PROCESS OF PREPARING ARSENIC TRIOXID.

A SHEETS—SHEET 1.

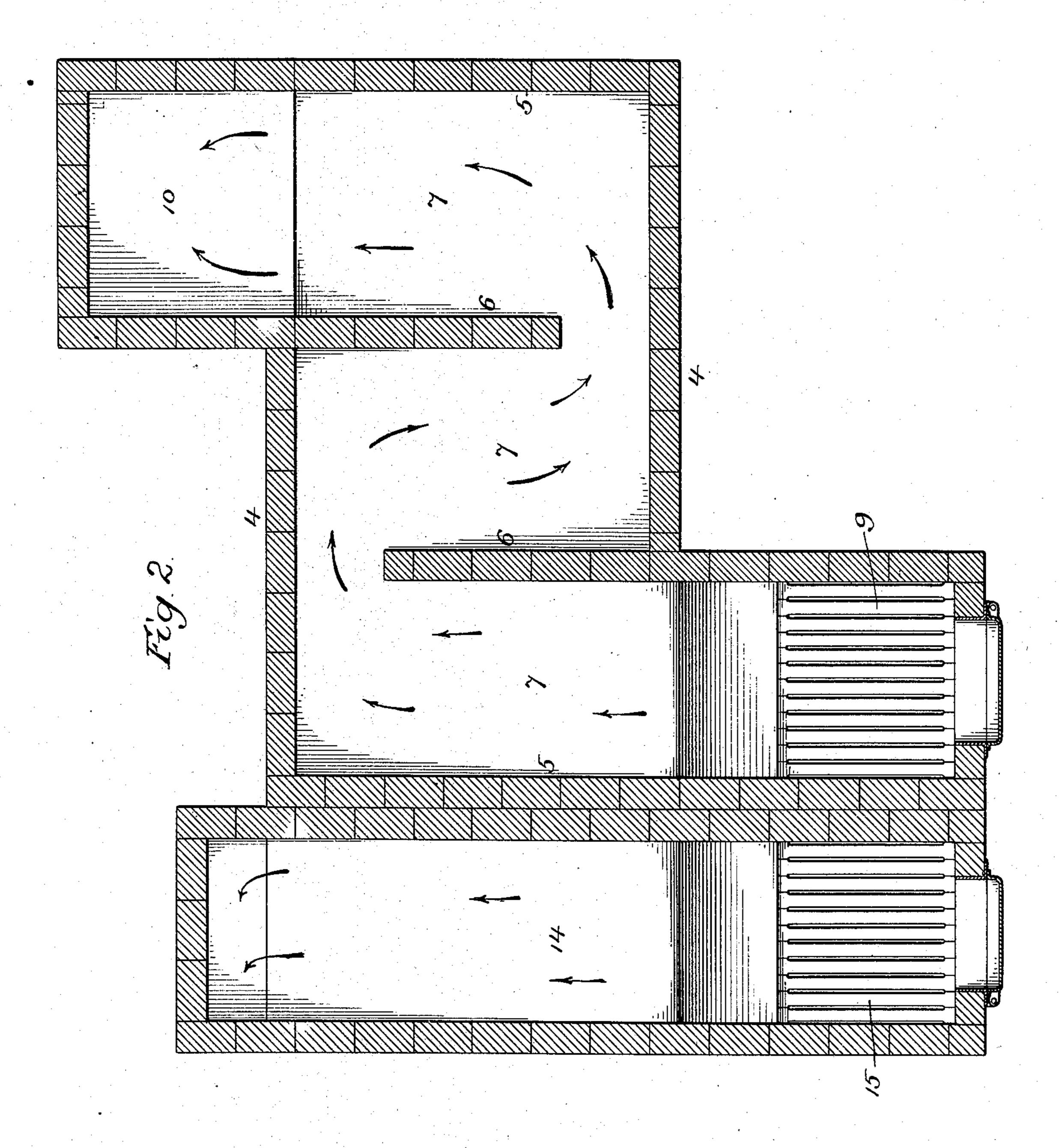
Witnesses Hamilton D. Zumer Kate a. Beadle

Utley Wedger Brither attorneys Smith Kazier

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APPLICATION FILED JUNE 15, 1906.

4 SHEETS-SHEET 2.

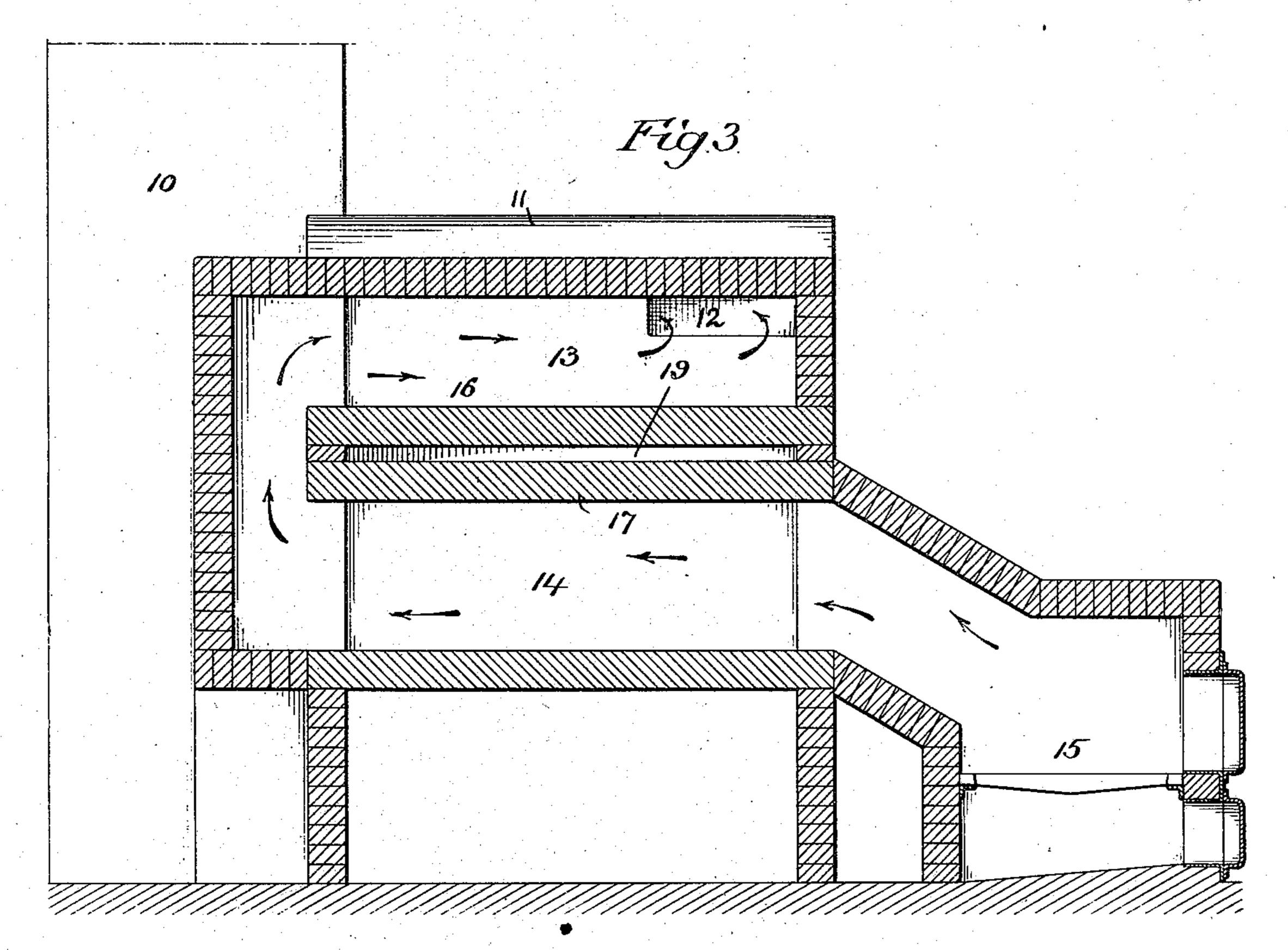


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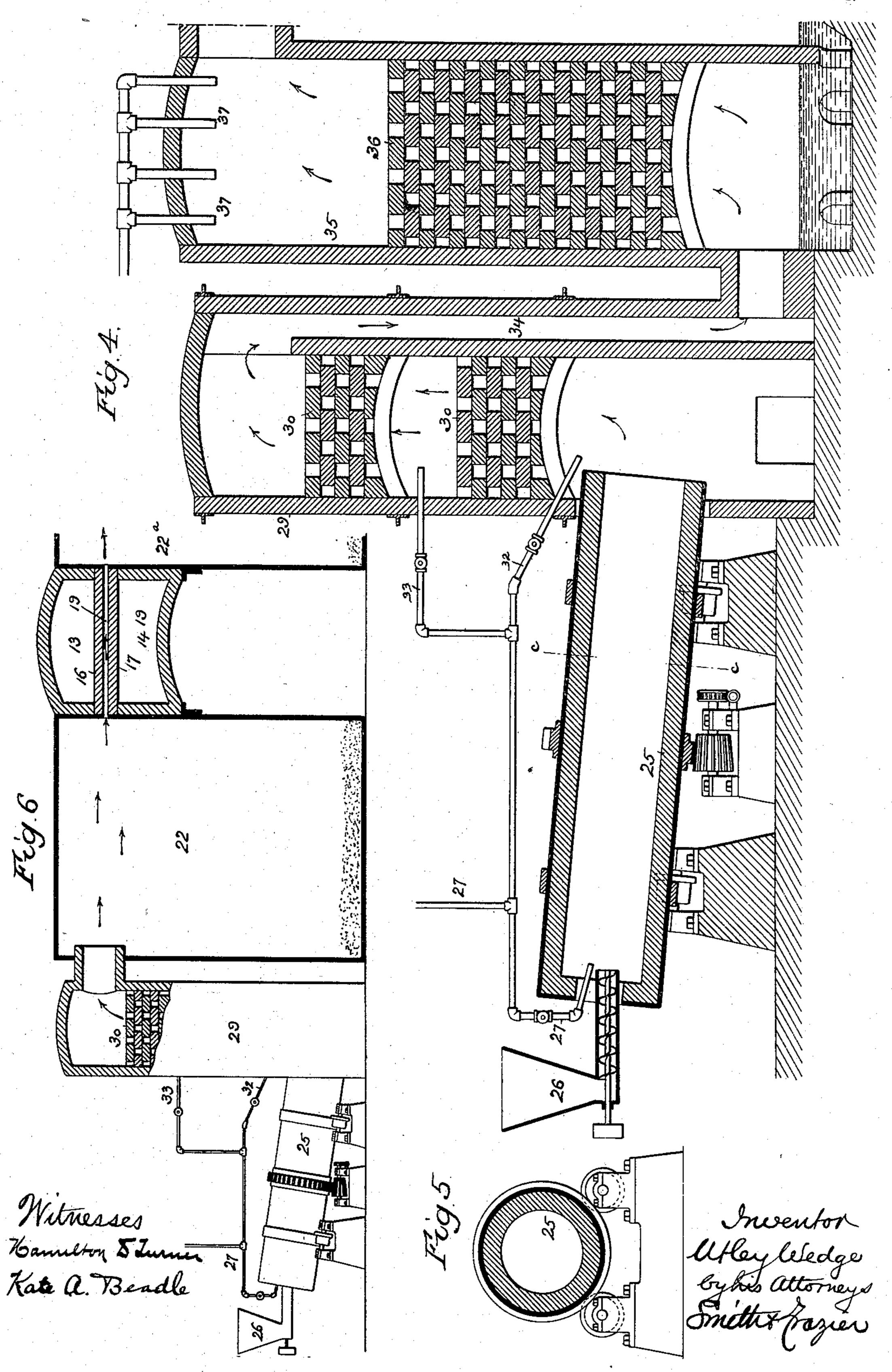


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4 SHEETS-SHEET 4.



UNITED STATES PATENT OFFICE.

UTLEY WEDGE, OF ARDMORE, PENNSYLVANIA.

PROCESS OF PREPARING ARSENIC TRIOXID.

No. 894,764.

Specification of Letters Patent.

Patented July 28, 1908.

Application filed June 15, 1906. Serial No. 321,887.

To all whom it may concern:

Be it known that I, UTLEY WEDGE, a citizen of the United States, residing in Ardmore, Pennsylvania, have invented certain Improvements in Processes of Preparing Arsenic Trioxid, of which the following is a

specification.

The object of my invention is to recover, in commercial form, the arsenic trioxid contained in the sulfid of arsenic which is at present a waste product of sulfuric acid manufacture. This object I attain by subjecting the sulfid to a sufficient degree of heat to fuse the same while at the same time it is gently agitated as by causing it to flow in a sluggish stream down the inclined bed or hearth of a furnace, the vapors evolved by this treatment being then subjected to a sublimating process whereby the arsenic trioxid is recovered while the sulfurous acid gas is permitted to escape.

is a longitudinal section of a stationary furnace which I have devised for the purpose of carrying out my invention; Fig. 2 is a sectional plan view of the same on the line a-a, Fig. 1; Fig. 3 is a transverse section on the line b-b, Fig. 1; Fig. 4 is a longitudinal section of a rotary furnace for the carrying out of my invention; Fig. 5 is a transverse section of the same on the line c-c, Fig. 4, and Fig. 6 is a view, partly in side elevation and partly in section, and on a reduced scale, illustrating another embodiment of my invention.

I will first describe the stationary furnace shown in Figs. 1, 2 and 3, and the method of carrying out my invention with the aid of

such furnace.

The hearth of the furnace has upper and lower horizontal sections 1 and 2 on different levels and an intermediate inclined connecting section 3, these sections of the hearth being composed of slabs, tiles or bricks of re-15 fractory material, and being supported upon side walls 4, end walls 5 and transverse piers or arches 6 so as to form, beneath the hearth, a series of connected flues 7, which receive the products of combustion from a furnace 9 50 and after they have circulated through the flues and beneath the hearth discharge them into a stack 10 at one corner of the furnace. Any desired flow of the products of combustion beneath the hearth of the furnace may 55 be provided for, by proper disposition of the piers or arches 6.

The arched top 11 of the furnace is hollow and contains a flue 12 which is also in communication at one end through a passage 12^a with the stack 10 and at the other end with 60 the uppermost of a pair of communicating transverse flues 13 and 14, the lowermost of which flues receives the products of combustion from a furnace 15.

Interposed between the upper and lower 65 flues 13 and 14 are two tiers of slabs or tiles 16 and 17 composed of refractory material, these tiers of slabs or tiles being separated slightly so as to form between them a contracted passage 19 communicating at one end 70 with the furnace chamber 20, and at the other end with a neck 21 which leads to the

sublimating chamber 22.

In either or both of the side walls of the furnace adjacent to the upper section 1 of 75 the hearth, is a feed opening 23, and in the end wall adjacent to the lower section 2 of the hearth is a delivery opening 24, these openings permitting the inflow of sufficient air to support the combustion of the mass de-80 posited upon the hearth of the furnace.

When the furnace is in operation the products of combustion circulate through the flues 7, 13 and 14 and through the flue or passage 12, and the hearth and roof of the 85 furnace and also the slabs or tiles 16 and 17

become highly heated.

The sulfid of arsenic which results as a waste product of sulfuric acid manufacture, is usually a combination of the trisulfid and 90 the pentasulfid, and owing to the loosely combined atoms of sulfur in the latter, the compound will, when thrown and spread upon the upper section 1 of the hearth, be readily fused or melted by the heat and will flow in a 95 sluggish stream down the inclined section 3 of said hearth and onto the base section 2 of the same. By reason of such flow new surfaces are being constantly exposed to the heat of the hearth and to the heat radiated 100 from the roof of the furnace, the result of such treatment being the evolution of arsenious oxid vapor and sulfurous acid gas, which pass through the contracted passage 19 between the slabs 16 and 17, and are there sub- 105 jected to further heat so as to raise their temperature and oxidize any particles of sulfid which may have been carried off either mechanically or as vapor with the other vapors or gases from the working chamber 20 110 of the furnace.

When the vapors or gases enter the cham-

2 894,764

ber 22 the arsenic trioxid gases or vapors are cooled and condensed and the sulfurous acid

gas passes to a suitable draft outlet.

In treating large quantities of the sulfid I prefer to employ the rotary furnace shown in Figs. 4 and 5, in which 25 represents a cylindrical kiln lined with refractory material and mounted at a slight inclination upon suitable supporting rollers, said kiln being provided with gearing whereby it may be slowly rotated, as usual in kilns of this type.

At the upper or elevated end of the kiln is a feeding device 26 for the sulfid, and a pipe 27 for supplying fuel in the form of gas or 15 vapor, this fuel being preferably such that perfect combustion can take place in the kiln in order that the arsenic trioxid may not be discolored or rendered impure by admixture of carbonaceous matter or other

20 impurities therewith.

The kiln 25 delivers into a vertical receiver 29 having therein a series of brick checker work or other obstructions 30 and 31 with fuel pipes 32 and 33 for maintaining the same 25 in a properly heated condition. In connection with this receiver, I have shown a form of condensing apparatus different from that illustrated in Fig. 1, and consisting of a tower 35, of somewhat the same character, 30 as the Glover tower used in sulfuric acid manufacture, the lower end of this tower communicating through a flue 34 in the receiver 29, with the upper end of the latter, whereby the gases and vapors are discharged 35 from the receiver into the tower, and pass upwardly through the latter, being spread or scattered by checkerwork obstructions 36. During such flow the gases and vapors are subjected to the action of downwardly flow-40 ing volumes of water, introduced at the top of the tower, and the arsenic trioxid is carried down by the water flow to the bottom of the tower, where it accumulates until removed for drying. In many cases, this 45 alternative method is the preferable one, as any sulfur which may be carried off by the gases or vapors from the furnace will float upon the surface of the water at the base of the tower and can be skimmed off, while any 50 sulfuric acid can be removed by decanting the water from the oxid after the latter has settled.

In a furnace of the type shown in Fig. 4 the fused or melted sulfid flows in a stream down the inclined bed of the furnace, and is also carried up on the rising side of the same and permitted to flow back into the main stream. As there may, in consequence of this agitation of the mass, be a greater tendency on the part of the evolved vapors or gases to carry off particles of unreduced sulfid, I provide a series of reheating or regenerating actions by the use of the succes-

sive bodies of heated checker work in the re-

ceiver 29.

In the passage of arsenic vapors through a 65 condensing chamber, ersenic trioxid will condense at an earlier stage of such passage than will the sulfid, and in some cases I take advantage of this fact by subjecting the vapors to heat in their passage from a primary to a 70 secondary condensing chamber, one means of accomplishing this result being shown in Fig. 6, in which 22 and 22^a represent two condensing chambers, connected by a reheater similar to that shown in Fig. 1, and compris- 75 ing a contracted passage 19 formed between upper and lower slabs 16 and 17 which are interposed between upper and lower heat conducting flues 13 and 14. This supplementary treatment will, in many cases, effect 80 oxidation of sulfid which has escaped the preceding treatment, and will thus increase the percentage of oxid deposited in the secondary condensing chamber 22^a.

By my method of treating the sulfid of 85 arsenic, I am enabled to recover the arsenic trioxid in a commercially pure state, and therefore utilize what is at present not only a waste product of sulfuric acid manufacture, but also a source of annoyance in establish-90 ments which have no adequate means for its

disposal.

I claim:—

1. The mode herein described of recovering arsenic trioxid from sulfid of arsenic by 95 first subjecting the fused and flowing sulfid to the action of heat and then cooling the evolved gases to condense the same.

2. The mode herein described of recovering arsenic trioxid from sulfid of arsenic by 100 first subjecting the sulfid to the action of heat, then further heating the evolved gases and then cooling the gases to condense the

same.

3. The mode herein described of recover- 105 ing arsenic trioxid from sulfid of arsenic by first subjecting the sulfid to heat, then repeatedly reheating the evolved gases, and then cooling the gases to condense the same.

4. The mode herein described of recover- 110 ing arsenic trioxid from sulfid of arsenic, by first subjecting the sulfid to heat, and then cooling the gases to condense the same by

subjecting them to a water flow.

5. The mode herein described of recover- 115 ing arsenic trioxid from sulfid of arsenic by first heating the same, then subjecting the evolved gases to successive coolings to condense the same, and reheating said gases between successive coolings.

In testimony whereof, I have signed my name to this specification, in the presence of

two subscribing witnesses.

UTLEY WEDGE.

Witnesses:

HAMILTON D. TURNER, KATE A. BEADLE.