RB 53/13

AUSTRALIAN MINERAL DEVELOPMENT LABORATORIES

REPORT No. AMDL-73

AN INVESTIGATION INTO RAW MATERIALS FOR THE MANUFACTURE OF ROOFING TILES IN SOUTH AUSTRALIA

by

H. Ellerton.

This report describes work undertaken by the Ceramics Research Section in Adelaide jointly sponsored by the C.S.I.R.O. and the South Australian Government. The work of this group was under the supervision of H. Ellerton, Senior Research Officer, C.S.I.R.O.

Issued November 1960. L. Wallace Coffer, Director.

CONTENTS

ABSTRACT

Raw materials used in the manufacture of Marseille pattern roofing tiles have been exhaustively examined and an optimum blend suggested.

The extrusion moisture content range suitable for repressing tiles has been found to be somewhat limited, namely of the order of $1\frac{1}{4}\%$.

The incorporation of Woodside fireclay has been found to increase the rather limited firing range of the materials at present in use, and should therefore make it possible to salt glaze a higher percentage of ware.

A kiln survey has revealed a wide temperature variation in different parts of some kilns. Oil firing by induced draught and soaking for a longer period during salt glazing when incorporating Woodside fireclay should reduce this temperature variation and at the same time increase the percentage of salt glazed ware without increasing firing losses.

INTRODUCTION

During the past 40 years roofing tiles of the Marseille pattern have been manufactured in South Australia from Cherry Gardens clay as the chief raw material.

When Modbury pipe clay was more readily available a small percentage was added to impart greater plasticity.

However, since the introduction (1, 2) of de-aired shale-clay blends in the manufacture of building bricks in this State, a similar procedure has been applied to roofing tile manufacture with some success. In one particular case a weathered shale, also from Cherry Gardens, was initially mixed with the clay in the proportions of 3/1 but latterly in equal proportions. Whilst this mix has been found to give quite good results at the present rate of production, some uncertainty has been felt as to whether it was the best possible blend from the point of view of extrusion, drying and firing properties.

Also, at present, low firing losses can be sustained with a production of 35 - 45% salt glazed ware, but in general, losses due to overfiring have been found to increase as the percentage of salt glazed ware exceeds these figures.

However, an increased public demand for salt glazed roofing tiles has made it imperative to increase the percentage of this ware produced from each kiln, and in order to meet this demand a comprehensive investigation has been made into the potentialities of not only the materials already in use, but also others which might be readily available within a reasonable distance (approx. 25 miles) of Adelaide.

The investigation described in this report has been divided into three main sections namely:-

(1) A detailed investigation into the blending of different proportions of Cherry Gardens clay and shale in order to find, from the overall properties of the materials, whether the present mix is the optimum one.

(2) The addition or substitution of alternative materials in order to (a) improve the working properties of the mix now in use, (b) increase the safe firing temperature of the ware.

(3) An investigation into the firing temperature and salt glazing of the ware in different parts of industrial kilns at present burning salt glazed roofing tiles.

The investigation has not proved to be an easy one, as the materials in use have been giving satisfactory results and low losses, apart from the fact that the production of salt glazed ware has been somewhat limited in quantity.

Some idea of the magnitude of the work involved will be gained when it is realised that in the course of the investigation, 8 different raw materials were incorporated in the blends in one form or another and that in all 70 full scale laboratory extrusion experiments, together with their accompanying tests, were carried out. Also in the course of the investigation at least 3,000 laboratory specimens were prepared for firing over a wide temperature range.

As the clay mix is at present being processed through a de-airing pug mill on the industrial scale, particular attention was given to this aspect of the investigation. Only in the initial stages of the work was the material passed through the laboratory pug mill in the not de-aired state. As soon as de-airing was attempted a marked improvement in the working properties of the materials was observed. This is in agreement with work previously undertaken on South Australian raw materials [1].

EXPERIMENTAL PROCEDURE AND RESULTS

1. The Cherry Gardens Clay and Shale

Material Examined.

The clay and shale are located at Cherry Gardens, some 13 miles S.S.E. of Adelaide.

The Clay occurs in part of Section 783, Hundred of Noarlunga, County of Adelaide.

The Shale is situated near a pine forest on adjoining property in Section 786, Hundred of Noarlunga, County of Adelaide.

The two materials are won by mechanical means and transported by road to Adelaide where they are stockpiled.

The two stockpiles were sampled and the materials allowed to air dry in the laboratory prior to being ground to 20's mesh ~~BSS~~. I.M.M.

Properties of the Materials.

The slaking, extrusion, drying and firing properties of the clay and shale were first examined individually. Then the properties of blends of the two materials in different proportions were investigated.

Clay (Lab. No. 1290) The clay was brown in colour and contained a few pieces of light coloured (underlying) shale-like material. It was found to be easy to crush and had a tendency to flake.

Slaking Properties. Small pieces of the unground clay were found to slake in approximately 12 minutes, but the lighter coloured shale-like material, although water absorbent, was unaffected over an extended period of several days.

Extrusion Behaviour (De-aired). A batch of the clay was prepared for extrusion with a water content of 20.5%. Although it was possible to extrude the clay, its speed was slow and a rather high power consumption was indicated by the meter attached to the machine.

Drying Test. In a drying test specimens cracked badly in a severe drying oven, but similar specimens were less affected under more moderate conditions.

Drying Shrinkage . The drying shrinkage was rather high at 7.5%.

After careful and thorough drying, specimens were fired in small batches to cover as wide a temperature range as practicable and compared with the shale (see Table 1).

Shale (Lab. No. 1291). The shale was pale light brown or fawn in colour, containing hard and soft material. It crushed easily but had a tendency to flake somewhat.

Slaking Properties. Soft pieces slaked down in approximately 10 minutes but harder ones, although water absorbent, remained unaffected over a period of several days.

Extrusion Behaviour (De-Aired). A batch of the shale was prepared for extrusion containing 26% moisture. It was faster to extrude and required considerably less power than the clay. It was found to be thixotropic, that is, it hardened on standing but became soft again on being worked.

It was shorter and less plastic than the clay even when de-aired and the extruded column developed faults varying from "dog-ear" cracks to small notches on the edges of the column, depending upon the type of die used.

When an attempt was made to press miniature tiles of the Marseille pattern from an extruded ribbon, the specimens so formed were weak and too soft to handle without deforming or breaking.

Drying Test. When subjected to the severe and moderate drying conditions, no specimens which had been extruded from the various dies showed any sign of cracking.

Drying Shrinkage. The drying shrinkage was only 4.5%.

Firing Tests. Carefully and thoroughly dried specimens were fired in small batches along with those made from the clay. The shrinkage and general appearance after firing are shown in Table 1.

TABLE 1. LINEAR SHRINKAGE AND GENERAL APPEARANCE AFTER FIRING

Firing Temperature	Clay Shrinkage Wet to Dry 7.5% Fired	Clay Shrinkage Total	Clay Remarks	Shale Shrinkage Wet to Dry 4.5% Fired	Shale Shrinkage Total	Shale Remarks
800°C	0%	7.5%	Hard, good ring, red colour.	0%	4.5%	Soft & chalky, salmon pink colour, poor ring.
850	0.5	8.0	" "	0	4.5	" "
900	1.0	8.5	" "	0	4.5	" "
950	1.3	8.8	" "	0	4.5	" "
1000	2.0	9.5	" "	0.9	5.4	" "
1050	3.0	10.5	" "	3.5	8.0	Soft, medium ring, salmon pink colour.
1100	5.5	13.0	Bloating commenced, darker colour.	4.5	9.0	Moderate ring, harder & darker.
1150	5.0	12.5	Bloated	5.0	9.5	Good ring, hard & red.
1200				8.3	12.8	Good ring, hard & darker.
1250				9.1	13.6	Very hard, dark maroon, semi-vitreous.
1300				9.2	13.7	Vitrified, dark brown.

From an examination of the fired specimens it was found that the clay matured at a lower temperature than the shale. Bloating of the clay commenced at 1100°C and became quite pronounced at 1150°C.

On the other hand, briquettes made from the shale were quite soft in the lower section of the firing range and did not commence to harden until 1050°C was reached. No bloating was apparent even at 1300°C, although the specimens became quite vitrified at this temperature.

After evaluating the various properties of the clay and shale, blending the two together was taken into consideration.

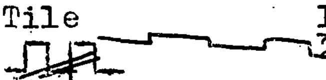
As one factory in particular is using the 1:1 blend, this was used as a starting point, followed by clay/shale blends in the proportions of 1:2 and 2:1.

It was found that the water of plasticity (or extrusion moisture content) of the blends was much lower than that of the shale alone, but the rate of extrusion and power consumption were nearer to those of the shale than the clay, the speed increasing and the power decreasing as the clay content decreased and the shale content increased.

Several different dies were used because a much better idea of the extrusion properties of materials can usually be obtained in this way rather than using only one die in an investigation of this nature.

The results are shown in Table 2.

TABLE 2. EXTRUSION PROPERTIES OF MATERIALS

Lab. No.	1290	1305	1303	1304	1291
Clay/Shale Blend.	Clay alone	2:1	1:1	1:2	Shale alone
Extrusion Moisture.	20.5%	20.75%	21.5%	21.75%	26%
Drying Shrinkage.	7.5%	6.5%	6.0%	4.5%	4.5%
Die Used.	Rate of Extrusion		Inches per Minute / Watts		
2" x 1" solid	40/2440	61/1850	64/2130	59/1830	58/1870
2" x 1" 3 large hole	52/2720	83/1980	87/2070	86/1950	91/1960
2" x 1" 6 hole	42/2680	71/1960	76/2060	82/1960	
2" x 1" 10 hole	39/2625	72/1980	70/2060	73/1980	
Tile (Ribbon)	118/2920	239/2120	259/2200	284/2160	322/1930
Tile 	104/2660	221/2080	224/2160	265/2200	262/1950
$1\frac{1}{2}$" x 1" standard solid briquette	57/2760	73/1910	81/2070	80/1960	82/1870
Pipe	67/2560	116/2110	129/2180	133/2180	144/1980

Faults such as "dog-ear" cracks and notching which were visible on the extruded shale column were less apparent in the case of the blends, the decreasing order of such defects being:- slight in the 1:2 clay/shale blend, very slight in the 1:1, and nil in the 2:1 blend.

In the severe drying test, cracking ranged from nil in the case of the shale, slight for the 1:2 blend, moderate for the 1:1 blend and severe for the 2:1 blend and the clay.

Although the extrusion moisture content was highest for the shale, the clay together with the 2:1, and 1:1 blends had a higher shrinkage than the shale alone or the 1:2 blend.

After thorough drying, briquettes from each blend were fired in small batches over a wide temperature range. The fired and total linear shrinkage figures are shown in Table 3.

It has already been pointed out (see Table 1) that the clay matures at a much lower temperature than the shale and the blends were found to be intermediate between the clay and the shale in this respect. The 1:1 blend had the lowest firing shrinkage but its total shrinkage was found to be higher than the 1:2 blend which had a lower drying shrinkage. However, taking all relevant factors into consideration, the most suitable blend would appear to lie between the 1:1 and 1:2 clay/shale ratios.

After investigating the possibilities of adding to or substituting other materials for the Cherry Gardens clay and/or shale without success (see section 2(a)), a works prepared blend was compared in some detail with laboratory prepared mixes.

All laboratory blends are measured by weight and the inherent moisture of the components taken into account. Works proportioning is usually by volume - for example, by mechanical shovel.

A laboratory investigation into the relationship between the weight and volume batching showed that a 20's mesh ~~BSS~~ I.M.M. compacted 1:1 volume blend was approximately equivalent to a 3:2 weight blend.

Further clay/shale weight blends were, therefore, made in the ratios of 1:1, 3:2 and 2:1 and compared with a sample of a works prepared 1:1 volume blend, which had been reground to 20's mesh ~~BSS~~ I.M.M. in the laboratory.

All extrusions were made as soon as possible after each other so that a close comparison could be made between the mixes. Likewise, drying and firing tests were compared.

TABLE 3. LINEAR SHRINKAGE

Firing Temperature	Clay (1290)		Clay/Shale Blends 2:1 (1305)		Clay/Shale Blends 1:1 (1303)		Clay/Shale Blends 1:2 (1304)		Shale (1291)	
	Wet to Dry 7.5%		Wet to Dry 6.5%		Wet to Dry 6.0%		Wet to Dry 4.5%		Wet to Dry 4.5%	
	Fired	Total	Fired	Total	Fired	Total	Fired	Total	Fired	Total
800°C	0%	7.5%	0%	6.5%	0%	6%	0%	4.5%	0%	4.5%
850	0.5	8.0	0	6.5	0	6.0	0	4.5	0	4.5
900	1.0	8.5	0	6.5	0	6.0	0	4.5	0	4.5
950	1.3	8.8	0	6.5	0	6.0	1.0	5.5	0	4.5
1000	2.0	9.5	1.5	8.0	1.0	7.0	1.5	6.0	0.9	5.4
1050	3.0	10.5	2.5	9.0	2.5	8.5	3.5	8.0	3.5	8.0
1100	5.5*	13.0	4.5	11.0	3.5	9.5	4.5	9.0	4.5	9.0
1150	5.0*	12.5	5.0	11.5	4.5	10.5	5.0	9.5	5.0	9.5
1200			5.0	11.5	6.0	12.0	7.0	11.5	8.3	12.8
1250			4.5*	11.0	6.0	12.0	7.0	11.5	9.1	13.6
1300			5.5*	12.0	5.5*	11.5	5.5*	10.0	9.2	13.7

* Bloated

Taking all factors into consideration, it was found that the laboratory 3:2 blend was the nearest to the works 1:1 blend. The laboratory 1:1 blend had a less tendency to crack than the factory 1:1 blend under severe drying conditions.

Earlier laboratory work has already indicated that the best blend appears to lie between the 1:1 and 1:2 clay/shale blends. The factory 1:1 volume blend is outside this range as it is nearer to the laboratory 3:2 blend. A factory 1:2 clay/shale blend should therefore give better all round results as it would compare more favourably with a laboratory 2:3 clay/shale blend.

2(a) <u>The Addition or Substitution of other Materials for the Cherry Gardens Clay and Shale</u>.

In an effort to improve the overall properties of the blend, other materials were added to or substituted for the clay or shale. The materials used included those available or likely to be available within a reasonable distance of the metropolitan area.

<u>Echunga Shale</u>

Bearing in mind that one manufacturer in particular has for some time past contemplated moving from the metropolitan area to a site near Echunga, a material from that area was incorporated in the work. This material, known as Echunga shale was examined in the laboratory some time ago, and although it was not suitable for use on its own, it was thought that it might be suitable for blending with other materials. It is a highly weathered shale-like material almost white in colour and occurring in Section 357, Hundred of Kuitpo, County of Adelaide, some 21 miles S.E. of Adelaide. When the site near Echunga was inspected during the earlier investigation of the material the deposit appeared to be extensive. For the above reasons, therefore, the material was investigated for its possible utilisation in the manufacture of roofing tiles.

<u>Alluvial Clay</u>

An alluvial clay, typical of those used in the manufacture of bricks and pipes and occurring in Sections 370 and 371, Hundred of Yatala, County of Adelaide in the Croydon suburb approximately 4 miles N.W. of the Adelaide G.P.O. was tried.

Washed Clay

A highly plastic washed clay from a Golden Grove sand washing plant, 14 miles N.E. of Adelaide and occurring on 128 acres of land in Section 5459, Hundred of Yatala, County of Adelaide, was also examined for its possible utilisation.

After all relevant facts had been gleaned from a considerable number of extrusions, drying and firing experiments incorporating the above materials, it was concluded that none of them was better than those already in use from Cherry Gardens.

The Echunga shale was not as good as that from Cherry Gardens, whilst the alluvial and washed clays were, in many respects, inferior to that from Cherry Gardens. These facts could explain why roofing tile manufacturers have, in the past, preferred Cherry Gardens clay to the alluvial clay occurring in the Adelaide Plains.

However two blends, both with a somewhat limited firing range, could be tried for making a light burning building brick in case an alternative were desirable to replace the dark burning tile blend already in use.

The blends are:

No. 1328 composed of

1 pt. of Cherry Gardens clay ⎫
1 pt. of Echunga shale ⎭ by weight

or,

No. 1329 composed of

3 pts. of Cherry Gardens clay ⎫
2 pts. of Echunga shale ⎭ by weight

2(b) Increase in the Safe Firing Temperature of the Ware.

Material Examined

In this section of the investigation three refractory materials from Cromer, Birdwood and Woodside respectively were added to or substituted for the Cherry Gardens materials both in the raw and the calcined state.

Cromer Clay. The Cromer clay, known as Cromer C clay is a very white kaolinitic material located 2 miles from Birdwood, and some 33 miles E.N.E. of Adelaide. It occurs in Sections 143, 144, 145 and 154, Hundred of Para Wirra, County of Adelaide.

Birdwood Clay. The Birdwood clay is an off-white material occurring in Section 6297, Hundred of Talunga, County of Adelaide, some 30 miles E.N.E. of Adelaide.

Woodside Fireclay. Woodside fireclay is a white highly weathered refractory shale of the Adelaide System. It is less kaolinitic and more siliceous than the other two, although the silica is present in a very finely divided state. This material is located in Section 3966, Head of Onkaparinga, County of Adelaide, some 23 miles east of Adelaide and approximately 2 miles N.E. of Woodside.

Results of Examination

Cromer Clay. The Cromer Clay when incorporated in blends with the Cherry Gardens materials and extruded de-aired did not disperse at all readily. No benefit was obtained from the dry and fired shrinkage point of view by its addition to the blend.

Birdwood Clay. The Birdwood clay dispersed more readily but this likewise did not reduce the shrinkage.

Blends incorporating the Cromer and Birdwood clays were weaker than the Cherry Gardens material blends in the unfired state.

Calcined Clay. Neither the Cromer, the Birdwood nor the Woodside material had any beneficial effect upon the blend when introduced in the calcined state. None of the calcined materials dispersed in the blends which were weaker than the 1:1 blend alone. Furthermore pre-calcining and regrinding would be costly and calcined material could cause more wear and tear on machinery due to abrasion.

Woodside Fireclay. Woodside fireclay in the raw state was found to give the most promising results. Work incorporating this material was therefore pursued in greater detail.

Blends incorporating Woodside Fireclay. The following Cherry Gardens Clay/Shale/Woodside fireclay blends were examined: 2:2:1, 2:1:1, & 1:1:1.

The 2:2:1 blend had the minimum fireclay content and therefore benefited least from its addition.

A works volume blend would contain approximately 5% more clay than the laboratory weight batching blend.

The 1:1:1 blend having the greatest fireclay content received the greatest benefit and showed the highest safe firing temperature although it was not quite as strong as the 1:1 clay shale blend in the unfired state.

The 2:1:1 blend had the highest clay content - a works batch would contain 5% more clay and 5% less shale than the laboratory weight batch. This blend was more plastic and stronger than the other two and had the greatest tendency to crack on drying. All blends incorporating the Woodside fireclay were as good as the Cherry Gardens materials alone from the point of view of extrusion properties.

The Cherry Gardens clay/shale blend had a very high dry (unfired) strength and should, therefore, be capable of carrying the weaker fireclay even in the 1:1:1 blend. However, this 1:1:1 laboratory blend has a somewhat similar clay content to the 1:2 clay/shale blend recommended in the earlier part of this investigation where a works volume blend of 1:2 would be nearer to the 2:3 laboratory weight blend.

The extrusion, transverse strength and firing characteristics of the blends are given in Tables 4, 5 and 6 respectively.

By the addition of Woodside fireclay to the Cherry Gardens materials it should be possible to raise the firing temperature by at least 50°C to 75°C depending upon the blend used.

In comparison with the 1:1 clay/shale blend fired at 1000°C (1832°F) it should be possible to fire the 2:2:1 and 2:1:1 blends to 1050°C (1922°F) and the 1:1:1 blend to 1075°C (1967°F).

No difference could be detected between small batches of pressed Marseille pattern laboratory tiles prepared from extruded batches of the 1:1, 2:2:1, 2:1:1 and 1:1:1 blends when placed side by side after firing to 1000°C, 1050°C, 1050°C & 1075°C respectively.

This should make it possible to salt glaze a much higher percentage of ware without unduly increasing the firing loss.

TABLE 4. EXTRUSION CHARACTERISTICS OF CHERRY GARDENS/ WOODSIDE BLENDS

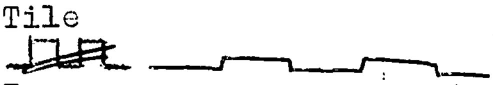
Lab.Reference No.	1451	1452	1453	1454
Blend	1:1	2:2:1	1:1:1	2:1:1
Extrusion Moisture or Water of Plasticity	21.75%	20.9%	21.08%	21.4%
Drying Shrinkage	6.7%	6.6%	6.0%	7.0%
Die Used	Rate of Extrusion Inches per Minute / Watts			
2" x 1" Solid	69/1730	69/1710	70/1730	77/1680
2" x 1" 3 hole block	102/1850	107/1800	104/1840	109/1690
2" x 1" 6 hole block	87/1880	87/1820	89/1870	94/1720
2" x 1" 3 hole briquette	72/1800	76/1780	70/1820	82/1690
2" x 1" 10 hole briquette	83/1850	84/1830	78/2020	82/1730
Plain (ribbon) Tile	340/1890	353/1900	404/1910	374/1750
Tile 	297/1890	328/1910	364/1930	352/1760
Tile 	279/1950	302/1890	328/1910	328/1730
1½" x 1" Standard briquette	82/1800	84/1770	90/1810	97/1700
Pipe	141/1940	141/1880	155/1860	163/1730

TABLE 5. LINEAR SHRINKAGE

Number	1451		1452		1453		1454	
Blend	1:1		2:2:1		1:1:1		2:1:1	
Firing Temperature.	Wet to Dry 6.7%		Wet to Dry 6.6%		Wet to Dry 6.0%		Wet to Dry 7.0%	
	Fired	Total	Fired	Total	Fired	Total	Fired	Total
850°C	0.3%	7.0%	0.2%	6.8%	0.2%	6.2%	0.5%	7.5%
1000	1.2	7.9	0.4	7.0	0.8	6.8	0.9	7.9
1050	2.3	9.0	2.0	8.6	2.0	8.0	1.6	8.6
1100	2.5	9.2	2.1	8.7	2.2	8.2	2.1	9.1
1150	4.0	10.7	3.0	9.6	2.9	8.9	3.0	10.0
1200	4.74*	11.44	3.9	10.5	3.36	9.36	3.4	10.4
1250	4.2*	10.9	4.7*	11.3	3.6	9.6	4.0*	11.0
1300	2.5*	9.2	3.4*	10.0	5.1	11.1	1.6*	8.6
1350			3.0*	9.6	4.6*	10.6	2.5*	9.5

* Bloated

In works practice a firing shrinkage of ¼" to the foot (i.e. 2%) is the target.

TABLE 6.

Modulus of Rupture (Transverse Strength) p.s.i.				
	No. 1451 Blend 1:1	No. 1452 Blend 2:2:1	No. 1453 Blend 1:1:1	No. 1454 Blend 2:1:1
Unfired Strength	1249 psi	1036 psi	901 psi	1100 psi
Fired to				
900°C	2010	1492	1039	1588
950	1876	1550	963	1459
1000	1997	1687	1226	1622
1050	2700	1998	1547	1996
1100	3083	2370	1697	2442
1150	3257	2272	1683	2466
1200	3006	2315	1907	2163

Effect of Variation of Extrusion Moisture Content upon the Extrusion Rate, Power Consumption, Dry & Fired Strength & Shrinkage of the 1:1, 1:1:1 & 2:1:1 Blends.

Batches of these blends were prepared with moisture contents varying in steps of approximately 0.5% above and below what had appeared to be the optimum water content in earlier work (see Table 4).

Particular attention was given to the way in which Marseille pattern tiles could be pressed from the extruded material in each case.

The 1:1 Blend (by wt.) (Lab. No. 1451) The best moisture content for pressing this blend was assessed to lie between 21.3% and 22.6% inclusive, that is, a latitude of approximately 1¼%.

Specimens pressed from batches in the higher moisture range tended to crack and warp in the oven drying test more than those in the lower moisture range. Tiles containing 21.3% moisture were least affected.

A moisture content of 20.6% was the lower limit for extrusion, the speed at this moisture content being the lowest, and the power consumption the highest. At this low moisture content the pressing of tiles was not easy owing to the rather stiff nature of the mix.

The dry strength of this blend was high, the best values being those in the middle of the range, namely 21.8% and 22.1% moisture content. Strength after firing at 1000°C was good, the highest being for that extruded at 21.8% moisture content.

Drying shrinkage increased as the moisture content increased but the firing shrinkage showed a minimum at the centre of the range.

The results are shown in Table 7.

The 1:1:1 Blend (by wt.) Lab. No. 1453). The extrusion moisture content of this blend ranged from 19.9% to 22.6%.

The results are shown in Table 8.

In this blend also, the speed increased and the power decreased as the moisture content increased, but the optimum moisture content for pressing tiles was found to lie between 20.7% and 21.9% inclusive, a range of approximately 1¼%.

The blend behaved extremely well in the severe drying test, only tiles containing 19.9% and 22.6% moisture warping slightly, the 19.9% moisture ones probably due to strains set up in the stiffer mix when deformed during pressing,and the 22.6% ones probably due to having too high a moisture content.

Although this blend was not as strong as the previous one, it should be quite strong enough particularly in the middle of the moisture range.

The drying shrinkage was lower than in the case of the previous blend and was particularly uniform as was also the firing shrinkage.

The 2:1:1 Blend (by wt.) (Lab. No. 1454) This blend was extruded at moisture contents ranging from 19.75 to 22.4%.

In this case also the speed increased and the power consumption decreased as the moisture content increased.

The best moisture content range for pressing tiles lay between 20.2% and 21.9% inclusive, a range of approx. 1¾%.

However, in the drying tests it tended to warp and crack more than the other blends because of its high clay content. Only if either of the other two blends incorporating Woodside fireclay were too weak should this blend be tried.

Its drying shrinkage was as high as that of the 1:1 blend.

The dry strength was not as great as the 1:1 blend but better than the 1:1:1 blend whereas its fired strength was better than either of the other two.

The results are shown in Table 9.

TABLE 7. BLEND 1451 (1:1)

Effect of Extrusion Moisture Content Variation upon Extrusion Rate, Power Consumption, Strength & Shrinkage.

Moisture Content	20.6%	21.3%	21.8%	22.1%	22.6%	23.0%
Die Shape Used	Rate of Extrusion (Inches per Minute / Watts)					
2" x 1" Solid Briquettes	58/1800	60/1745	63/1675	68/1725	70/1660	70/1650
2" x 1" 10 Hole Briquettes	74/1925	83/1860	83/1770	87/1780	83/1710	86/1680
Plain Tile (Ribbon)	307/2010	319/1950	338/1840	310/1830	320/1725	309/1700
$1\frac{1}{2}$" x 1" Solid Briquette	72/1810	73/1800	74/1695	88/1730	90/1700	92/1675
Dry Strength (Modulus of Rupture)	1240 psi	1283 psi	1317 psi	1345 psi	1225 psi	1240 psi
Fired Strength (Fired at 1000°C)	2052 psi	1920 psi	2200 psi	1990 psi	1993 psi	1890 psi
Drying Shrinkage	6.7%	6.9%	7.2%	7.4%	7.6%	7.3%
Firing (1000°C) Shrinkage	1.2%	1.3%	1.1%	1.0%	1.2%	1.5%

TABLE 8. BLEND 1453 (1:1:1)

Effect of Extrusion Moisture Content Variation upon Extrusion Rate, Power Consumption, Strength & Shrinkage.

Moisture Content	19.6%	20.7%	21.0%	21.5%	21.95%	22.6%
Die Shape Used	Rate of Extrusion (Inches per Minute / Watts)					
2" x 1" Solid Briquette	60/1725	70/1660	64/1660	68/1615	66/1665	74/1595
2" x 1" 10 Hole Briquette	74/1805	86/1730	80/1730	85/1660	79/1650	78/1615
Plain Tile (Ribbon)	362/1925	390/1795	369/1800	384/1725	362/1725	386/1650
1½" x 1" Solid Briquette	69/1780	81/1710	75/1725	83/1650	82/1625	93/1615
Dry Strength (Modulus of Rupture)	824 psi	810 psi	894 psi	890 psi	883 psi	865 psi
Fired Strength (Fired at 1075°C)	1430 psi	1640 psi	1585 psi	1540 psi	1640 psi	1585 psi
Drying Shrinkage	5.8%	6.1%	6.1%	6.1%	6.4%	6.5%
Firing (1075°C) Shrinkage	2.2%	2.1%	2.0%	2.1%	2.5%	2.4%

TABLE 9. BLEND 1454 (2:1:1)

Effect of Extrusion Moisture Content Variation upon Extrusion Rate, Power Consumption, Strength & Shrinkage.

Moisture Content	19.75%	20.15%	20.75%	21.4%	21.9%	22.4%
Die Shape Used	Rate of Extrusion (Inches per Minute / Watts)					
2" x 1" Solid Briquette	62/1750	76/1735	66/1630	74/1600	69/1580	77/1590
2" x 1" 10 Hole Briquette	73/1840	86/1820	83/1690	83/1640	86/1625	96/1640
Plain Tile (Ribbon)	340/2030	356/1870	355/1775	365/1710	346/1640	328/1630
1½" x 1" Solid Briquette	74/1820	80/1760	84/1675	85/1590	92/1600	98/1620
Dry Strength (modulus of Rupture)	1018 psi	1130 psi	1145 psi	1192 psi	1130 psi	1083 psi
Fired Strength (Fired at 1050°C)	2195 psi	2135 psi	2135 psi	1925 psi	1870 psi	1800 psi
Drying Shrinkage	6.8%	7.0%	7.2%	7.4%	7.5%	7.6%
Firing (1050°C) Shrinkage	2.3%	2.3%	2.0%	2.2%	2.3%	2.0%

Conclusions.

Summarising the results of this section of the investigation it can be concluded that the extrusion moisture content range for optimum extrusion, pressing, drying and firing shrinkage and strength, is somewhat limited, the margin being only $1\frac{1}{4}$ to $1\frac{3}{4}$%. Too high a moisture content could lead to greater shrinkage and more tendency to warp. This is important particularly in the case of a material such as the 1:1 blend which is on the borderline.

However, the incorporation of the Woodside fireclay in a blend such as the 1:1:1 could reduce the risk of drying losses and increase the firing range.

A determination of the moisture content of the column of the 1:1 volume blend during extrusion on a factory gave a figure of 21% whereas the laboratory 3:2 blend which corresponds to the factory 1:1 blend was extruded at a moisture content of 20.8%. This indicates how, by experience, it is possible to attain the optimum working properties of a raw material in factory practice.

3. A Temperature & Salt Glazing Distribution Survey of Four Industrial Kilns.

Initially it was intended to examine only two kilns (No. 1 & 5) but the results were so interesting that it was later arranged to examine two other kilns (No. 4 & 8) as well.

3 "bolts" or vertical sections were examined in numbers 1, 5 & 8 Kiln and 5 "bolts" in number 4 kiln.

Thermoscopes and test pieces were placed in 8 different positions in each "bolt", 3 in the bottom, 3 half way and 2 in the top as illustrated in the diagrams. The trial number, the temperature recorded and the degree of glazing, if any, are also indicated in the diagram.

The results are given below.

No. 1 Kiln - Coal Fired, Induced Draught.

The Centre Bolt. In the centre bolt the temperature variation of 32°C was not excessive, the lowest reading being 963°C in positions 7 and 8 and the highest 995°C in position No.3.

Trial specimens were only glazed in 3 positions, completely in No. 1 and incompletely in Nos. 2 and 3.

The $\frac{1}{4}$ Bolt. The bolt approximately $\frac{1}{4}$ the length of the kiln from the end, had a greater variation than the centre or the one near the end. The highest temperature recorded was 1025°C in position No. 13 and the lowest, 965°C, in position No. 14. However, more salt glazing had taken place in this bolt than in the previous one.

<u>The Bolt 1 from the End of the Kiln.</u> In the bolt 1 from the end of the kiln, there was only a temperature variation of 23°C.

<u>Comments.</u>

Complete salt glazing occurred in the top position, No. 18, but incomplete glazing occurred in positions Nos. 17, 20 and 21, and only slight glazing in Nos. 19, 23 & 24.

The highest temperature recorded in any of the 3 bolts in this kiln was 1025°C and the lowest 960°C, a difference of 65°C.

The highest temperatures recorded in the centre and ¼ bolts were not, as might have been expected, in the top of the kiln but half way down, probably in the vicinity of a firebox.

A considerable amount of fly ash was deposited on the trial specimens throughout the kiln particularly at the top where it caused a rough surface on the salt glazed ware.

<u>No. 5 Kiln - Oil Fired, Induced Draught.</u>

<u>The Centre Bolt.</u> The centre bolt did not show an excessive temperature variation, there being a difference of 40°C between the highest temperature of 1000°C at position No. 26 and the lowest, 960°C at position No. 30

Salt glazing occurred only in the top course, trial Nos. 25 and 26 being completely glazed. No glazing of specimens occurred on any trials in the middle or bottom courses.

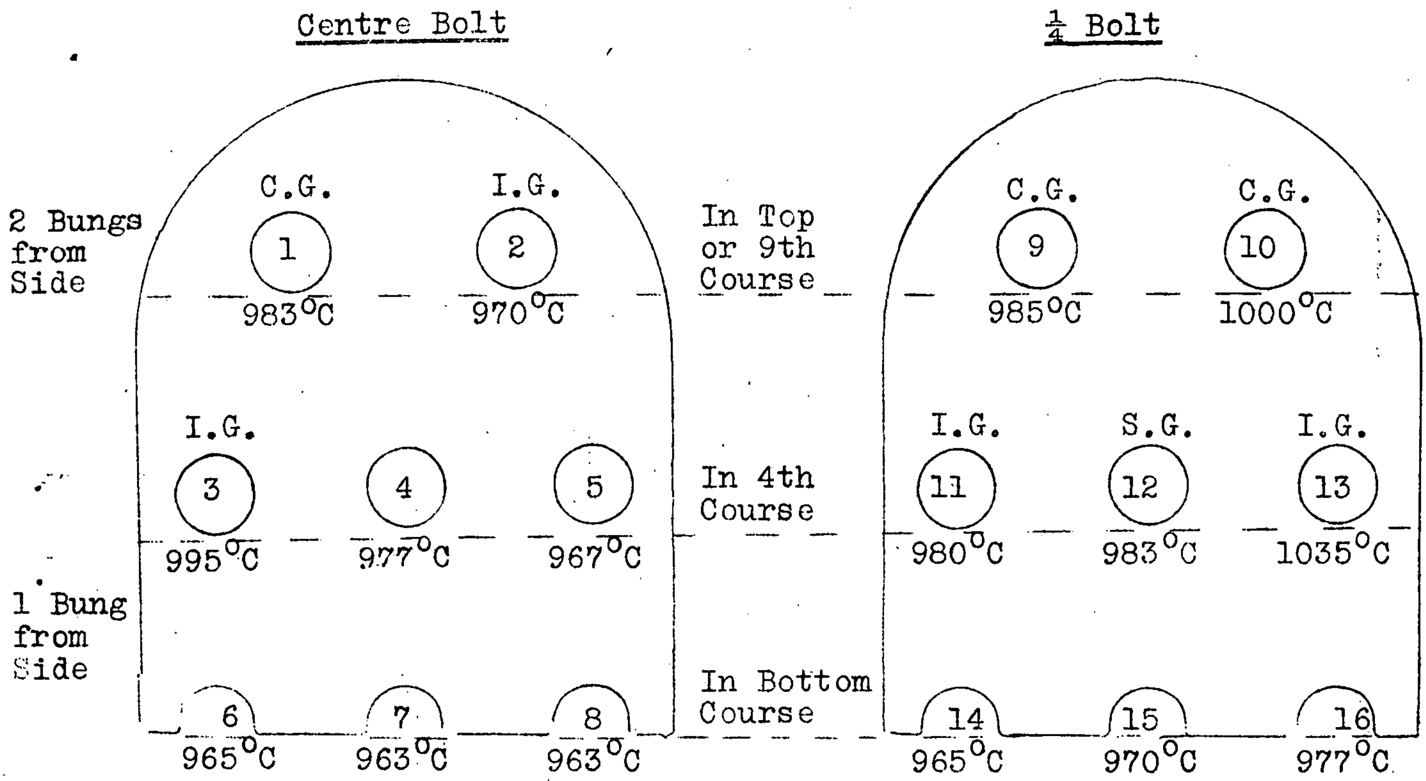
<u>The ¼ Bolt.</u> The ¼ bolt showed a wider temperature variation (57°C) than the centre bolt. The maximum temperature recorded was 1020°C in position No. 34 and the minimum of 963°C in all bottom positions (Nos. 38, 39 & 40).

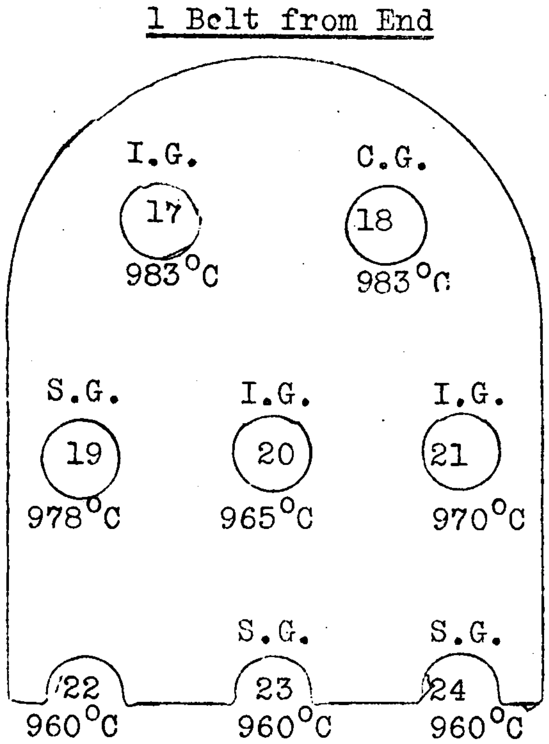
Salt glazing was complete on the top trials (Nos. 33 & 34) but only slight on the middle course (Nos. 35, 36 & 37).

<u>The Bolt 1 from the End.</u> The bolt 1 from the end was more evenly fired than the other two, there being only 21°C difference between the maximum of 981°C in the top position No. 42 and the minimum of 960°C in all bottom positions.

However, salt glazing was less apparent than in the previous bolt.

No. 1 Kiln - Coal Fired, Induced Draught.



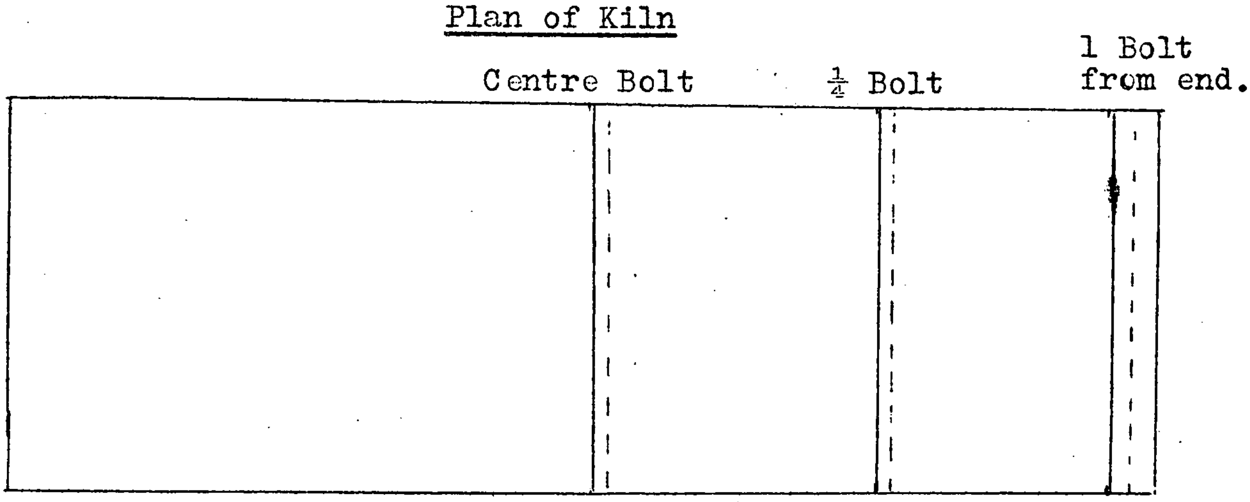


Max. Temp. Recorded		1025°C
Min.	" "	960
Variation		65°C

Key:

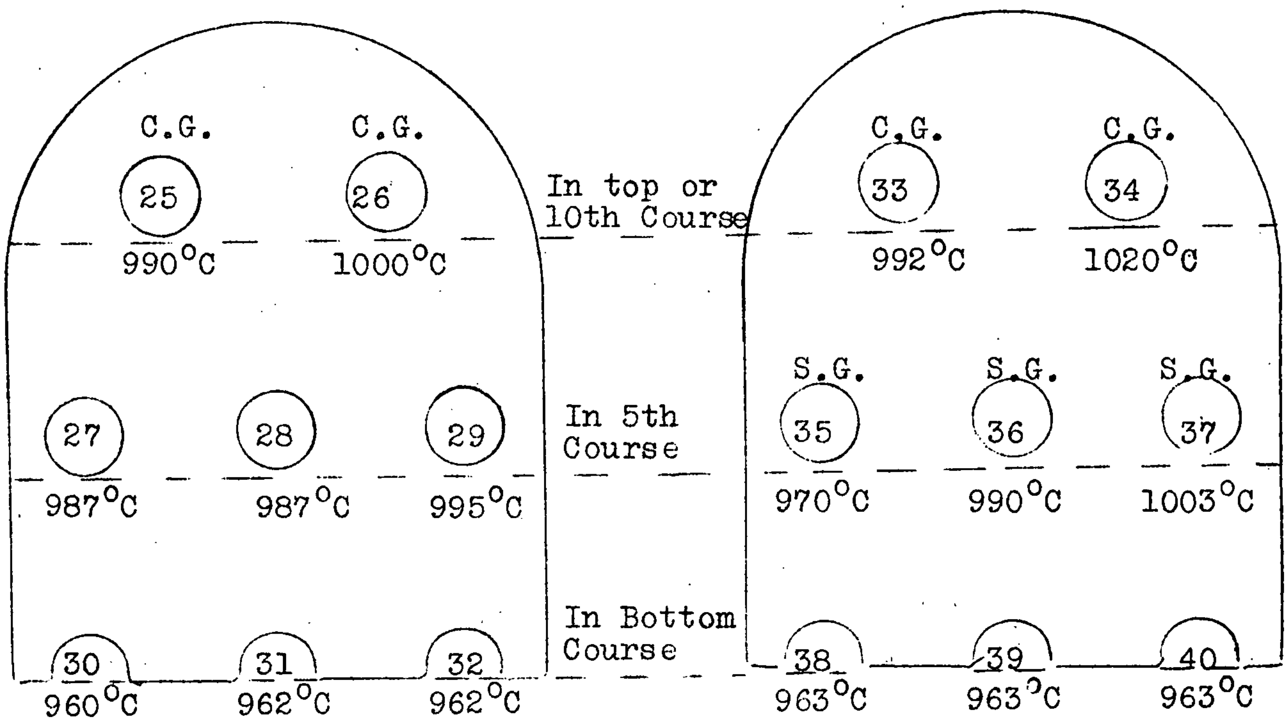
C.G. ≡ Completely Glazed
I.G. ≡ Incompletely "
S.G. ≡ Slightly "

Plan of Kiln

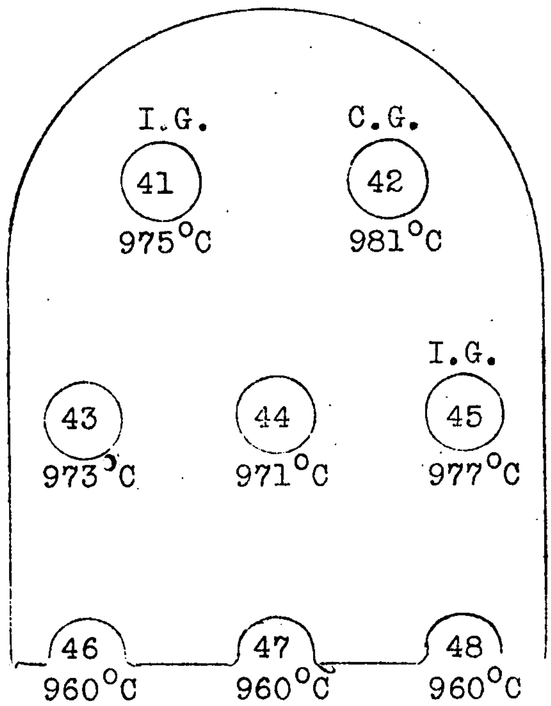


No. 5 Kiln - Oil Fired, Induced Draught.

Centre Bolt — ½ Bolt

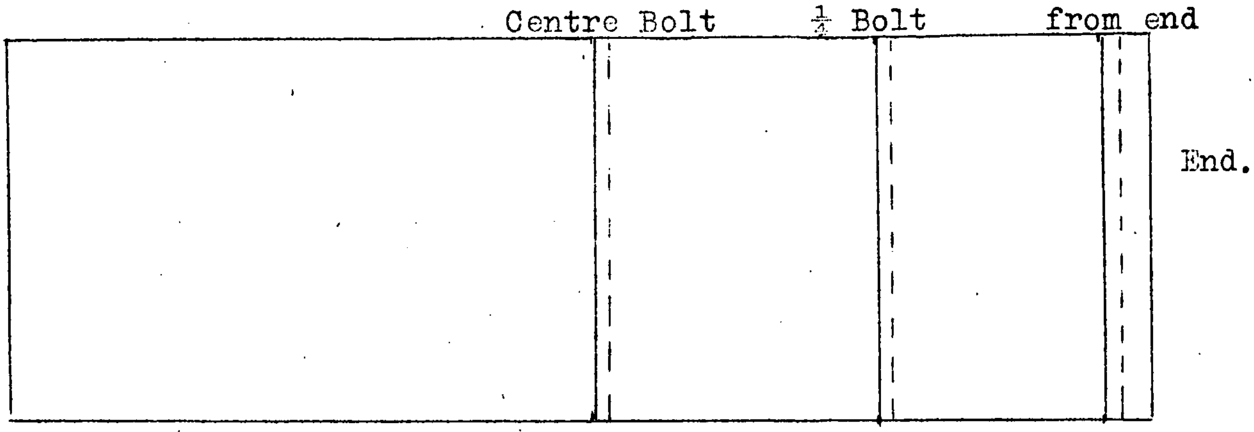


1 Bolt from End



Max. Temp. Recorded		1020°C
Min. "	"	960
Variation		60°C

Plan of Kiln



Comments.

All ware and trial specimens fired in this kiln were very clean.

There was an overall maximum temperature variation of 60°C in all the positions examined in this kiln.

This completed the survey of the first two kilns.

Kilns Nos. 8 and 4 were then examined.

No. 8 Kiln - Coal Fired, Natural Draught

The Centre Bolt. The centre bolt had been subjected to a wide temperature variation of 90°C, ranging from 980°C in the bottom course (position 56) to 1070°C in the 5th or middle course (position 51).

Glazing was complete in the top course (positions 49 & 50) but only slight in the middle course and none in the bottom.

The ¼ Bolt. The ¼ bolt did not have such a wide temperature variation (50°C) the highest being 1020°C in the middle course (position 59) and the lowest of 970°C in positions 63 and 64 in the bottom course.

Salt glazing was complete in the top course for positions 57 and 58 but incomplete or only slight half way down the setting.

The Bolt 1 from the End. The bolt 1 from the end was more evenly fired than the other two, the maximum being 995°C at the top (66) and a minimum of 965°C in all positions along the bottom giving a variation of 30°C.

Glazing was incomplete in both the top and middle courses.

Comments.

There was an overall temperature variation of 105°C in this kiln. Again the maximum temperature was not recorded at the top of the setting but at the side of the middle courses in the centre and ¼ bolts, probably due to being in the vicinity of a firebox.

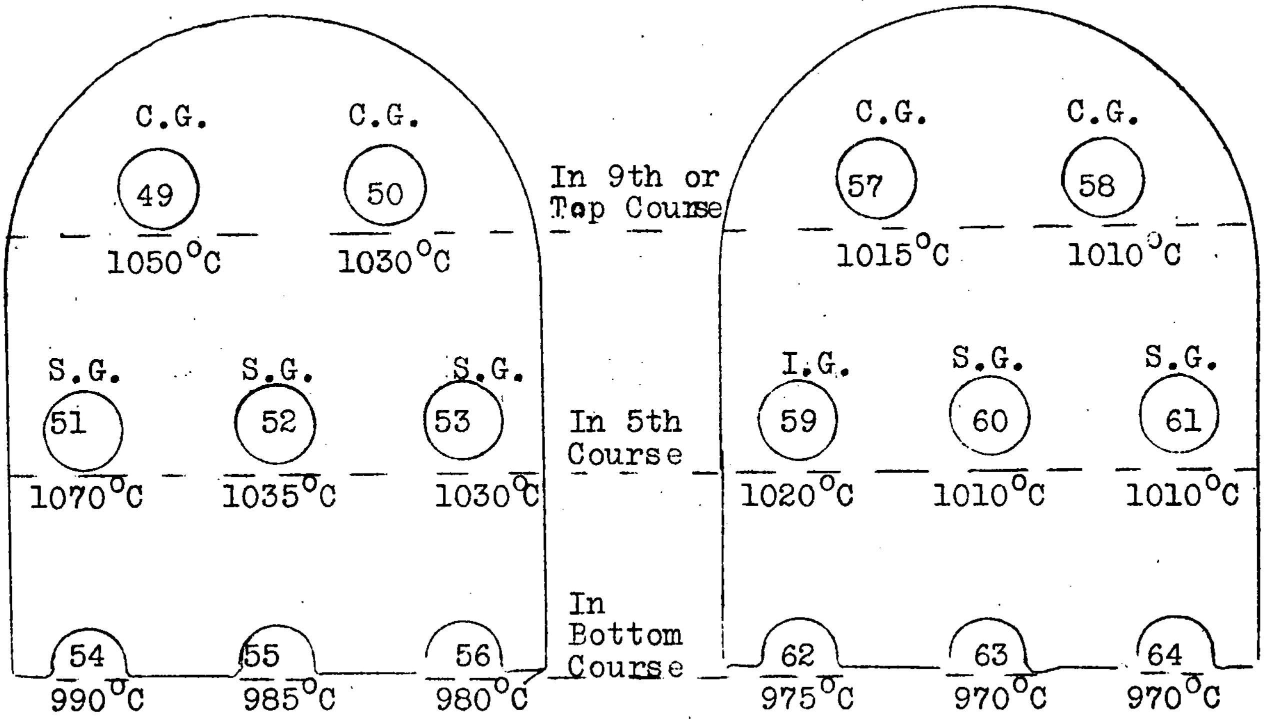
A considerable amount of fly ash was deposited on all ware in this kiln.

No. 4 Kiln - Coal Fired, Induced Draught

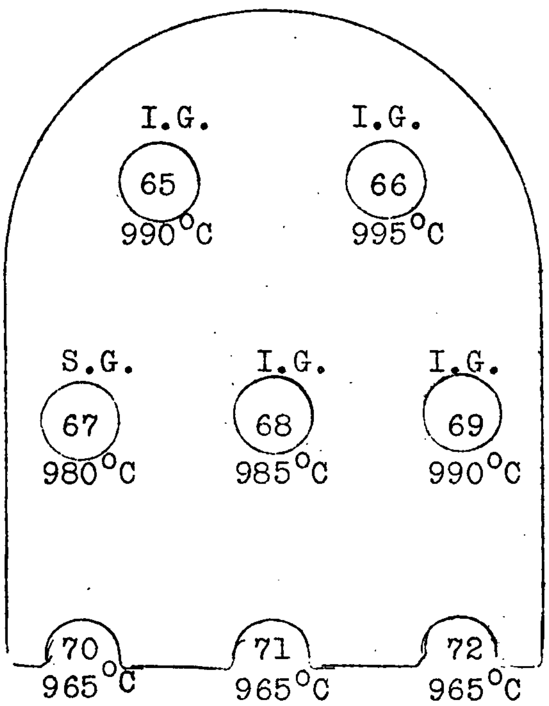
The Centre Bolt. In the centre bolt the trial specimens were evenly fired at each level but there was an overall temperature difference of 75°C throughout the bolt.

No. 8 Kiln - Coal Fired, Natural Draught

Centre Bolt $\qquad$ $\frac{1}{4}$ Bolt

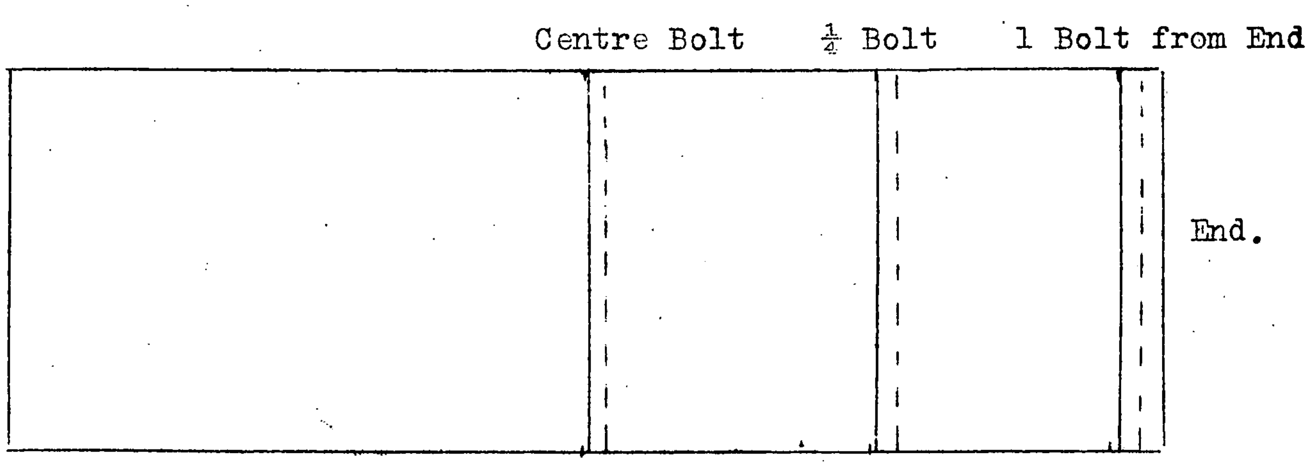


1 Bolt from End



Max. Temp. Recorded	1070°C
Min. " "	965
Variation	105°C

Plan of Kiln



Glazing was confined to the upper portion of the bolt.

The Southern ¼ Bolt. The southern ¼ bolt was somewhat similar to the centre bolt being evenly fired at each level and having a maximum temperature variation of 74°C.

Salt glazing was less pronounced than in the centre bolt.

The Bolt 1 from the Southern End. The bolt 1 from the southern end was evenly fired at each level, there being an overall temperature variation of 50°C.

Salt glazing was better than in the centre or ¼ bolt.

The Northern ¼ Bolt. The northern ¼ bolt was evenly fired at each level but had an overall temperature variation of 85°C.

Glazing was comparable with the southern ¼ bolt.

The Bolt 1 from the Northern End. The bolt 1 from the northern end was also evenly fired at each level and had an overall temperature variation of 60°C.

Salt glazing was comparable with the corresponding southern bolt.

Comments.

This kiln showed the best salt glazing effects of all four kilns examined, having the most pronounced glazing effects in the two bolts near the end of the kiln.

The kiln had an overall temperature variation of 85°C, the highest temperature recorded being in the centre and ¼ bolts.

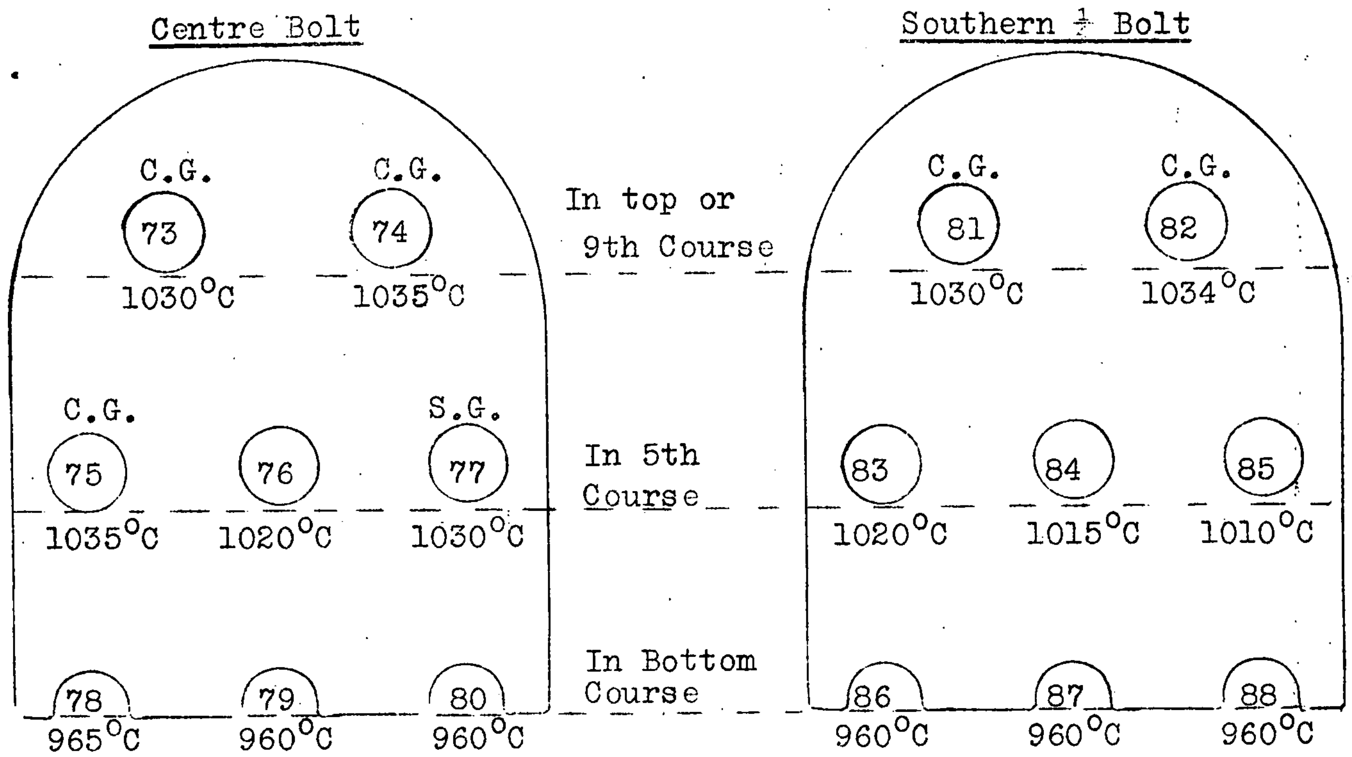
Summary of the Kiln Surveys

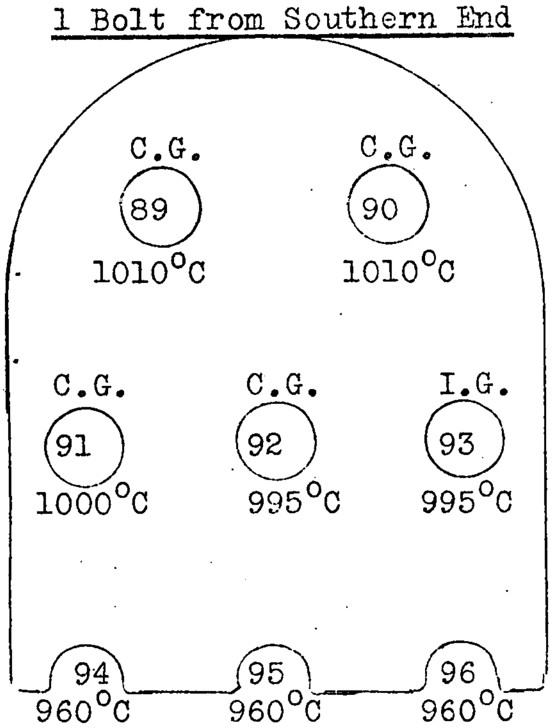
The first pair of kilns to be examined, namely Nos. 1 and 5, showed an overall temperature variation of 65°C and 60°C respectively. No. 1 kiln was coal fired and No. 5 fired by oil, both using induced draught.

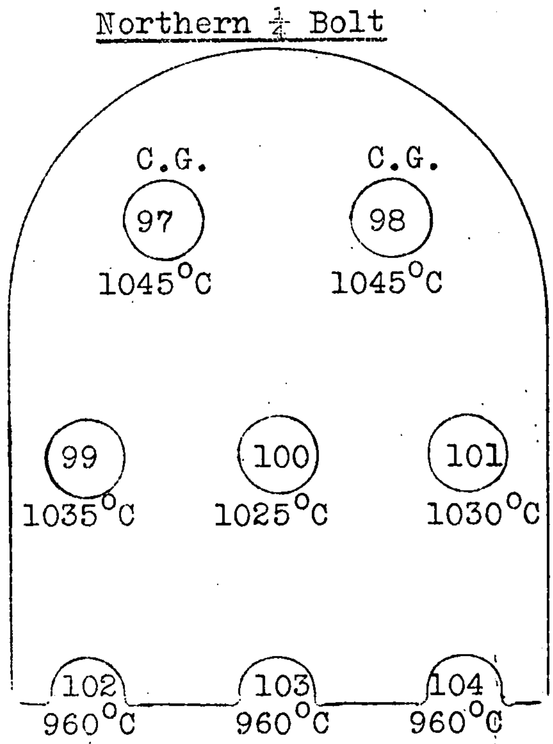
Salt glazing was limited to the upper section of both kilns, being bright and clean in the oil fired one but rough and rather dull in the coal fired one, due to fly ash.

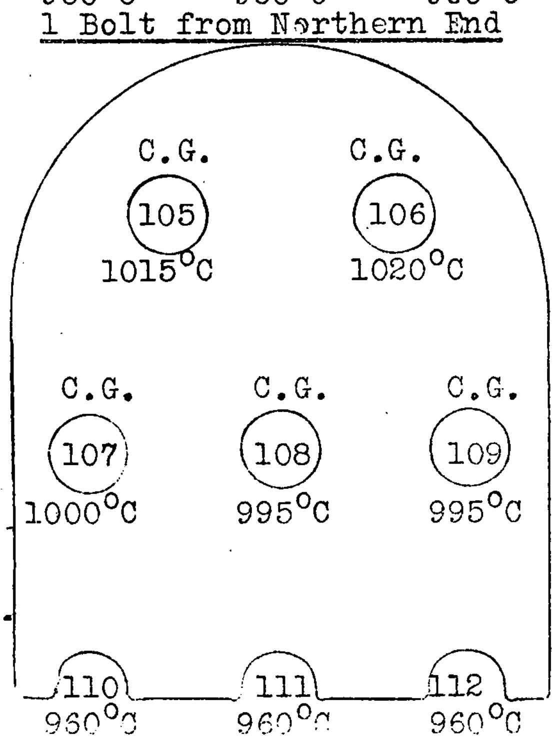
The second pair of kilns examined, namely Nos. 4 and 8, were both coal fired, No. 4 by induced draught and No. 8 by natural draught. They showed a greater overall temperature variation than the first pair, namely 85°C and 105°C respectively. In these kilns also, the salt glazing was restricted to the upper section only. Fly ash was apparent on all ware, being most noticeable on ware which was glazed.

No. 4 Kiln - Coal Fired, Induced Draught



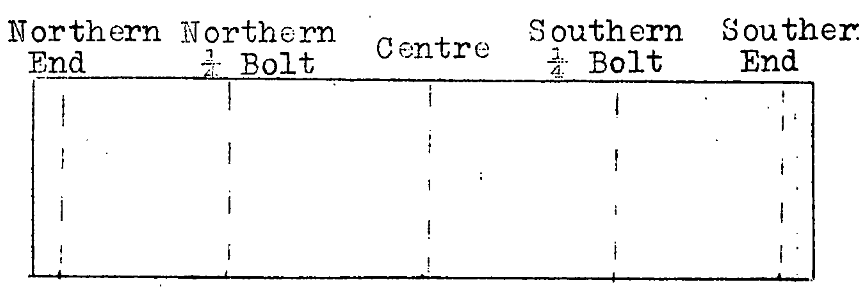






Max. Temp. Recorded	1045°C
Min. " "	960
Variation	85°C

Plan of Kiln



However the end bolt in No. 4 kiln showed effective glazing and less temperature variation than any other bolt in that kiln.

The induced draught kilns Nos. 1, 4 and 5 were fired for approximately 36 hours, whereas No. 8 kiln using natural draught had to be fired for approximately twice as long to attain a somewhat comparable heat treatment throughout the kiln and similar salt glazing effect. This procedure is standard practice.

Although in general there was an appreciable temperature variation in the upper section of each kiln, the bottom of all kilns received a more even heat treatment, the range lying between 960°C and 970°C or thereabouts.

GENERAL CONCLUSIONS AND SUGGESTIONS

As a result of a comprehensive and exhaustive investigation into the characteristic properties of available raw materials and a survey of kiln performance, the following conclusions are drawn and recommendations suggested for the manufacture of salt glazed Marseille pattern roofing tiles in South Australia:-

1. When using Cherry Gardens clay and shale only, the optimum blend of

   1 part of clay by volume
   2 parts of shale by volume

is suggested in order to give the best overall properties of the two materials instead of the 1:1 volume blend already in use.

An optimum extrusion moisture content was found to facilitate the pressing of the tiles from the de-aired extruded clot. The narrow range of approximately 1¼% was found to give the maximum dry and fired strength together with the minimum fired shrinkage.

It is significant that the optimum moisture content of the extruded 3:2 laboratory weight blend should be found to be within 0.2% of the corresponding industrial 1:1 volume blend. This indicates that the laboratory findings can be applied to industrial practice.

2. Greater benefit should accrue from incorporating Woodside fireclay in the Cherry Gardens clay/shale blend. The following blends were investigated successfully and are suggested as the starting point for industrial trials:

Cherry Gardens Clay	Cherry Gardens Shale	Woodside Fireclay	
2	2	1	by Volume
2	1	1	
1	1	1	

These blends also showed an optimum extrusion moisture content to facilitate tile pressing and gave maximum dry and fired strength together with fired shrinkage.

By replacing 1 part of Cherry Gardens shale by 1 part of fireclay in the 1:2 clay/shale blend, the 1:1:1 blend is obtained. This has the highest fireclay content of the blends examined and should give maximum benefit.

The cost of transporting the fireclay some 23 miles from Woodside should be offset by the improved quality of the ware obtained and the higher percentage of salt glazed tiles possible.

3. An improved method of raw material preparation could have an important bearing upon the consistent proportioning of the ingredients and plastic consistency of whichever blend is adopted.

It has been found in the laboratory that the most satisfactory method of blending raw materials is by mixing the dry ground ingredients prior to tempering.

On the industrial scale it is suggested that ingredients of the blend be dry ground separately, stored in bins, fed on to conveyor belts in the correct proportions by means of disc feeders and emptied on to a main conveyor belt for final dry grinding in ~~a hammer mill or~~ an impact crusher. The dry blend may then be tempered in a double shaft mixer and/or wet pan mill~~s~~ prior to passing through the de-airing pug mill.

4. With regard to the kiln surveys, some improvement in the firing technique should be possible by converting all kilns to oil firing using induced draught. The extra cost of the fuel oil should be offset by the reduced overall labour costs incurred in the handling of solid fuel and attending to kilns. More even firing of the kilns and better quality ware could result.

The incorporation of Woodside fireclay in the blend should make it possible to fire the ware to a higher temperature, 50-75°C (approx. 100° - 150°F) or even higher without the fear of overfiring or spoiling the ware, thus making it possible to soak at the peak temperature for a longer period and salt glaze further down the kiln setting.

ACKNOWLEDGEMENTS

The author wishes to thank Messrs. Geoff. R. Tilley and Lloyd R. Ridge for their assistance with the laboratory work; also the staff of Messrs. Wunderlich Ltd. for their co-operation and interest in the many phases of the investigation, particularly Mr. R. Skinner, formerly the firm's South Australian branch manager who initiated the preliminary work, Messrs. W. Baker, the present works manager and Geo. Graham the works foreman for their keen interest in and discussions on the various aspects of the work.

REFERENCES

(1) "The Blending of an Alluvial Clay from Hindmarsh with a Weathered Shale from Houghton" by H. Ellerton, S.A. Mining Review, No. 99, 1956.

(2) "South Australian Building Brick Manufacture with Special Reference to the Blending of Raw Materials and their Extrusion Characteristics" by H. Ellerton, S.A. Mining Review, No.108, 1959.