(No Model.) 4 Sheets—Sheet 1.

L. M. C. FOLIEDESJARDINS.

APPARATUS FOR THE MANUFACTURE OF PHOSPHORUS AND ALKALINE SILICATES.

No. 460,004. Patented Sept. 22, 1891.

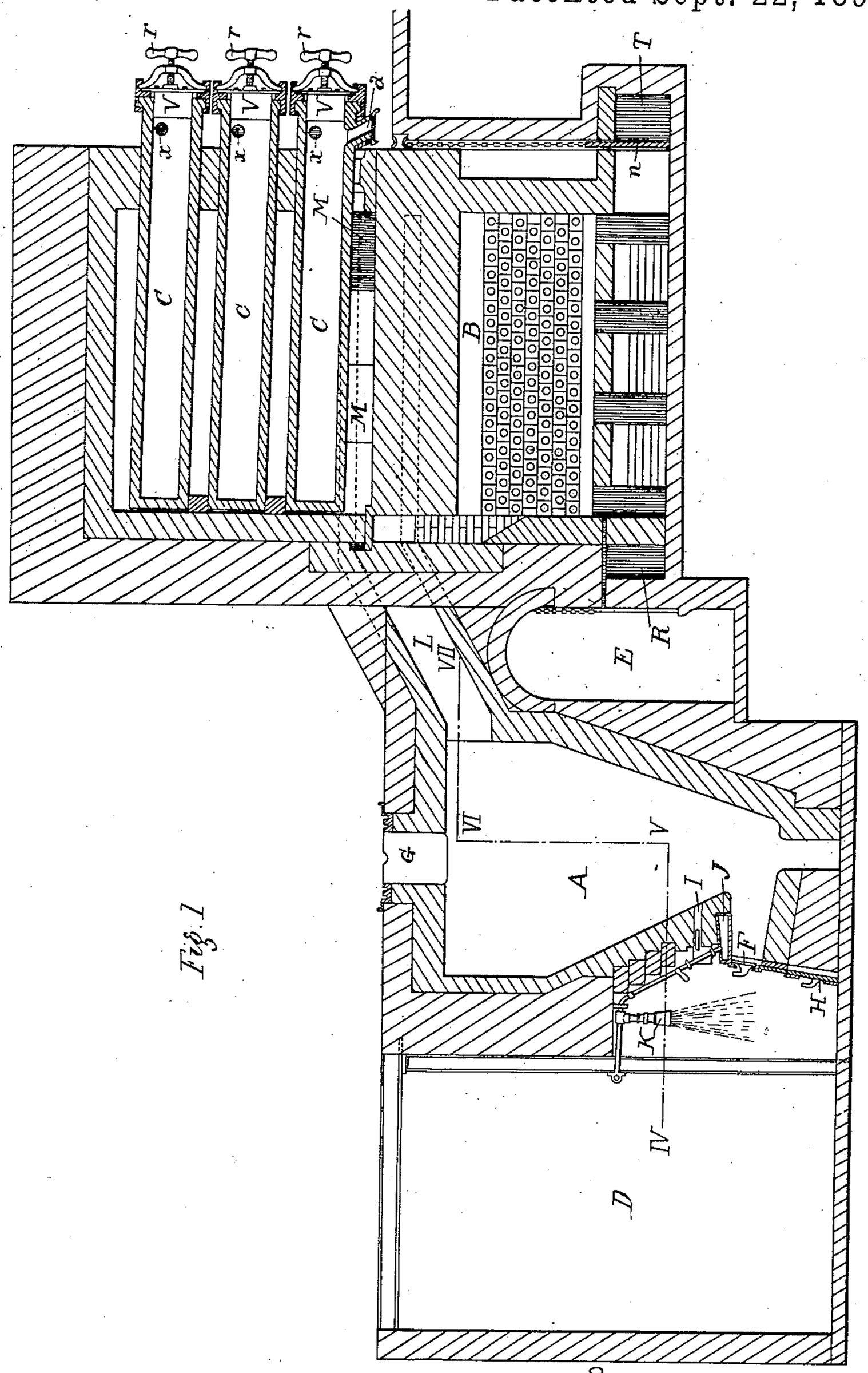

Fig. 1.

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Fig.

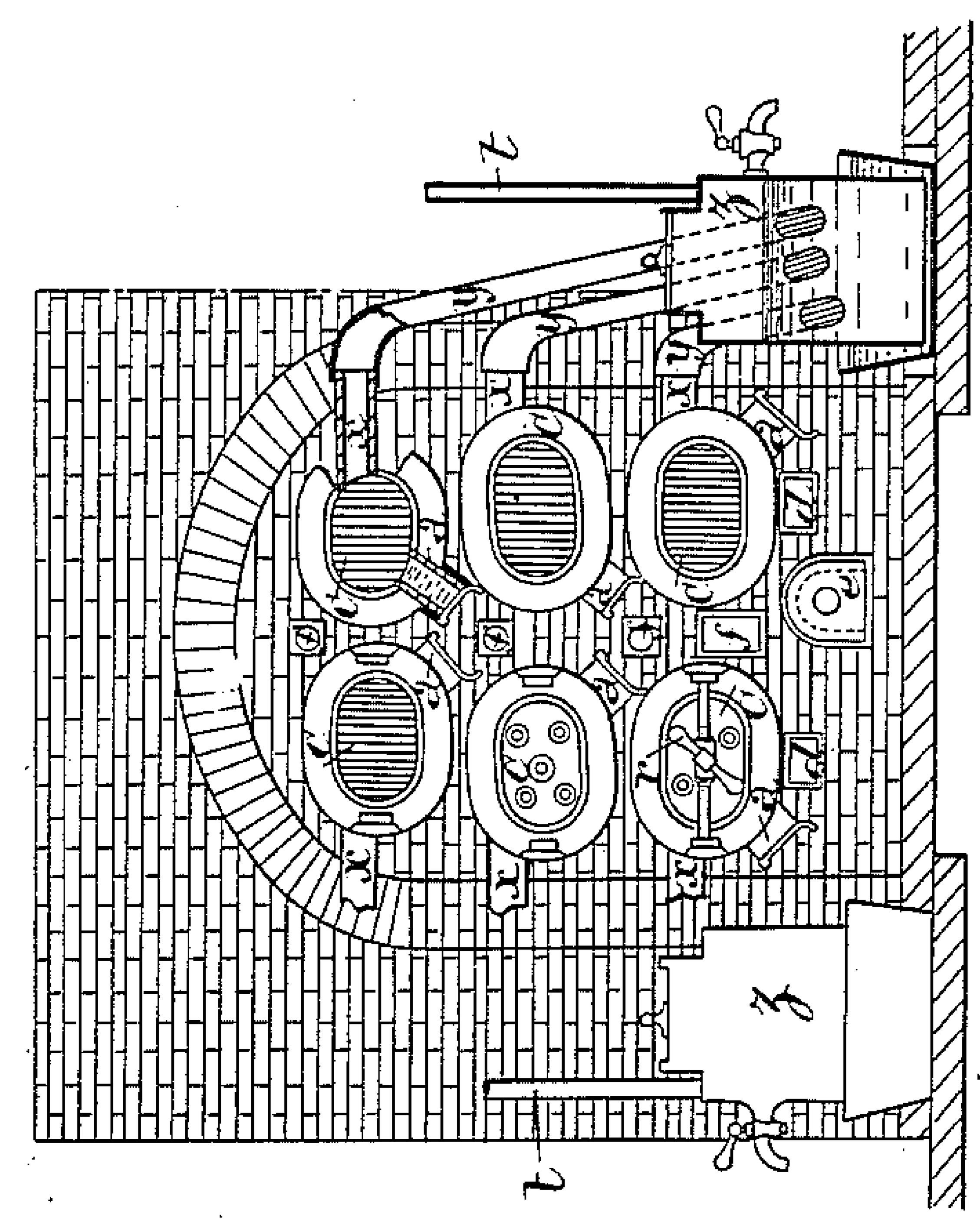

Fig. 2

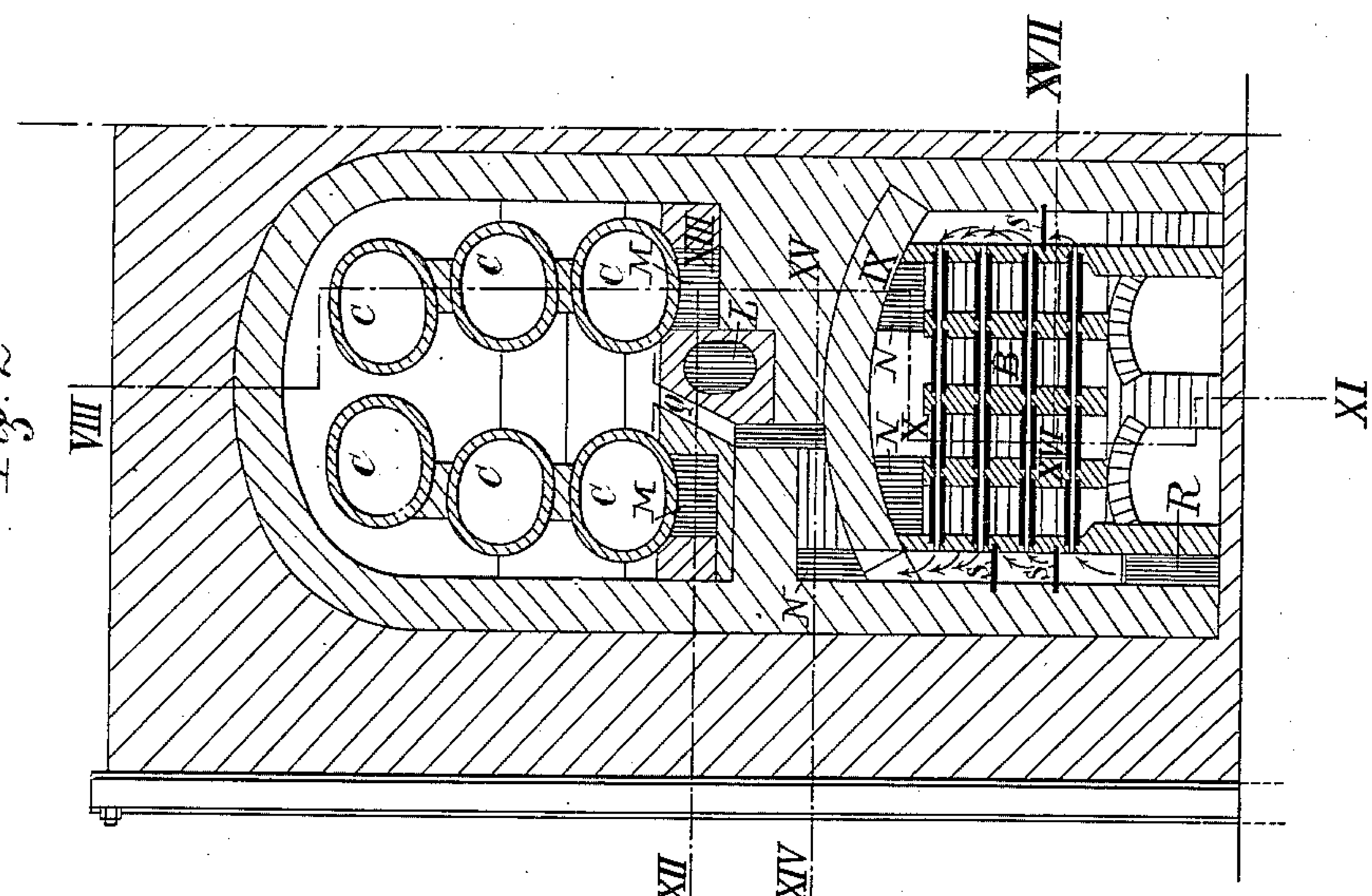

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Fig. 3.

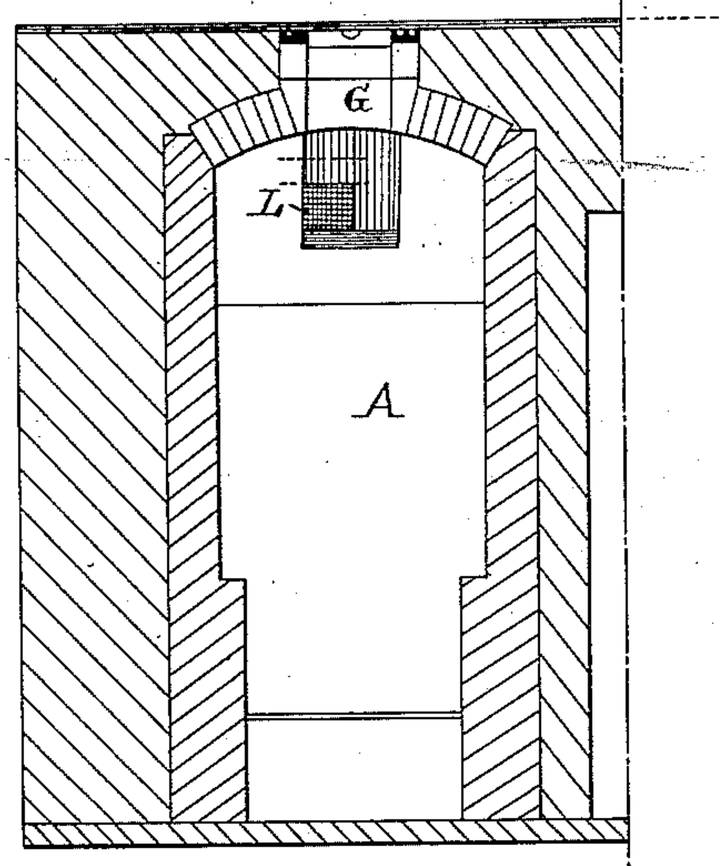

Fig. 4.

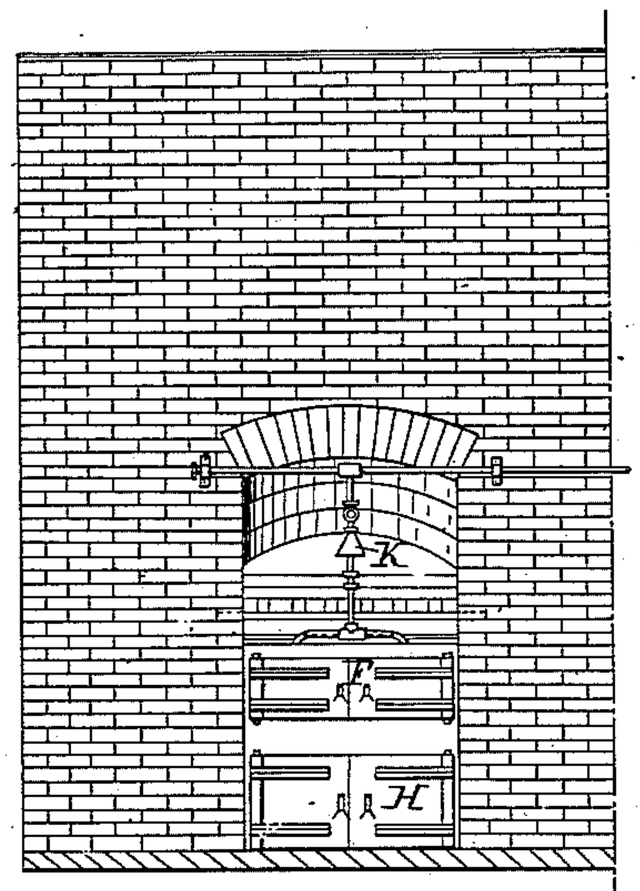

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Fig. 6.

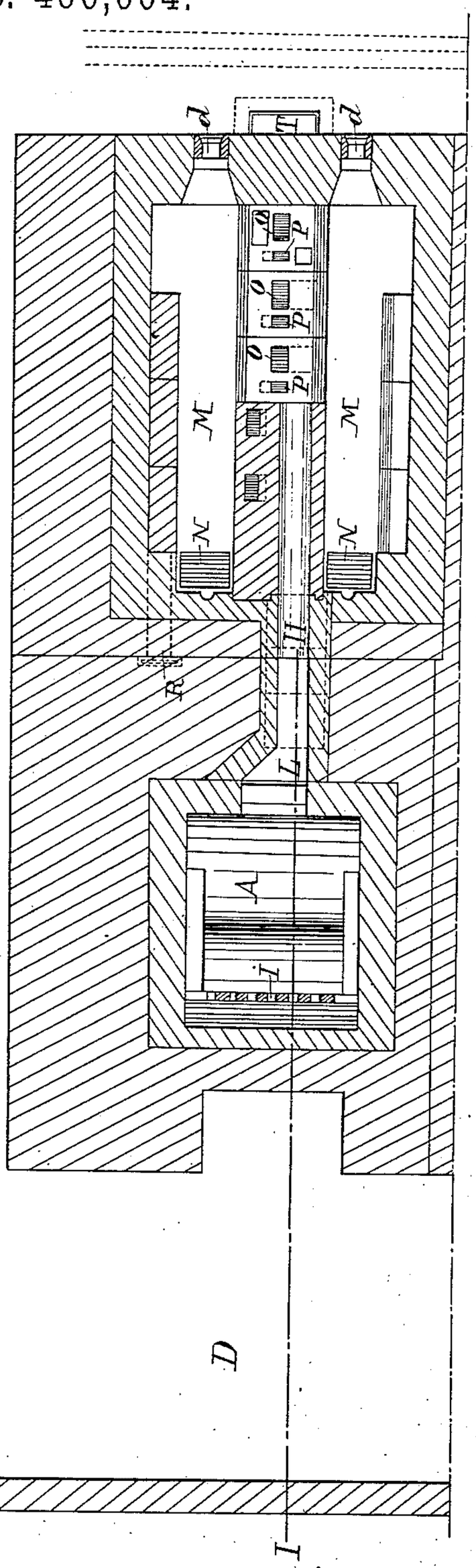
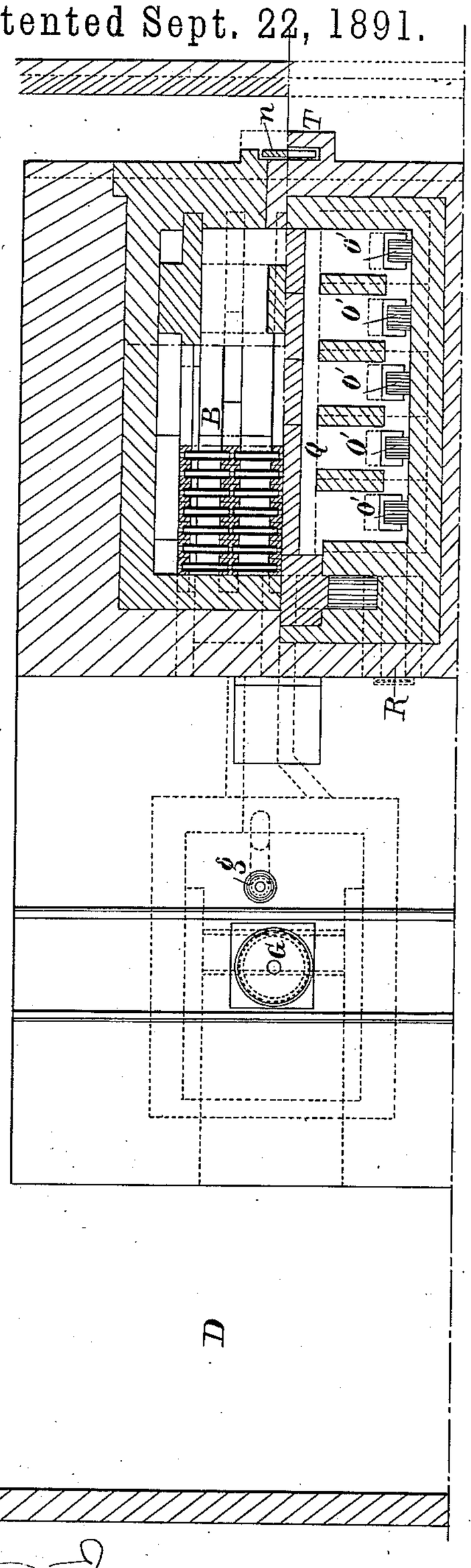


Fig. 7.



Witnesses:-
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Percy B. Hills.

Inventor:
Louis M. Charles Foliedesjardins
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UNITED STATES PATENT OFFICE.

LOUIS MARIE CHARLES FOLIEDESJARDINS, OF TOULOUSE, FRANCE.

APPARATUS FOR THE MANUFACTURE OF PHOSPHORUS AND ALKALINE SILICATES.

SPECIFICATION forming part of Letters Patent No. 460,004, dated September 22, 1891.

Application filed September 13, 1890. Serial No. 364,874. (No model.) Patented in France February 24, 1890, No. 203,942.

To all whom it may concern:

Be it known that I, LOUIS MARIE CHARLES FOLIEDESJARDINS, chemist, a citizen of the Republic of France, residing at Toulouse, Haute-Garonne, France, have invented a new and useful Apparatus for the Simultaneous Manufacture of Phosphorus, Alkaline Phosphates, and Alkaline Silicates, (for which I have obtained a patent in France, No. 203,942, dated February 24, 1890;) and I do hereby declare that the following is a full, clear, and exact description of the same.

My invention relates to an improved apparatus for the simultaneous manufacture of phosphorus, alkaline phosphates, and alkaline silicates on a commercial scale, and has for its object to transform the manufacture of phosphorus from an unhealthy industry into one that is unattended by danger to the health or comfort of the work people and without inconvenience or detriment to adjacent property or to the public health.

In the annexed drawings, Figure 1 shows in vertical section the gas-generator and the furnace or oven with its regenerator. The right-hand portion is a vertical section of the furnace or oven with its regenerator on the line VIII IX X XI of Fig. 2. The left-hand portion is a vertical section of the gas-generator on the line I II of Fig. 6. Fig. 2 is a cross-section of the furnace or oven with the regenerator. Fig. 3 is a cross-section of the gas-generator, taken through the charging-opening G. Fig. 4 is a front elevation of the gas-generator. Fig. 5 is a front elevation of the furnace or oven with its condensers. Fig. 6 is a horizontal section of the gas-generator on the line IV V VI VII of Fig. 1, and a horizontal section of the regenerator on the line XII XIII XIV XV of Fig. 2. Fig. 7 is a plan of the gas-generator and a horizontal section of the regenerator on the line IX X XVI and XVII of Fig. 2.

The apparatus comprises a furnace or oven in which are arranged a suitable number of retorts. These retorts C, which are of refractory earth, may, if it be thought desirable, be provided with an internal lining of magnesite. They have a slight inclination to allow of the overflow either of the product resulting from the mineral phosphates or the bone-ash in the first operation or of the residual alkaline silicate from the second operation.

The retorts are furnished with a tap-hole *a*. The plugs V for closing the retort and the tap-hole *a* are furnished with an internal lining of refractory material surrounded by a sleeve of asbestus for insuring tightness of joint. They are closed by means of a pressure-screw *r*. Each retort is provided at its upper part at the side near the opening of the retort with a pipe *x* for allowing of the escape of gases and formed in one piece with it for a certain length. Brass extensions *y* (in the manufacture of phosphorus) are fitted on the pipe *x*, and serve to conduct the gas into the condenser *z*. On the right-hand side of Fig. 5 the condenser *z* is in section, and is shown as connected by the extensions *y* to the pipes *x*, whereas on the left of said figure the condenser is in elevation and the extensions *y* leading thereto have been broken away. A condenser *z*, provided with a draft flue or chimney *t*, communicates with a central conduit, which conveys the carbonic oxide and other uncondensed gases into a gasometer or gas-holder. (Not shown.)

One or more gasometers are provided in the manufacture of phosphorus for the reception of combustible gases, which on passing from the last gasometer are conveyed by a pipe to the burners, where they are to be utilized according to requirement.

A is the gasometer; B, the regenerator of the waste heat; D, the gallery or passage for working the gas-generators; E, the gallery or passage for working the regenerators; F, the door of the furnace or oven; U, the charging-plug, and H the ash-pit door.

g, Fig. 7, is a sight-hole, closed by a cast-iron plug, for enabling the conduit L for carbonic oxide to be examined and to be cleaned from dust carried along in suspension.

I, Figs. 1 and 6, are openings provided with cast-iron plugs for the passage of cleaning-bars during the cleaning.

J is a cast-iron port serving for the introduction of air and water into the gas-generator.

K, Figs. 1 and 4, is the rose of a sprinkler,

serving for the rapid extinction of the ashes resulting from the cleaning.

L is a flue for carbonic oxide.

M are flues for smoke and products of combustion which pass through N underneath the roof of the regenerator, and after passing between the tubes of the regenerator enter the chimney.

O, Figs. 2 and 6, are exit-orifices for hot air.

o', Fig. 7, are orifices for placing the last rows of tubes into communication with the air-chamber Q.

P, Fig. 6, are exit-orifices for carbonic oxide.

Q, Fig. 7, is an air-chamber placing the orifices O for hot air into communication with one another.

R, Figs. 1, 2, 6, and 7, is an inlet for cold air into the regenerator.

S, Fig. 2, are internal baffles compelling the cold air to pass into all the rows of tubes, as indicated by the arrows.

T, Figs. 1, 6, and 7, is a chimney furnished with a register *n*.

d e f, Figs. 5 and 6, are sight-holes lined with cast-iron.

The treatment of the phosphates in the first operation by means of alkaline sulphates or carbonates may be effected by means of a reverberatory furnace having a slightly-inclined bottom or hearth. However, if it is desired to utilize the carbonic acid or the sulphurous acid, retorts and furnaces or ovens may be employed similar to those just described, except that tube *x* of the retorts C communicates in such a case with a central conduit and not with a condenser, and the carbonic-acid gases or sulphurous-acid gases are conducted to apparatus which are intended to store up or else directly utilize the same.

The first operation to be performed is the elimination of the phosphoric acid from the phosphate of alumina or phosphate of lime by means of an alkaline sulphate or carbonate, or by means of a mixture of carbonate of soda and carbonate of potash, or by means of a sulphate of these two bases. As already stated, this may be effected either in a reverratory furnace or in retorts of refractory earth heated by means of carbonic oxide.

X is the tube serving for the escape of the carbonic acid, (CO_2.) This gas passes by a central conduit toward the place of storage or utilization. Thus the carbonic acid may serve for the treatment of the residual silico-aluminate of soda (NaO,Al_2O_3,SiO_2) or for any other purpose.

The calcined matter obtained is composed of silico-aluminate of soda and of tribasic phosphate of soda, (3NaO,PO5.) It is porous and can be easily lixiviated. When subjected to lixiviation, the alkaline tribasic phosphate is dissolved out. It is then evaporated to dryness and pulverized. The residue of this first operation is a silico-aluminate of soda, (NaO,Al_2O_3,SiO_2.)

The second operation has for its object to eliminate from the alkaline phosphate the phosphoric acid (PO_5) and to reduce the said acid. For this purpose the alkaline phosphate, silicic acid, and carbon are mixed intimately together. The mixture is subjected to the action of heat in the retorts C of a furnace or oven heated by means of carbonic oxide, as illustrated in Figs. 1 and 5 of the annexed drawings.

The reaction to be produced is illustrated by the following equation in the example I have taken.

$$3NaO,PO_5+3SiO_2+5C=3(NaO,SiO_2)+P+5CO.$$

The intimate mixture being raised to a bright red heat in the retorts, the silicic acid attacks the base, forming an alkaline silicate, and liberates the phosphoric acid. The latter being free is reduced by the carbon, which unites with the oxygen of the acid to form carbonic oxide, (CO.) The volatilized phosphorus (P) passes by the pipes *x* and *y* into the condenser *z*, where it is collected. The carbonic oxide and the uncondensed gases pass into the central conduit and through the chimney *t* into a gas-holder. The combustible gases thus collected are then, according to the requirements of the case, conveyed to the burners of furnaces, or preferably to furnaces for regenerating the waste heat. The products of the operation are therefore, first, phosphorus, which is to be purified by the usual means; second, a simple or complex alkaline silicate, such as in the example chosen—a silicate of soda. The degree of acidity of the alkaline silicate may vary at the will of the operator, according to the use for which it is intended.

The residual silico-aluminate of soda contains sesquioxide of iron. This residue may be treated by known means so as to yield a precipitate of alumina and sesquioxide of iron, gelatinous silica, and a caustic alkaline or an alkaline carbonate.

The separation of the sesquioxide of iron from the alumina may be effected by means of trimethylamine, ($C_2H_2NH_3$,) which precipitates the sesquioxide of iron and the alumina and then redissolves the latter. This residue must not, therefore, be considered a waste product, for it is possible to recover the greater part of the alkaline salt. If in place of phosphate of alumina, phosphate of lime, and an alkaline carbonate are employed, for instance, the addition of silica may be dispensed with, the carbonate of lime obtained remaining in suspension in the melted material, being insoluble in it.

The first operation will thus be represented by one of the following equations: first, bone-ash $= 3(PO_5,3CaO) + 18(NaO,CO_2) = 3(PO_5,3NaO)+9NaO+9CaO,CO_2+9CO_2$; second, fossil phosphate of lime $=(3PO_5,3CaO)+CaFl+18(NaO,CO_2)=3(PO_5,3NaO)+CaFl+9NaO+9CaO,CO_2+9CO_2$. The product of the reactions when subjected to a slight lixiviation and allowed to rest deposits the carbonate of

lime (or, as the case may be, carbonate of lime and fluoride of calcium) and there remains a solution of tribasic phosphate of soda and hydrated protoxide of sodium which is to be evaporated to dryness. With the product an intimate mixture is made with silica and carbon, which is submitted to the action of heat, as hereinbefore stated.

The reactions obtained are represented by the following equation:

$$3(PO_5,3NaO)+9NaO+9SiO_2+15C=3P+9Na_2O,2SiO_2+15CO.$$

If desired, the acidity of the silicate may be modified by increasing the proportion of silica, as before mentioned. The result is, first, impure phosphorus; second, an alkaline silicate; third, carbonic oxide.

As it is possible to modify the degree of acidity or of alkalinity, so is it also possible by mixing the alkalies to obtain complex silicates of potash and soda.

I claim—

In an apparatus for the manufacture of phosphorus, alkaline phosphates, and alkaline silicates, the combination of a furnace or oven, the retorts C, having pipes *x*, provided with extensions *y*, the condenser *z*, having a chimney *t*, the gasometer A, and the regenerator B, substantially as shown and described.

In witness whereof I have hereunto signed my name in the presence of two subscribing witnesses.

LOUIS MARIE CHARLES FOLIEDESJARDINS.

Witnesses:

D. H. BRANDON,

A. LE CAM.