (A)	1.25	6.25	Good ring. Moderately hard. Cream colour.
1050 (B)	1.8	6.8	* Good ring. Better strength. Cream colour.
1100 (C)	2.5	7.5	Good ring. Harder. Cream colour.
1150 (D)	4.0	9.0	As above.
1200 (E)	5.0	10.0	As above.
1250 (F)	7.4	12.4	Hard. Dark cream colour. Small pockets showing incipient fusion.
1300 (G)	7.8	12.8	Vitrified. Grey colour. Small pockets showing more pronounced fusion.

* Suggested optimum firing temperature for cream bricks is 1050°C.

TABLE 3A: LINEAR SHRINKAGE AND GENERAL APPEARANCE AFTER FIRING
Blend of Bottom Quarry Material (No. 1556)
Pressed Briquettes
Linear Drying Shrinkage Nil

Temp ^{0}C	Linear Shrinkage Firing %	Linear Shrinkage Total %	Appearance after Firing
800 (AE)	0	0	Soft and chalky. Poor ring. Fine mottled pale brownish salmon pink colour.
850 (AD)	0	0	As above.
900 (AC)	0	0	Ring improving. Colour as above.
950 (AB)	0	0	Becoming harder. Same colour.
1000 (A)	0. 3	0. 3	As above, but some flashing due to salt scum.
1050 (B)	0. 8	0. 8	Good ring. Same colour as above and some flashing.
1100 (C)	1. 3	1. 3	*Hard. Good ring. Slightly darker mottled colour. Some flashing.
1150 (D)	3. 0	3. 0	Darker colour.
1200 (E)	4. 7	4. 7	Hard. Good ring. Dark mottled brown colour. Darker coloured flashing.

* Suggested optimum firing temperature of 1100^{0}C.

TABLE 3B: LINEAR SHRINKAGE AND GENERAL APPEARANCE AFTER FIRING
Blend of Bottom Quarry Material (No. 1556)
De-Aired Extruded Briquettes
Linear Drying Shrinkage 4 per cent

Temp °C	Linear Shrinkage Firing %	Linear Shrinkage Total %	Appearance after Firing
800 (AE)	0	4	Moderate ring. Not very hard. Pale salmon pink colour.
850 (AD)	0	4	As above
900 (AC)	0.5	4.5	Slightly harder and better ring. Same colour as above.
950 (AB)	0.5	4.5	Slightly harder and better ring. Slightly darker colour. Some flashing.
1000 (A)	1.0	5.0	Hard. Good ring. Slightly darker colour. Some flashing.
1050 (B)	2.0	6.0	*Harder and slightly darker colour. Pronounced flashing.
1100 (C)	3.0	7.0	Hard. Darker than above.
1150 (D)	5.2	9.2	Darker and harder. Some flashing.
1200 (E)	6.4	10.4	Hard. Pale brown colour. Some flashing.
1250 (F)	8.6	12.6	Very hard. Vitrified. Dark grey colour. Scumming on ends.

* Suggested optimum firing temperature of 1075°C.

TABLE 4A: LINEAR SHRINKAGE AND GENERAL APPEARANCE AFTER FIRING
Blend of Top and Bottom Quarry Material (No. 1557)
Pressed Briquettes
Linear Drying Shrinkage Nil

Temp °C	Linear Shrinkage Firing %	Linear Shrinkage Total %	Appearance after Firing
800 (AE)	0	0	Soft and chalky. Poor ring. Fine mottled pale brownish salmon pink colour.
850 (AD)	0	0	As above.
900 (AC)	0	0	As above.
950 (AB)	0.3	0.3	Ring improving but still soft. Colour same as above with some flashing.
1000 (A)	0.5	0.5	As above.
1050 (B)	1.4	1.4	As above.
1100 (C)	2.1	2.1	Good ring, becoming harder. Slightly darker colour.
1150 (D)	3.3	3.3	*Good ring. Hard. Same colour as above.
1200 (E)	5.7	5.7	Very good ring. Hard. Mottled purplish brown colour.

* Suggested optimum firing temperature of 1150°C.

TABLE 4B: LINEAR SHRINKAGE AND GENERAL APPEARANCE AFTER FIRING
Blend of Top and Bottom Quarry Material (No. 1557)
Extruded De-Aired Briquettes
Linear Drying Shrinkage 5 per cent

Temp °C	Linear Shrinkage Firing %	Linear Shrinkage Total %	Appearance after Firing
800 (AE)	0.7	5.7	Good ring but rather soft. Salmon pink colour.
850 (AD)	0.7	5.7	As above but paler colour. Dark coloured flashing.
900 (AC)	0.7	5.7	Good ring but not very hard. Same colour as above with some flashing.
950 (AB)	0.7	5.7	Harder. Good ring. Same colour as above but severe flashing.
1000 (A)	1.1	6.1	As above.
1050 (B)	1.8	6.8	*Hard. Good ring. Slightly darker colour.
1100 (C)	3.1	8.1	As above.
1150 (D)	5.0	10.0	Hard. Sound ring. Darker colour.
1200 (E)	6.0	11.0	Hard. Sound ring. Dark brown colour.
1250 (F)	8.0	13.0	Very hard. Vitreous. Good ring. Grey colour.

* Suggested optimum firing temperature of 1050°C.

Some specimens showed pronounced flashing on edges and faces, particularly those made of material from the bottom quarry. The flashing is very probably due to the presence of soluble salts in the raw material.

Pressed ware requires a higher firing temperature than extruded ware. A suggested optimum firing temperature for ware made from the different materials is given in Table 5.

TABLE 5: SUGGESTED OPTIMUM FIRING TEMPERATURE

Material	Method of Forming	Optimum Temp °C
Top quarry		
(Stockpile No. 1548)	Pressed	1150
	Extruded de-aired	1025
Bottom quarry		
(Best white No. 1549)	Pressed	1100
	Extruded de-aired	1050
Bottom quarry		
(Blend No. 1556)	Pressed	1100
	Extruded de-aired	1075
Blend of top and bottom quarries		
(No. 1557)	Pressed	1150
	Extruded de-aired	1050

Miniature roofing tiles made from the composite sample of the top and bottom quarries and blends thereof fired satisfactorily without distortion.

In the manufacture of roofing tiles and sewer pipes the materials from the top and bottom quarries or blends of these together would have to be fired to at least 1150°C. At this stage the materials appear to be promising but further work involving blending with other materials for this type of ware should be undertaken.

The white material from the bottom quarry cannot be regarded as refractory because incipient fusion takes place at 1250°C. Apart from its colour, no appreciable difference was noted when compared with specimens of the other materials, fired at the same temperature.

5.6 Transverse Strength (Modulus of Rupture)

Bars formed by the pressing and extrusion methods were tested for their transverse strength in the unfired and fired state. Unfired specimens were dried at 105-110°C before being tested whereas fired ones were soaked in water for 24 hours. The results are shown in Table 6.

TABLE 6: TRANSVERSE STRENGTH – (MODULUS OF RUPTURE)

Material	Strength (psi)					
	Dry Unfired	After Firing to 1000°C	1050°C	1100°C	1150°C	1200°C
Top Quarry (No. 1548)						
Pressed	81	-	377	587	757	1185
Extruded not de-aired	346	1047	-	-	-	-
Extruded de-aired	432	1323	-	-	-	-
Bottom Quarry best white (No. 1549)						
Pressed	194	-	682	1025	1252	1947
Bottom Quarry Blend (No. 1556)						
Pressed	81	-	332	870	985	2090
Extruded de-aired	177	698	-	-	-	-
Extruded de-aired) Oil lubricated)	241	787	-	-	-	-
Blend of Top and Bottom Quarries (No. 1557)						
Pressed	124	-	335	555	755	1632
Extruded de-aired	244	1017	-	-	-	-

The results indicate that the strength of all unfired bars was low. Extruded de-aired ones were considerably better than those pressed or extruded not de-aired. Bars made from the top quarry material gave a higher unfired strength than those made from the bottom quarry material.

Specimens pressed from the white material developed better strength than those made from other materials when fired at the same temperatures in the range 1050-1150°C.

On the whole the strength of pressed bars did not develop until they had been fired to at least 1100-1150°C. This was found to be in agreement with the remarks on the fired briquettes (see Tables 1 to 4).

The strength of extruded bars was not high at 1000°C although it was similar to that of the pressed ones fired at 1150-1200°C.

5.7 Efflorescence

A small amount of efflorescence developed on briquettes which had been fired at temperatures up to and including 1050°C. No efflorescence was detected on specimens which had been fired above 1050°C.

6. CONCLUSIONS AND RECOMMENDATIONS

The results of this investigation have shown that good bricks can probably be made from the materials by the pressing or de-aired extrusion method. A composite blend of material from both quarries will probably lend itself to the stiff de-aired extrusion process as the rate of extrusion was found to be high and the power consumption low. Extrusion by the not de-aired method is not recommended.

The ware will need to be well burnt to acquire adequate strength whatever the method of manufacture. In particular if the pressing method is adopted it will be necessary to fire to a relatively high temperature for sound ware. On the other hand should the de-aired extrusion process be used, a rather lower temperature will be necessary but still higher than local practice.

From the refractories point of view the white material (No. 1549) has no better overall heat resisting properties than the other materials and cannot therefore be recommended for use in refractories. In fact when fired to 1250°C it developed small pockets of incipient fusion. However it can be regarded as the best of the series of materials examined.

Whilst it was possible to make pipes and roofing tiles from the materials in the laboratory it would seem necessary to add a plastic bond or pipe clay to the shale for the satisfactory manufacture of this type of ware on an industrial scale. From this point of view the materials appear to be promising but the question of blending with other raw materials has not been dealt with and therefore no definite recommendations can be made in this respect.

The colour of fired ware was somewhat erratic due to flashing caused, presumably, by the presence of soluble salts in the raw material. This was more pronounced with material from the bottom quarry.

As a result of the investigation it would seem that the deposit would be best utilised by an overall blending and stock piling of all the different types of material present. Selective mining is likely to work out the best material and leave the lower quality material which may be difficult to utilise at a later date.

At this stage it can be said that the deposit is promising but there appear to be potential difficulties such as the relatively low strength of ware in the green and fired states and variation in the fired colour.

Finally it is recommended that the whole deposit be bored in a systematic grid pattern to determine the quality, quantity and consistency of the material present in the 80-acre site. This will govern the overall blending and stockpiling of the material and so make it possible to utilise the whole deposit to the best advantage.

If a large modern works is contemplated on or near the site and we sincerely hope that this will eventuate, it is recommended that further laboratory work and/or works trials be undertaken on the material available after the recommended drilling programme has been completed.