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## A PROCESS FOR MAKING REFRACTORY INSULATING BRICK

BY

# ALBERT LLOYD TETLEY

A

#### THESIS

submitted to the faculty of the

SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI

in partial fulfillment of the work required for the

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52403

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## A Process For Making Refractory Insulating Brick

#### INTRODUCTION

In recent years the tendency in kiln and furnace design has been to decrease the wall thickness and to use insulation. The decrease in thickness reduces the heat stored in the walls, which is of considerable importance in periodic types of kilns. Insulation decreases the heat loss through the walls in both periodic and continuous operations. Contrary to previous beliefs, properly constructed thin walls in most cases have as long a life as thick ones, and the initial cost is less.

The use of thin walls creates a demand for a refractory somewhat different from the usual type. This may be termed a refractory insulator. It should have properties intermediate between a refractory and an insulator. Such a material may be used in two ways: first as an insulator behind a thin layer of refractory where the temperature is too high for the usual insulator, and second as a refractory exposed to the furnace gases.<sup>1</sup>

High temperature insulation, although developed in the last 20 years, has played an important part in the stepping up on industrial temperatures.

Insulating refractories have three purposes in kiln and furnace construction: first, they equalize the heat more evenly over the furnace; second, they reduce heat losses by keeping the outside wall to a minimum temperature, and third, they make a lighter structure occupying less space.

<sup>1</sup> Refractories, F. H. Norton, 1st. ed., p421

Heat is transmitted from one place to another by three methods:-radiation, convection and c nduction, which are all familiar. In the loss of heat from a surface of a furnace there is first the transmission through the wall to the outer surface where it is carried away by convection. The quantity of heat transmitted through the wall varies directly with the area of the wall, the temperature difference betweent he hot and cold surfaces, the thermal conductivity of the materials from which the wall is made, and inversely with the thickness of the wall.

The rate of emission of heat from the cooler face of the wall depends on: the temperature of the wall, the difference in temperature of the wall and the surrounding air, the rate of circulation of the air, and the nature of the surfaces of the wall.

Radiation losses rise rapidly with rise in temperature and it is consequently increasingly important to reduce heat losses at higher temperatures.

For some time there was a general belief that since air is a poor conductor of heat, air spaces built into the walls of boiler's and furnaces would prevent or reduce heat losses through the walls. Ray and Kreisinger<sup>2</sup> of the United States Eureau of Mines ran a series of tests on Fir spaces used as an insulator and showed that at high temperatures this belief does not hold true, because of the fact that hot surfaces of air space radiate heat so rapidly that a greater amount is lost across the space by radiation than would be conducted were the Fir space filled with material of relatively high conductivity such as fire brick. The reason for

Loss of Heat From Furnace Walls, U. S. Bur. Mines Bull. No. 8

this is easily seen when we realize that the quantity of heat passing through a portion of solid wall by conduction depends upon the difference between the two plances limiting the portion of the wall, while the quantity of heat that passes across the air space in the wall depends on the difference between the fourth powers of the absolute temperatures of the surfaces enclosing the air space.

A good refractory material is essentially strong and dense and is consequently highly conductive. In order to prevent excessive heat losses, a furnace wall must either be built up of great thickness, which is costly, gives an inelastic wall and absorbs a large amount of heat not used in productive work, or be built to include as a component of the wall, a layer of some material of lower thermal conductivity, called the insulator.

The rate of heat flow through a wall depends upon the resistance of the component materials to the heat waves. A material having low apparent density is considered to be a good insulator. Most such materials contain small voids or cells. If the cells are large enough to be seen with the naked eye, they are large enough to be instruments of heat transfer.

While fuel saving is the principal advantage of insulation, there are others such as the imporvement in the quality of treated products, which are brought about by a more uniform and more accurate temperature control.<sup>3</sup>

<sup>3</sup> High Temperature Insulation, Celite Products Co. Los Angles, California.

Cellularity of insulating brick may be produced by one of the following methods:

1. Burning out of a combustible.

2. Aeration of the powdered body by mechanical methods.

3. Bloating clay slips by chemically evolved gases.

4. Use of a stabalized foam in the slip.

Refractory insulators for industrial use should have crushing strength values which, in proportion to their weight, approximate those of fire brick. The PCE should be rather high, around **cone** 30, to withstand furnace temperatures. Insulating brick should show good resitance to load deformation under loads proportional to the bulk density of the brick.<sup>4</sup>

The characteristics desired in an insulating brick listed in the order of their importance are:<sup>5</sup>

1. Uniformity of pore structure.

2. Minimum shrinkage.

3. Spalling resistance.

4. Physical strength.

5. Insulating values.

6. Thermal capacity.

The most common method used today in the manufacture of insulating brick is to mix organic material such as sawdust or cork with the clay and form roughly into shape. Upon firing, the organic 4 Physical Properites of Some Insulating Brick. W.C. Rueckel, Jour. Am. Cer. Soc. vol. 18, pp 18-22. (1935) 5 Insulating refractories, W.M. Hepburn, Jour. Am. Cer. Soc., Vol. 18, pp 13-17 (1935)

material is burned out leaving pores in the finished brick. In this method of manufacture the fired brick must always be ground down to the desired size. The use of such material as sawdust, cork, and coal usually lowers the refractories of a brick because of the fluxing action of the ash which is left after combustion. However, substitutes such as monthalene, which volatilizes in the arving process and is reclaimed, having been used and leave no ash to act as a flux.

#### OBJECT

The object of this study was to investigate the fundamental principles of producing an insulating brick by making the pore structure of air bubbles entrapped in a cley slip. Since a clay water mixture or any material containing entrapped air will loose this air when placed under vacuum, it is though that placing the clay flips under a pressure would cluse a considerable pore structure in the slip when the pressure is released.

In the common method of making insulating orick, the fired brick must be ground to size asthey cannot be molaca to a definite size and shape. This operation is an expensive one and if it could be avoided would aid in the production of insulating brick. By making the pore structure as mentioned above, it is thought that the bricks may be molded into definite shapes and fired, thus eliminating the grinding operation.

#### PROCEDURE

Proposed Procedure:

A six-member triaxial series, using refractory clays, is to be tested. The clays are tobe ground to pass a 100-mesh sieve and made into slips of such a consistency that they can be handled easily. The slips are stabalized with organic deflocculators and binders. The organic material is used because it will burn out on firing and will not give a fluxing action. The organic binders also give more strength to the molded pieces in the green state.

The cley slips are to be pleced in an air-tight tank and subjected to a pressure and agitated to insure intimate mixture of the slip and air. The slip is then released into molds and allowed to dry.

It is known that carbon dioxide gas will dissolve in appreciable quantities in water when placed under pressure, and the water will give up the carbon dioxide when the pressure is released. Therefore it is proposed to put the clay slips under pressure using carbon dioxide and treat the same as in the case of the air pressure. The release of the carbon dioxide gas when the pressure is released is to form a porous structure.

## Materials and Apparatus:

The clays used are Forbes' Flint Clay, Fulton Plastic Clay, and Georgia Kaolin, which are mined in Phelps County, Missouri. The clays are rather abundant in Missouri and easily obtained. It is refractory and has little plasticity. The Fulton Plastic clay is from the coal-bearing strata of north Missouri. It is quite abundant in that area and is a semiplastic refractory fire clay.

The Georgia Kaolin is a primary white clay. It is a lightweight material and somewhat refractory with moderate to low plasticity. It has already been suggested as a refractory insulation material.

The deflocculators and organic binders used were Ammonium Hydroxide, Binderine, a product manufactured by the International Paper Company, and a substance known as C-35 1CP, manufactured by the Mondanto Chemical Company.

The mixing apparatus was a  $\frac{1}{4}$  H.P., 110-volt, 60-cycle, 1725 R.P.M. electric motor, which turned a propelter that extended down into the clay-water mixtures.

The pressure tank (see drawing) was made of heavy piping and had a capacity of about 3 gallons. It contained a system of valvesby which it could be operated and a pressure gage to control the pressure.

Plaster of Paris molds 1" x 1" x 6" were used to mold the specimens.

A balance which measured to the nearest .01 of a pound was used for determining the absorption and porosity.

The total porosity or bulk volume was determined in the seger type volumeter.

Analytical balances and accurately controlled weigning bottles were used in determining the true specific gravities of the different bodies. Diagram no. 1: Drawing of Pressure Apparatus



Procedure No. 1:

In order to determine which one of the three deflocculating agents as well as binders was best suited for the experiment, 500 gram samples of each clay was weighed out and mixed with water containing the different deflocculators. The Binderine, C-35 10P and Ammonium Hydroxide were mixed with water in the proportions shown in Table I and the amount of solution required to bring the clay mixture to a consistency at which it would barely flow from a container was recorded. The mixtures were agitated thoroughly with an egg beater and placed in evaporating dishes to dry. After drying, the specimens were examined and the deflocculator giving the best results determined.

TABLE I: MIX COMPOSITIONS WITH SOOgm. SAMPLES

oc of	Fli	nt Cla	У	Fult	on Plas	tic	Kaolin			
per 100 cc H <sub>0</sub> 0	Bind.NH40H		C-35 Bind. 10P		NH40H C-35 lop		Bind. NH <sub>4</sub> OH		C-35 10P	
2	CC	00	cc	cc	cc	сc	CC	cc	co	
0	520	520	520	580	580	580	5 <b>0</b> 0	<b>5</b> 00	500	
2.5	300	320	325	380	<b>40</b> 0	<b>4</b> 00	300	300	<b>3</b> 50	
5	275	250	280	320	350	<b>37</b> 5	270	2 <b>7</b> 5	<b>30</b> 0	
10	255	245	260	290	310	325	260	260	<b>2</b> 85	
15	250	245	255	280	300	310	<b>2</b> 55	<b>2</b> 55	270	



cc per 100cc of Water

#### Results:

After thoroughly drying, the cloys prepared were excinned for pore structure and strength. In every case the clays having the best pore structure were these prepared with the binderine, and their strength in the green state was also equal to those cloys prepared with Ammonium Hydroxide and reagent C-35 loP. From the standpoint of the amount of deflocculator used, the binderine also proved superior. Therefore for the other tests binderine was used in the slips as a deflocculator to reduce the amount of water necessary to cause the slip to flow. From greph No. 1, we find that 5cc of Binderine per 100 cc of water will give practically as m ch deflocculation effect as higher concentrations. Therefore 5cc of binderine per 100 cc of water were used in making the slips.

### Procedure No. 2:

Samples of 3 pounds of the clays were weighed and mixed with water containing binderine in the proportion given above. It was found that the resulting slips were too viscous to handle easily in the apparatus, so it was decided to determine the minimum amount of b nderine that would give the lowest viscosity.

For this determination, the class were made into slips by using water alone and brought to a specific gravity of 1.55. Att his specific gravity the clay-water slips were so thick and viscous that they would hardly flow. The Flint clay was tested by adding binderine inthe slips in the proportions given in Table II. After each addition of binderine the viscosity of the slip was tested with a Merriot tube viscosimeter. TABLE II. VISCOSITY TEST

Cor	ncentration of Binderine	Time of Flow for 100 cc
5500	1000cc slip	63 sec.
10	Ħ	42 #
15	Π.	40 *
Pure W	ter	26 "

From the above table it is seen that binderine in concentrations above 1% by volume of the 1.55 specific gravity slip has little effect on the viscosity. Therefore it was decided to use 1% binderine by volume in the 1.55 specific gravity slips.

The viscosity using this proportion is such that the slips will flow easily and can be handled without trouble.

### Procedure No. 3:

Here the six-member triaxial system is tested to develop a pore structure. The clay slips are adjusted to exactly the same specific gravity to facilitate blending to obtain the intermediate members.

Each member in the amount of 1000 cc is separately placed in a gallon bucket and 10 cc of binderine is added. The bucket and contents are placed under the electric mixer and thoroughly stirred.

The viscosity of the prepared body is then tested andbrought to a time of flow value of 42 seconds per 100 cc. This is done by the addition of water of if the slip is too thin by the addition of clays in the proper proportions.

A small sample, sufficient for casting a bar 1" x 1" x 6" is taken from the prepared batch and poured into the plaster mold. The remainder of the slip is placed in the pressure tank previously described. Air pressure from the supply line is then turned into the tank and allowed to reach 75 pounds per square inch, at which the valve is closed and disconnected from the line. The contents of the tank are shaken to insure thorough mixing of the air and slip. It is then let stand 5 to 6 minutes in order that the slip may become saturated with the air. A small sample is then drawn off through the tap valve in the bottom of the tank and molded in 1" x 1" x 6" plaster molds.

The air pressure is then released and the contents of the tank are subjected to a carbon dioxide pressure of 100 pounds per square inch. It is treated similiar to the sample subjected to air pressure and molded in the same manner. The same procedure is duplicated for the other members of the triaxial system.

After the bars have been molded and dryed sufficiently, they are removed from the plaster molds and cut into test specimens 1" x 1" x 2" and marked for identification.

Diagram No. 2 and Table III shows the system of numbering and the composition of each member in the system.



Diagram No. 2

#### TABLE III

COMPOSITIONS OF MEMBERS OF TRIAXIAL DIAGRAM Body No. Composition 1;.... 100 % Flint Clay 2.... 50 % Flint Clay 50 % Fulton Plastic Clay 3.... 50 % Fling Clay 50% Kaolin 4.... 100% Fulton Plastic Clay 5.... 50 % Fulton Plastic Clay 5.... 100% Kaolin 6.... V. 100% Kaolin

The letters A, B, and C on the specimens are used to represent the treatment given the bars.

A. Nopressure

B. 75 pounds of air pressure

6 100 pounds of Carbon Dioxide pressure

Thus No. 1-A-2 represents body No. 1 (Flint Clay), treated with no pressure and specimen no. 2.

The test pieces are then dried thoroughly in a dryer at 200°C for 12 hours and fired in a Globar furnace to cone 8 (2300°F) in 8 hours.

The following determinations were made:

1. Dry weight.

2. True specific gravity of each bdy

3. Absorption or open pore space.

4. Bulk volume.

From these determinations the percentage of open and total porosity was calculated.

The open pore space is determined by soaking the test pieces in kerosene under a vacuum of 27.5 inches of mercure for 12 hours and then weighing the saturated blocks. The difference between the saturated weight and dry weight divided by the density of kerosene gives the volume of the open pores.

The bulk wo lume is determined by recording the displacement of the saturated blocks by means of burettes arranged to receive the overflow from a filled ontainer. This is known as the Seger type volumeter. Formulas used for Calculations: True volume = Dry weight True specific gravity Bulk Volume = Displacement of kerosene True specific gravity = (Wt. of Pic. and sample)-(Wt of Pic) = Wt of Pic and sample - wt of Pic)-(wt of pic, sample, water - pic and water) Vol. open pores = Saturated wt. - Dry wt. Density of kerosene Total Pores vol. = Bulk vol. - True vol. % open porosity = - Vol. open pores Bulk volume x 100

% Total Porosity = Vol. total pores x 100 Bulk vol.

#### DATA AND RESULTS

The data and results obtained are shown in Table IV which is one the following pages. TABLE IV: DATA ON TOTAL AND OPEN PORE VOLUME

Numbe <b>r</b>	Dry wt	Sat wt	Bulk vol.	True vol.	Vol. open pores	Tol. total pores	% open pores	% total Porosity
1-A-1	33 <b>.</b> 38	38,60	18.80	11.45	6 <b>.76</b>	7.35	36.0	39.1
1-A-2	42.45	49.12	<b>3</b> 3 <b>.</b> 70	14.57	8.34	9.13	35.2	38.5
					Av	erage	35,6	38.8
1-B-1	37.90	44.15	21.2	12,98	7.82	8.22	36,85	38.8
1-8-2	36.68	42.70	20.6	12.6	7,51	8.0	36,44	38.8
					A	verage	36.63	38.8
1-C-1	88.10	35.71	19.9	9.66	9.40	10.24	47,30	51.5
1-C-2	23/59	29.78	17.0	8.09	7.74	8.91	45.50	52.5
					A	verage	46.4	52.0
2*A-1	46,98	51.38	23,4	17.3	5,50	6.1	23,48	26.2
2 <b>-</b> A-2	<b>48</b> ,99	53.46	24.3	18.06	5,59	6.24	23.0	25.64
					Av	erages	23.24	25.92
2-B-1	40,36	44.83	20.7	14.9	5,59	5.8	27.0	28.0
2-B-2	41.32	46.16	21.6	15.2	6.05	6.4	27,9	28.3
					Av	erage	27.45	28.15
2-C-1	13,36	15,15	7.5	4,93	2,24	2.57	30,00	34.3
2-0-2	10,15	11.60	5.7	3,75	1,81	1.95	31.88	34.1
					Ave	erage	30,94	34.2
3-A-1	42,34	47.39	23.0	16.52	6.32	6.48	27.42	28.2
3-A-2	44.43	48.61	24.6	17.35	7.06	7.25	28.7	29.42
3- <u>A</u> -3	40,20	44.81	22.0	16.1	5,76	5,90	26.2	26.81
					Ave	erage	27.44	28,14

TABLE	IV	,	Con't.

Number	Dr₩ wt	Sat wt	Bulk vol.	True Vol.	Vol. open pores	Vol. Total pores	% open pore <b>s</b>	% <b>Total</b> porosi <b>t</b> y	
3-B-1	<b>3</b> 5 <b>.7</b> 0	40.84	20,5	13.94	6.43	6,56	31.3	32.0	
3-B-2	40.40	46.42	23.5	15,80	7.52	7,70	32.0	32.8	
3-B-3	43.54	49.49	24.9	17.03	7.44	7.87	29.82	31.6	
					Ave	age	31.04	32.1	
3-C-1	20,55	25.87	14,90	8.05	6,67	6.85	44.7	45.9	
3-0-2	25.19	32.09	18,5	9.80	8.62	8.70	46.5	47.0	
					Ave	age	45.6	46.5	
4 <b>-A</b> -1	26.62	29.48	13.5	9,80	3.58	3,70	26.5	27.4	
					Aver	age	26.5	27.4	
4-B-1	26,98	29.86	14.0	9.92	3,60	4.08	25.75	29.2	
4-B-2	30,72	34,12	16.0	11,30	4,25	4.7	26.57	29.41	
					Ave	ages	26,16	29.30	
4-C-1	26.10	28,68	13.3	9,60	3.21	3 <b>.7</b> 0	24.1	27.8	
4-C-2	20,96	23.18	10.7	7.70	2.78	3.0	23.75	27.92	
					Ave	age	23,93	27.86	
5 <b>-A-</b> 1	51.51	56.89	25.9	18.5	5.48	7.4	21.2	28.6	
5-A-2	<b>4</b> 5 <b>.7</b> 5	50.57	22.8	16.4	6.03	6 <b>.40</b>	26,42	28.1	
5-4-3	<b>4</b> 9.66	54 <b>.96</b>	24.8	17.80	6.62	7.0	26.7	28.22	
					Avera	age	24.77	28.31	
5-B-1	49,63	55.70	26.0	17.74	7.60	8,26	29,24	31.8	
5 <b>-B-2</b>	48.06	54,57	26.3	17.21	8,15	9.09	29 24	31.8	
5-B-3	<b>4</b> 6 <b>,</b> 96	52,60	24.2	16,81	7.05	7.39	29,12	30,55	
					Av	erage	29.79	32.29	

TABLE IV: Con't.

Number	Dry wt	Sat wt	Bulk Vol.	True vol.	Vol. open pores	Vol. Total pores	% open pores	% Total Porosity
5-C-1	31,16	36.44	17.8	11.15	6.61	6.65	37.2	37.5
5-0-2	33,00	39,24	19,9	11,82	7,80	8.08	39.2	40.6
5 <b>-C-</b> 3	32,97	38,75	19,4	11.80	7.22	7.60	37.22	39.2
						Average	37.87	3911
6 <b>-</b> A-1	28.71	35,54	20.0	11.31	8.54	8 <b>.69</b>	42.5	43.4
6 <b>-A-</b> 2	31,97	39.67	21.9	12.11	9.63	9.79	43.9	44.7
						Average	43.2	44.1
6-B-1	30,32	38.32	22.0	11,96	10,00	10.04	45.5	45.5
6-B-2	34.74	44.18	25.4	13.7	11.67	11.70	45.8	46.2
						Average	45.6	45.8
6 <b>-C-1</b>	19,64	25.34	14.9	7.75	7.10	7.15	47.6	48.0
6 <b>-C-</b> 2	23.63	30.43	17.9	9.35	8,50	8,55	47.5	47.8
6- <b>C-</b> 3	21.75	28.33	16.9	8.57	8.23	8.33	48.6	49.1
					_	Average	47.9	48.3
A. P. (	Greene B	rick:						
no.1	17.43	32.0	25.47	6.53	18.9	18,94	74.2	74.4
no. 2	15.84	30,16	24.0	5.94	17.9	18.06	74.5	75.2
					د	Average	74.3	74.8





#### TABLE V: TRUE SPECIFIC GRAVITIES

Body No. Specific Gravity 1 . . . 2.9028 2 . . . 2.7141 3 . . . 2.5552 4 . . . 2.7141 5 . . . 2.7866 6 . . . 2.5359 Kerosene. . . 800

#### DISCUSSION OF DATA

The data shows that a considerable pore structure can be developed by treating clay slips with gas pressure. The results obtained show that the air pressure and the carbon dioxide pressure caused a definite increase in the porosity over the untreated samples except in one case where 100% of Fulton Plastic clay was used. Here the treatment with pressure apparently has no effect on the porosity.

From the standpoint of porosity, the best insulator was obtained from Flint Clay treated with carbon dioxide gas. In this sample the total porosity was 52%. This value is low however as compared to a commercial insulating brick which has a porosity of 74.8 when tested along with the other specimens.

The body showing the greatest increase in porosity due to treatment is No. 3 which contains 50% Flint clay and 50% Kaolin. In this body the porosity is 46.5% for the carbon dioxide treat-

ed blocks and 28.14% for the untreated blocks, or an increase of 18.36% in the porosity.

Each separate treatment shows rather uniform results, which indicates that the higher porosities obtained in the blocks which were treated with pressure were not merely accidental.

The open pores and the total pores in each specimen show very little difference in volume, which indicates that most of the pores are open.

#### SUMMARY

An insulating brick was to be made by forming the pore structure with dissolved air and carbon dioxide in a clay slip. To aid in the operation, deflocculating agents were placed in the slips to reduce the amount of water necessary to form the slip and also keep the clays in suspension. Organic binders were to be used to wid the green strength of the clay. The best reagent found to deflocculate the slips and also act as a binder was a substance known as binderine.

The binderine was used as a reagent in the slip which was brought to a specific gravity of 1.55 and treated with air pressure and carbon dioxide gas of 75 pounds and 100 pounds of respectively. The treated slips were then molded in plaster of Paris molds and porosity run on the fired pieces. A Flint clay showed the greatest porosity of 52% when treated with a pressure of 100 pounds per square inch in an atmosphere of carbon dioxide. The body showing the greatest response to the treatment contained 50% flint clay and 50% kaolin.

#### CONCLUSIONS

1. Air pressure and carbon dioxide gas pressure on a clay slip will cause an increase in the porosity of the fired body for some clays and clay mixtures.

2. The porosity will vary with the clays even though the treatments are identical.

3. The porosity depends upon the viscosity of the slip and upon the reagents used in the clay slips.

4. The drying operation seems to offer the most difficulty.

## APPLICATION TO INDUSTRY

The specimens dried in non-absorptive molds showed very uneven porestructure, while those dried in plaster of Paris molds had an even pore structure. This would tend to favor drying in porous molds, which is a long and expensive process for solid pieces. However it maybe made profitable if a proper system of automatically handling the molds could be arranged.

When the properreagents and treatment have been found that give a suitable pore structure to the clay slips when treated with pressure, the equipment for plant production will be simple. A large tank in which clays could be mixed under pressure would suffice for the mixing apparatus. A plant equipped to manufacture its own gases as a by-product of another process could probably make thebrick on an economical basis. However, much work remains to be done before a definite method or process can be put into use.

## RECOMMENDATIONS FOR FURTHER RESEARCH

For further research I would suggest studying the effect of violently agitating the clay slip hile it is being subjected to pressure. This may be done by means of a shaft through the walls of the pressure tank, using a stuffing nut to make the tank air tight. The effect of different gases which are reasonably soluble in water may also be investigated.

In this experiment the slips that had been treated with air pressure were then subjected to carbon dioxide pressure. It would be well to know the effect of each of these gases independently of the other. In this case a new sample would be required in each treatment.



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