THESIS

on

## PROPERTIES OF LITHARGE AND GLYCERINE MORTARS

## Submitted to the

## OREGON STATE AGRICULTURAL COLLEGE

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MASTER OF SCIENCE

in

## CHEMICAL ENGINEERING

by

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# TABLE OF CONTENTS

Chapte	<u>r</u>	Page
I	Introduction	5
II	The Chemistry of the Compound	9
III	Physical Properties of the Mortars	16
IV	Conclusions	29
V	Bibliography	32
VI	Appendix	34

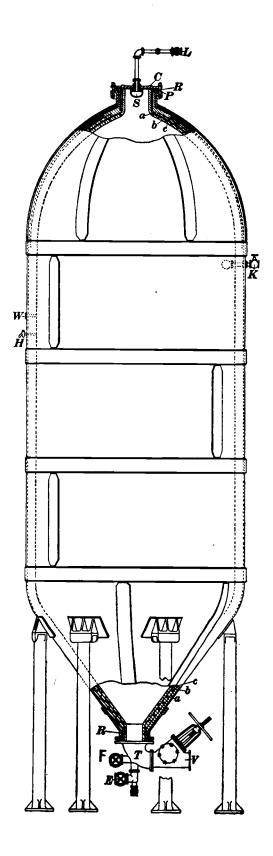
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INTRODUCTION

Litharge-glycerine mixtures have long been used for cements, for they form a workable, quick-setting mortar which sets with slight expansion into a hard, strong, chemically resistant material. Admixtures or diluents such as sand, portland cement, fire clay, quarts dust, or combinations of these are often added. The only other use known by the author to which the litharge-glycerine reaction is put is for the purification of glycerine by forming the lead glyceroxide, which is separated from the solution by filtration and the glycerine recovered by precipitating the lead.

At present, from the quantity standpoint, the most important use for these mortars is as a brick setting mortar for acid resistant linings in chemical equipment, of which the greatest single use is in the linings of sulfite pulp digestors in paper mills. Figure 1, page 7, shows the construction of a modern digestor.

"The steel shell is protected by an acid resisting brick lining; and all fittings projecting through the lining are made of acid resistant bronze or of hard lead. Before the digestor is lined it is exposed to high temperature and pressure several times, and the inside of the shell is thoroughly cleaned by means of wire brushes for the removal of oil and grease. It is then covered with a coat (a), Fig. 1, 1-1/2 to 2 inches thick, consisting of



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FIGI

cement and crushed quartz (1 part cement, 1/2 part fire clay and 1/2 part quartz), mixed with 4° Be. solution of silicate of soda and water. Against this backing is laid two courses of brick, (b) and (c), in a mixture of cement and quartz and silicate of soda. The inner course, (c) must be carefully selected, acid-resisting brick, with a resistant joint stock consisting of one part litharge, one part cement, and one-half part quartz and glycerine with a small proportion of silicate of soda.<sup>81</sup>.

At least one firm is now using as a mortar for the inner coat (c), a mixture of one part litharge, one part quartz dust and one part quartz sand and glycerine with a small proportion of sodium silicate.

Other typical uses to which the mortar has been put are packing in values, comenting tile joints, comenting pins and wall inserts in concrete, etc. It is also used as a bonding agent in the plates of lead storage batteries. It is believed that the mortar would be very satisfactory for casting resistant linings in small chemical towers by centrifugal means, for it may be handled quite plastic and sets up quickly.

# THE CHEMISTRY OF THE COMPOUND

When litharge is mixed with a large excess of glycerine, orthorhombic crystals form, which analyze according to Merwin and Oleeson<sup>3</sup> to have the composition  $C_3 H_6 OPb0$ . This salt has also been prepared by Norawski<sup>4</sup>. by treating 82 grams of lead acetate, dissolved in 250 cc. of water, with 20 grams of glycerine, heating, and adding 15 grams of potassium hydroxide. On standing, the precipitate of crystals forms.

Neville<sup>5</sup>. has investigated the reaction, and by mixing the litharge and glycerine in molar porportions of 1:1. 5:2 and 2:1 respectively, and measuring the rise in temperature he obtained the greatest rise at the proportions of 5:2. Also by carefully drying the litharge and glycerine in an oven at 106° C, and mixing them in molar preportions of 5 litharge to 2 glycerine, or richer in litharge, too much water was given off to satisfy Merwin's formula, and the amount more nearly corresponded to one and onehalf molecules of water removed from each glycerine molecule. These results were sheaked by the author by mixing on a watch glass a mortar of litharge and glycerine (both dried at 110° C.) with litharge in 80% excess of the 3:8 molar ratio, and drying in an oven at 110° C. Also mixtures were made in a weighing bottle with the glycerine in great excess and the water driven off was almost one mol per mol of litharke present.

Thus, it is quite definite that two salts may be formed in the litharge-glycerine reaction, depending mainly on the proportions of litharge and glycerine, as follows:

1. Reaction when glycerine is in excess of the 1:1 ratio of litharge to glycerine:

H HCOH HCOH	+	Ръо	 H HCO HCOPD	+	нон
HCOH			HCOH		
H			H		

2. Reaction when glycerine is in less than 3:2 ratio of litharge to glycerine:

H					Ħ			
HCOH					HCO,			
HCOH					HCO	20		
HCOH					HCO			
H					H	۲.		
	+	8	Pb0	#		Pb	+	3HOH
H					H	/		
HCOH					HCO			
HCOH					HCO,	<b>132</b>		
HCOH					HCO	rD		
H					H			

The salt formed in the sacond reaction ((C3H5O3)<sub>2</sub> Pb3) has also been prepared by reacting basic lead acetate with glycerine to which has been added a solution of lead oxide in potassium hydroxide.<sup>4</sup>.

It is evident that at ratios between 1:1 and 3:2, litharge to glycerine, both reactions will probably take place.

Neville<sup>5</sup> considers the process of setting to involve two stages: (1) a preliminary adsorption of the liquid phase by the solid; followed by (2) a chemical reaction between the adsorbed liquid and the solid. Plaster of Paris acts in a similar manner. That the process is two stage may be shown by the time-temperature curves of setting. See Fig. 2, page 13. At first practically no heat is evolved; this is the adsorption period. Then heat is rapidly evolved and the temperature rises abruptly; this is the chemical reaction period. Neville also studied the volume changes during setting, and found them to consist of a contraction during the adsorption stage, followed by a slight expansion during the chemical reaction stage.

Hydroxyl ions are apparently necessary for the reaction to go, for the dried material sets considerably slower than the undried. Small amounts of alkali or alkaline hydrolizing salts accelerate setting; larger amounts prohibit setting. Acids prevent setting. Stirring accelerates setting, probably by causing more even and thorough adsorption.

Table I, from Neville's work, shows some of the effects of stirring, acids and alkalies on the setting of equimolar litharge and glycerine.

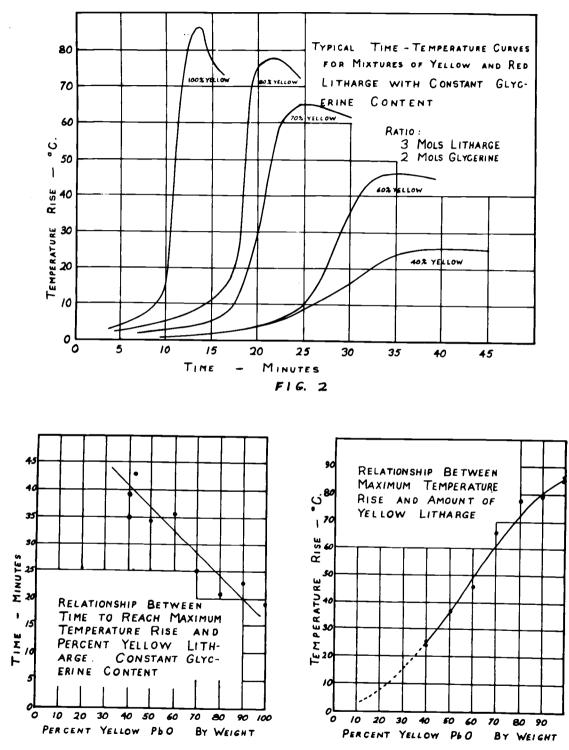


FIG. 3



#### TABLE I

Description of Materials	Stirred
	one min. three min.
1. Litharge and glycerine	4 hrs. 66 min.
2. Both dried at 106° C.	l day 3 hrs.
3. 10% water added to glyserine	30 min. 20 min.
4. 10% dil HCl to glycerine	does not set
5. 10% water added to litharge	20 min. 13 min.
6. 10% dil. KOH to glycerine	13 min.

There are two kinds of litharge, yellow orthorhombic and red tetragonal. The international critical tables give some of their properties as follows:<sup>6</sup>.

Formula	Name	Mol.Wt.	Crystal	Density	M.P.
<b>P</b> b <b>0</b>	litharge	223,20	tetragonal	9,53	888
PDO	massicotite	223.20	orthorhombic	8.0	

Commercial litharge is usually buff colored, though for some things the red form is often used. The red tetragonal form is produced by crystallization from the molten stage. If it is heated at a dull red heat it changes to the canary yellow powder. The buff litharge is considered by Neville to be amorphous, but the buff litharge the author used, appears to be a mixture of the red and yellow crystalline forms. At least both forms are present, as may be seen by streaking it across a paper with a spatula to break up the red and yellow agglomerates. The particles were all so fine that an estimation of the composition could not be made by a microscopical examination.

The yellow litharge used by Reynolds and the author was "Baker's C.P. Yellow Litharge." In appearance it was "buff," though much yellower than ordinary commercial litharge, and is believed to be a mixture of yellow and red crystalline forms, though the red present probably did not exceed five per cent as estimated by streaking with a spatula. The red litharge used was "Baker's C.P. Red Litharge," and was estimated to contain less than five per cent yellow.

The relationship between time of setting and temperature rise for different mixtures of red and yellow litharge was investigated by Reynolds,<sup>7</sup> and the results of his work are shown graphically by Fig. 2, 3, and 4, page 13. It will be seen that the time of setting increases directly as the proportion of red to yellow is increased.

# PHYSICAL PROPERTIES OF THE MORTARS

## REVIEW OF PAST INVESTIGATIONS.

Litharge-glycerine mortars are more plastic or "fat" than most other mortars and are therefore harder to mould bubble free. The consistency is changed considerably by a small change in the glycerine content.

Gleeson's work<sup>3</sup> on factors affecting yield and strength is all that is available in the literature. The practical range of plasticity for mortar use is indicated in his curve, which shows the effect of the glycerine content upon the yield in 1:1:1 mixes of litharge, cement and sand; Fig. 5, page 18.

The results of his volume studies on litharge mortars are shown by his curves given in Fig. 6. He considers these curved to be of sufficient accuracy for quantity estimations and cost calculations.

The relation between the composition of the glycerine solution (per cent glycerine) and the strength of the resulting mortar is shown in Fig. 7. It will be seen that both the tensile and compressive strengths increase with an increase in the glycerine-litharge ratio.

The relation between the richness of mix and the strengths are shown in Fig. 8. The leaner mixes are stronger than the very rich ones--even stronger than the straight litharge-glycerine mortar of the same consistency.

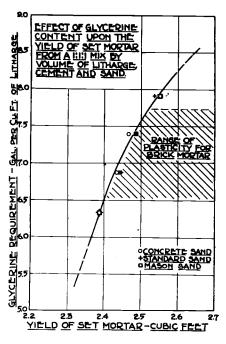


FIG. 5

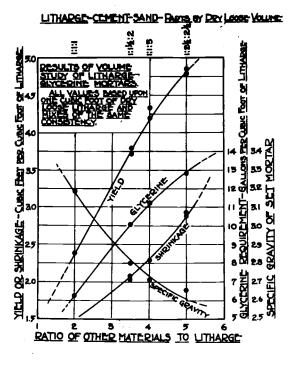
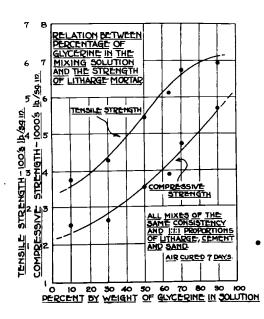
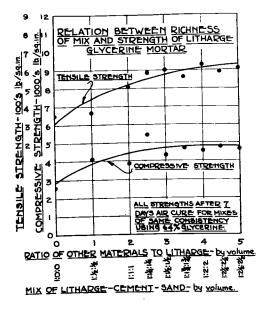


FIG. 6





FIG, 7

F16. 8

Gleeson believes this increase in strength of the leaner mixtures may be due to lack of "seed" in the richer mixes, and hence much of the litharge may act as centers of crystal growth, and as such are not contacted by the glycerine and therefore do not act as bonding substance. The author believes this to be somewhat true, but also is convinced that the strength of the mortar is dependent upon which litharge-glycerine compound is formed, C3H603Pb or  $(C_3H_5O_3)_pPb_3$ . This view is further substantiated by the fact that if the glycerine-litharge ratio is increased, either by diluting the mortar with solid admixtures requiring more glycerine to wet them, or by using more concentrated glycerine-water solutions for mixing, the strength is increased. Hence it appears that the strongest crystal, and probably the most desirable for mortar use, is formed when litharge and glycerine react in equimolar proportions.

The effect of age on strength was also investigated by Gleeson, and he found that, over a period of twentyeight days, the tensile strength increased to a maximum in about seven days and then fell off slightly, whereas the compressive strength increased for about the same period and then remained constant.

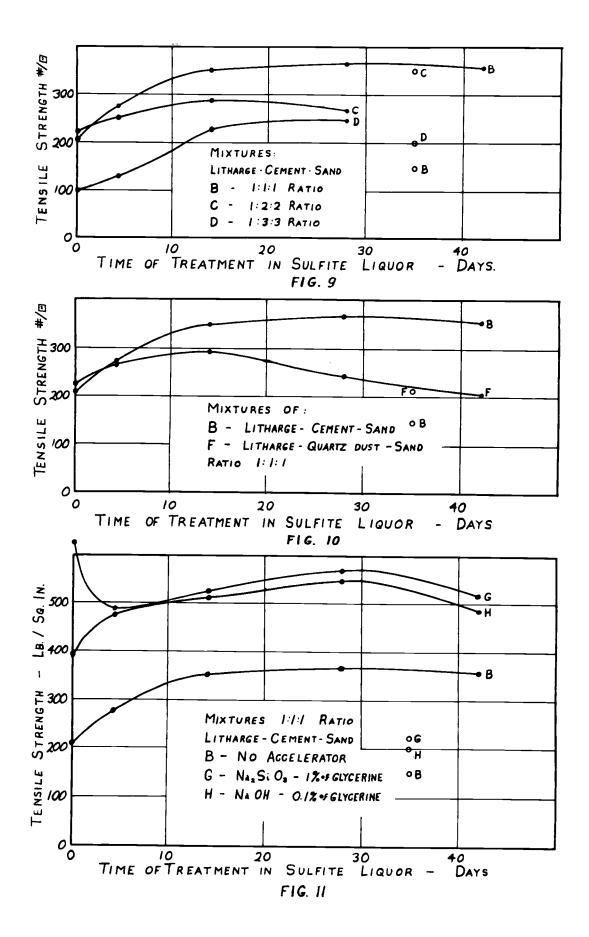
#### AUTHOR'S INVESTIGATIONS

The corrosion resistance of the mortars to hot sulfite liquor was investigated by the author by making A. S.

T.M. standard tension briquettes, scaling them in sulfite liquor in ordinary glass fruit jars, and heating them with direct steam in the autoclave shown in figure 15. Each week the sulfite liquor was replaced with fresh. It was also found necessary to replace the jars each week, for after about five days use, they began to develop little cracks which quickly spread, completely breaking the jar in the next day or so. Almost no jars broke in less than five days. To obtain comparative results the briquettes were dried three days in air before being tested. The tests were made with the Tinus Olsen testing machine shown in Fig. 16. The litharge used was redder than that used by Gleeson and the mortar set slow, so all were allowed to cure a month in air before the sulfite liquor treatment was begun.

The results of the experiment are shown graphically by Fig. 9, 10 and 11, page 21.

Fig. 9 shows the effect of the treatment upon 1:1:1; 1:2:2 and 1:3:5 mixtures of litharge, cement and sand made to the same consistency. The lettered points in each Fig. (indicated by circles) are the average strengths of specimens wh ch did not receive treatment, but were stored in air for the time indicated after the treatment of the other specimens was begun. It will be noted that the strengths increased with treatment. This was not expected in any case but was most probably due to the cement content which hydrolized on exposure to the liquer, for the glycerine



used in mixing the batches was of C.P. quality and contained very little water. The specimens of the lil:1 mix appeared in good condition even at the end of the treatment, but the briquettes of the leaner mixes scaled off badly on the surface. The extent of this scaling may be seen from the photographs taken after four days treatment, Fig. 13 and 14, page 23. The effective cross sectional areas of the speciemns were reduced about twenty per cent.

Fig. 10 shows the relative effect of the treatment upon lil:1 mixes of litharge, cement and sand, and litharge, quartz dust and quartz sand. It appears that mixtures containing cement withstand sulfite liquor and heat better than those containing quartz dust and no cement, but that the quartz dust mixtures remain stronger when only exposed to air. This may be due to the greater fineness of the cement particles than the quartz flour, resulting in a closer, more impervious mortar, and also to the setting of the cement.

Fig. 11 shows the effect of the treatment on mortars containing sodium silicate and sodium hydroxide. It will be noticed that these compounds have greatly increased the strength of the mortars. The dip in the curve G is unexplained; it appears to be too large for experimental error. The specimens appeared in good condition after the treatment, as may be seen from the photographs, Fig. 12.



FIG. 12



FIG. 13



FIG. 14

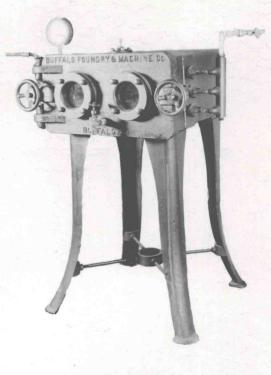


FIG. 15

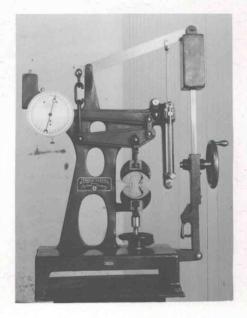


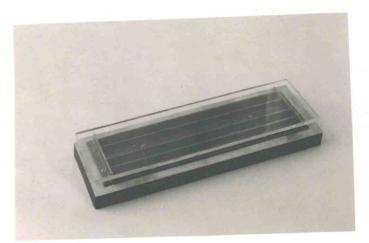
FIG. 16

If the experiment were being re-ran a check would be made using water instead of sulfite liquor to determine the effect of heat and water alone on the strengths, and also a dry run would be made to test the effect of heat only.

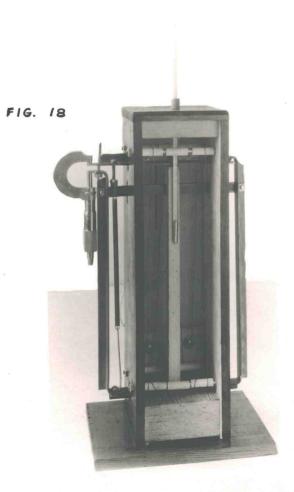
From an examination of the briquettes and consideration of the strength data obtained it appears that the corrosion effect is almost entirely on the surface and that where the mortar is subjected to abrasion, even though mild, the corrosive action would be much more important than where there is little or no abrasion.

The thermal coefficient of expansion is a very important property of such a mortar, in that it should be as nearly as possible the same as the bricks used in order to avoid being cracked away when the wall is heated. Therefore the coefficient of expansion was determined for 1:1:1 mixtures of litharge, cement and quartz sand, and litharge, quartz dust and quartz sand. It was also determined for a neat mortar of litharge and glycerine in 3:2 molar ratio.

To do this specimens were made  $10^{n} \ge 1/2^{n} \ge 1/2^{n}$  in moulds shown in Fig. 17, page 26. A thermocouple was cast in the center of each specimen. The coefficient of expansion was obtained by heating the specimen by radiation from an electric heating element within a wooden box and measuring the expansion, as multiplied by a lever, with an ordinary screw micrometer. The construction of the apparatus is clearly shown by its photograph, Fig. 18. The temperature







rise was measured by means of a potentiometer and the thermocouples in the specimen. The experimental error was probably within five per cent. Greater accuracy was not sought because of the variation in quarts sands. See the appendix for experimental data obtained and method of calculation.

The values obtained are as follows:

THERMAL COEFFICIENT OF EXPANSION

 $20 - 100^{\circ}$  C.

Litharge, quartz sand, quartz dust 1:1:1  $28.0 \times 10^{-6}$ Litharge, quartz sand, cement 1:1:1  $18.8 \times 10^{-6}$ Litharge, glycerine 3:2 mols  $27.9 \times 10^{-6}$ 

Table 2 gives the coefficient of expansion for several types of brick.

## TABLE II

Thermal	Expansion	of Brick <sup>6</sup> *
	25 - 100 <sup>*</sup>	C. #
Alumina	* • • • •	· · · · .7.2 - 8.0
Alundun		• • • • 7.1 - 8.5
Bauxite	• • • • •	
Carborundum .	* * * * *	6.58
Fire clay bric	k	5.00 - 11.0
Magnisite brie	k	10.5 - 12.5
Silica brick	# x 10	• • • • 25.0 - 31.0 6

Since it was thought that the coefficient of expansion might vary with the proportion of red and yellow litharge used in a mortar this was tried using l:l:l mixtures of litharge, quartz dust and quartz sand, and varying the proportions of red to yellow litharge. The glycerine content was held constant. No noticeable variation was obtained.

CONCLUSIONS

I

Two different chemical compounds may be formed depending on the litharge-glycerine ratio. The compound  $C_3H_6O_3Pb$ is formed when the glycerine-litharge ratio is 1:1 or more; whereas the compound  $(C_3H_5O_3)_2Pb_3$  is formed when the glycerine-litharge ratio is 2:3 or less. In between both will be formed. It appears that the crystal  $C_3H_6O_3Pb$  is the most desirable from the mortar strength standpoint. This litharge-glycerine ratio in mortars, comparable to the water-cement ratio in concrete, deserves more study.

### II

Thorough mixing or stirring would probably result in a much stronger mortar. This should be investigated, The strength of plaster of paris is increased by stirring.

#### III

Heat and moisture accelerate setting and produce a stronger mortar. The effect of heat alone and moisture alone should be studied, expecially of the mortars containing cement.

IV

Cement gives a stronger mixture if exposed to heat and moisture than does quartz dust as an admixture. This is most probably due to the setting of the cement.

V

Basic accelerators increase the strength of the final set mortar, expecially on exposure to sulfite liquor. These should be investigated to find the optimum concentrations, also whether they increase the final strengths of quartz dust mortars as much as coment mortars

### VI

Leaner mixtures than of the 1:1:1 ratio would probably be satisfactory for backing where abrasion is not present. All exposed mortar, though, should be as rich as 1:1:1, for the weaker mortars scale off, and hence, though strong, will not stand abrasion. This applies to acid resistant linings.

#### VII

Samples of the mortar tended to reach a maximum strength on ageing and then to decrease. In some cases this decrease was very considerable. Just why this happens or what might be done to remedy it is not known. It certainly warrants an investigation.

## VIII

Quartz dust gives a better coefficient of expansion than coment as an admixture in the mortar for use in setting silica brick, which is the usual brick used in acid resistant linings.

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## APPENDIX

This appendix contains the data obtained by the author, and the calculated results of his studies on:

1. The chemical reaction

- 2. The corrosion resistance to sulfite liquor.
- 3. The coefficient of thermal expansion.

Gleeson's analysis<sup>3.</sup> of the crystals formed by litharge, and glycerine in an excess of glycerine, is also included.

# LITHARGE - GLYCERINE REACTION

## Water Liberated.

	1	2		4
Watch glass or bottle	3.9705	5.8390	14,3985	14.3990
Glass and glycerine	6,1440	7.7890	28 <b>.3670</b>	32,4160
Glycerine	2.1735	1.9500	13.9685	18.0170
Litharge	9.50	8.52	10.00	10.00
Méls litharge/mols glycerine	1,80	1.80		
Total weight	15.6430	16.3065	23.9695	28.0155
Dry weight	15,0890	15.8700	23,2130	27.2745
Water driven off	0.5540	0,4365	0.7565	00.7410
Mols water/mol glyc.	1.30	1.35		
Mols water/mol lithar	3 <b>8</b>		0.94	0.92
Theoretical mol ratio	1.50	1,50	1.00	1.00

ANALYSIS OF CRYSTALS

Excess Glycerine By Gleeson<sup>3</sup>

															1	_2
РЪ	-	•	-	-	**	-	**	**	- 1884	-	-	*	**	•	69.40	69.36
C	-	•	-	•	-	<b>145</b>	-	**			-	-	-	-	11.82	11.96
H		-	-	-	-	**	-	-	*	**	-	-	-	-	1.39	1.63
0 (	( 63	7 0	111	ffe	9r(	9n(	30	)	•	-	-	-	-	+_	17.39	17.05
															100.00	100.00

## MATERIAL DENSITIES

## (Dry loose volumes.)

	Specific Gravity	Density 1b./cu. ft.
Sand	1.52	94.5
Quartz sand	1.32	82.2
Quartz dust	1.39	76.3
Cement	1.39	86,7
Litharge	3,45	208,0

## COMPOSITION OF TENSION BRIQUETTES

(Materials indicated by dry loose volumes; glycerine in 1b. per 1b. litharge.)

	Litharge	Cement	Sand	<u>Glycerine</u>	Mols glycerine Mols litharge
В	1	1	1	0.440	1.07
C	1	2	2	0.683	1.66
D	1	3	3	0 <b>*855</b>	2.08
F	1	11	1*	0.410	1,00
G	1	1	1	0,427*	1.04
H	1	1	1	0.4104	1.00

1 Quartz flour used in place of cement.

- Quartz sand used.
- <sup>5</sup> 1% sodium silicate used as acceperator (glycerine basis)
- 0.1% sodium hydroxide used as accelerator (glycerine

basis.)

## TENSILE STRENGTH

## OF TENSION BRIQUETTES

(Underlined values are averages as used in the curves.) (1 After 35 days in air after treatment was started all specimens were cured in air 30 days before treatment.) TIME OF EXPOSURE TO SULFITE LIQUOR

		4	14	28	42	35
B	<b>215</b> 225	330 225	275 450	390 370	225 450 <b>425</b>	155 145 135
	220	280	360	380	375 <u>370</u>	<b>155</b> 150
C	225 235	235 265	<b>305</b> 270	260 290 280		<b>400</b> <b>30</b> 0
	230	250	290	230 265		350
D	100 95	150 110	220 260	250 235 260 250		190 205 210
	100	130	240	250		210 205
F	2 <b>45</b> 225	270 <b>30</b> 5	270 310	235 250	230 225 200 160	240 235 180
	235	285	<u>290</u>	240	205	220
Ģ	655 530 715	535 445	610 440	550 610	550 475 440 550	2 <b>40</b> 200
	635	490	525	580	510	220
H	63 <b>5</b> 320 270 355	525 450	<b>49</b> 0 5 <b>40</b>	525 575	460 450 550 460	220 180
	<u>395</u>	<u>485</u>	<u>515</u>	550	480	200

## THERMAL COEFFICIENT OF EXPANSION

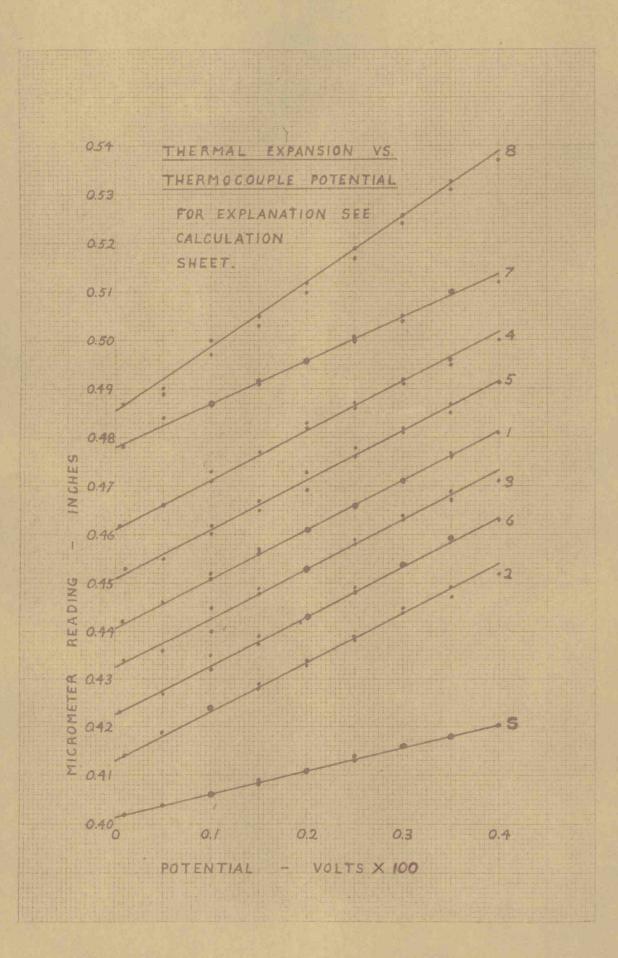
#### Data.

- Samples No. 1 to 6 are litharge, quartz dust and quartz sand (1:1:1) with the per cent red and yellow litharge varied as indicated. - Glycerine-litharge molar ratio, 1:1.
- Sample No. 7; cement used instead of quartz dust. Litharge 100% yellow.
- Sample No. 8; neat mortar of litharge and glycerine in 3:2 molar ratio.

Sample No. s is a bar of steel used as a check bar.

8. <b>N.</b> F.				Mic	romete	r Read	ings		
X100	1 100%Y	2 8 <b>0%Y</b>	3 60%Y	4	5	6 100%R	7	8	8
(See	0,015	0,035	0,020	0.910	0.020	0.010	0,020	0.020	0.010
note)	0.442	0.414	0.434	0.462	0.453	0.423	0.478	0,487	0.402
.0500	0.446	0.419	0.436	0,466	0.455	0,427	0.484	0,490	0.404
0.1000	0,451	0.424	0.440	0.471	0.462	0.432	0.487	0,500	0.406
.1500	0.456	0,429	0.448	0.477	0.467	0.437	0.492	0.505	0.408
<b>.20</b> 00	0.461	0.434	0.453	0.482	0.473	0.443	0.496	0.512	0.411
.2500	0.466	0.439	0,459	0.487	0.478	0.448	0,501	0,519	0.414
. <b>30</b> 00	0.471	0.445	0.464	0.492	0.482	0.454	0.505	0.525	0,416
.3500	0.477	0,449	0.469	0.496	0.487	0,459	0.510	0.533	0.418
.4000	0.481	0.452	0.471	0.500	0.491	0.463	0.512	0.537	0,420
.3500	0.476	0.447	0,467	0.495	0.485	0.459	0.510	0.531	0,418
.3000	0.471	0.444	0.463	0.491	0.481	0.454	0.504	0.524	0.416
.2500	0.466	0.438	0.458	0.486	0.476	0.449	0.500	0.517	0.413
• <b>200</b> 0	0,461	0,438	0.453	0.483	0,469	0.443	0.496	0.510	0,411
.1500	0.457	0.428	0.449	0.477	0.466	0.439	0.491	0.503	0.409
.1000	0,452	0.424	0.445	0.473	0.460	0.435	0.487	0.497	0.406

# Note: The first and second lines of data are the initial E.M.F. and micrometer readings respectively.



## THERMAL COEFFICIENT EXPANSION

## Calculations.

The coefficients of expansion were calculated by first plotting the values of expansion as measured vs. the thermocouple potential, and drawing the best straight line through them (See curve sheet, page 40), then calculating the mean coefficient from the slope of the survey as indicated in each case below.

Length of specimen - - -• - - 10 inches Multiplication of expansion by lever - 2.40 x Thermocouple calibration - - - - - - 0.01 volt = 200° Coefficient of expansions. No. 1 - 6Litharge, quartz dust, quartz sand 1:1:1 Olycerine-litharge ratio 1:1 (mols)  $(0.041)(10 \times 2.40 \times 0.400 \times 0.200) = 22.0 \times 10^{-6}$ No. 7 Litharge, cement, quartz sand 1:1:1 Glycerine-litharge ratio 1:1 (mols)  $(0.036)/(10 \times 2.40 \times 0.400 \times 0.300) = 18.8 \times 10$ No. 8 Litharge and glycerine 3:2 (mols)  $(0.052)/(10 \times 2.40 \times 0.400 \times 0.200) = 27.9 \times 10^{-6}$ No. s Steel bar  $(0.019)/(10 \times 2.40 \times 0.400 \times 0.200) = 103 \times 10^{-6}$