A. HOUGH.

APPARATUS FOR NITRATION OF ORGANIC LIQUIDS.

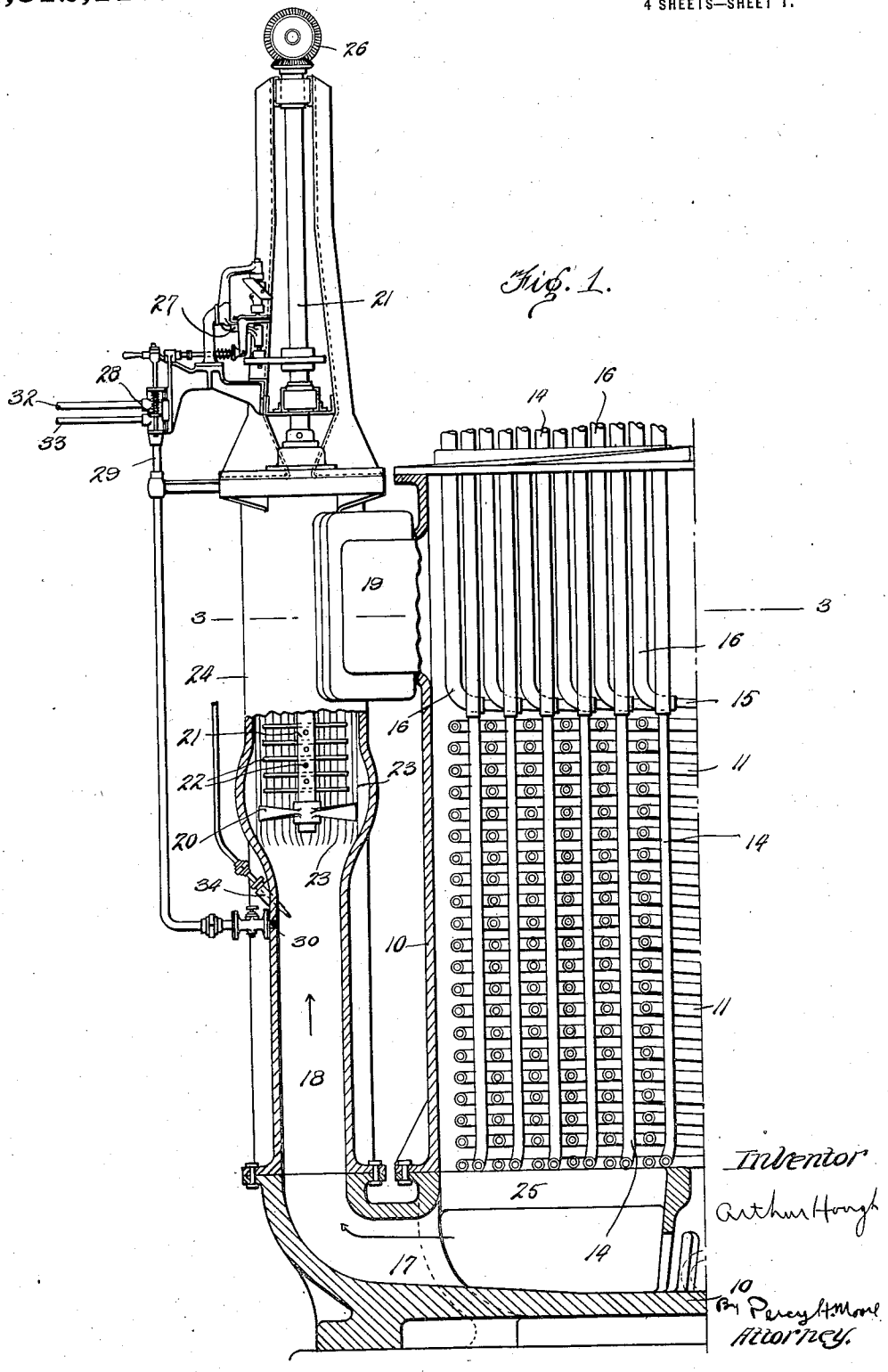
APPLICATION FILED MAY 18, 1918.

1,312,119.

Patented Aug. 5, 1919.

4 SHEETS—SHEET 1.

Fig. 1.



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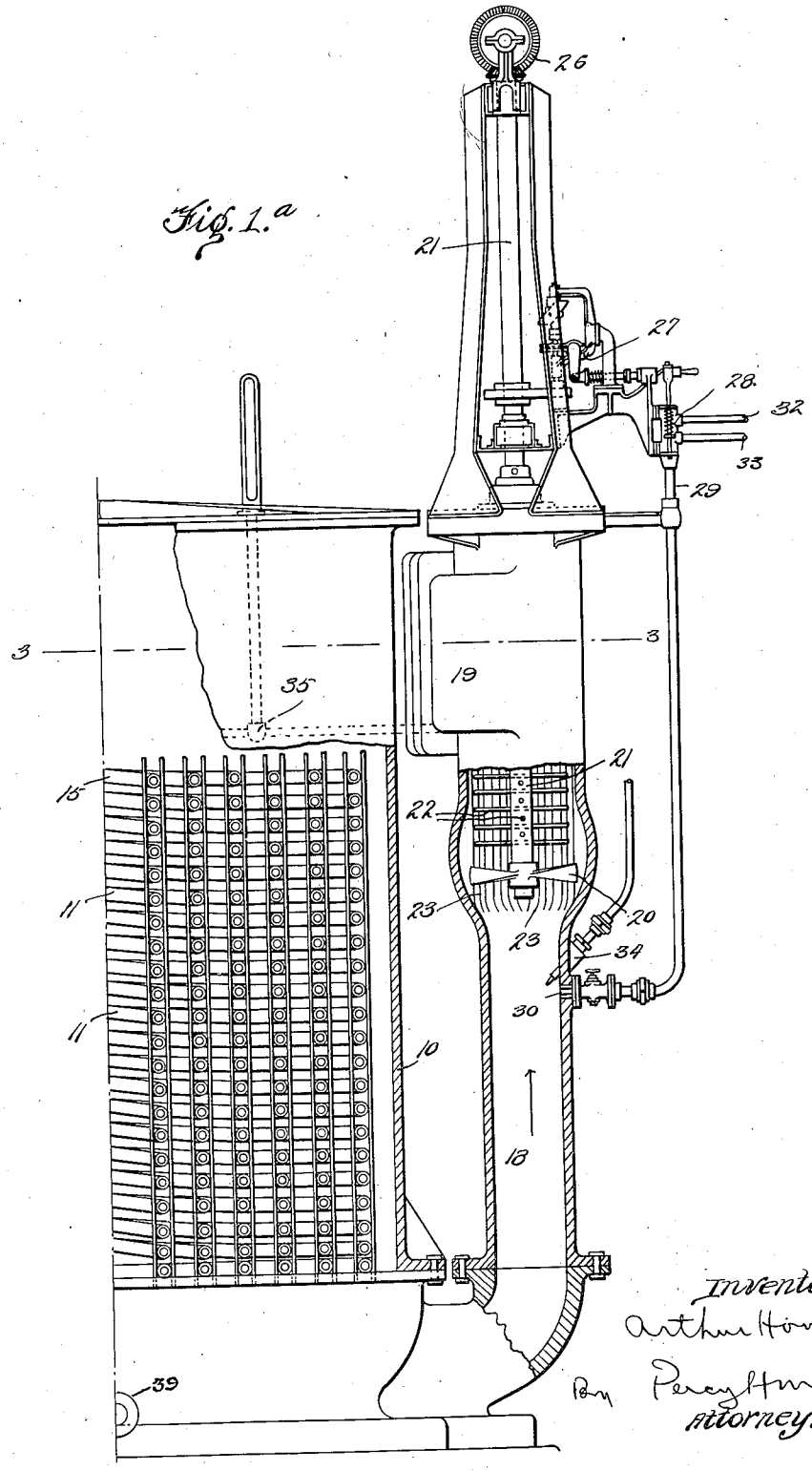
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4 SHEETS—SHEET 2.

Fig. 1.a



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4 SHEETS—SHEET 3.

Fig 2.

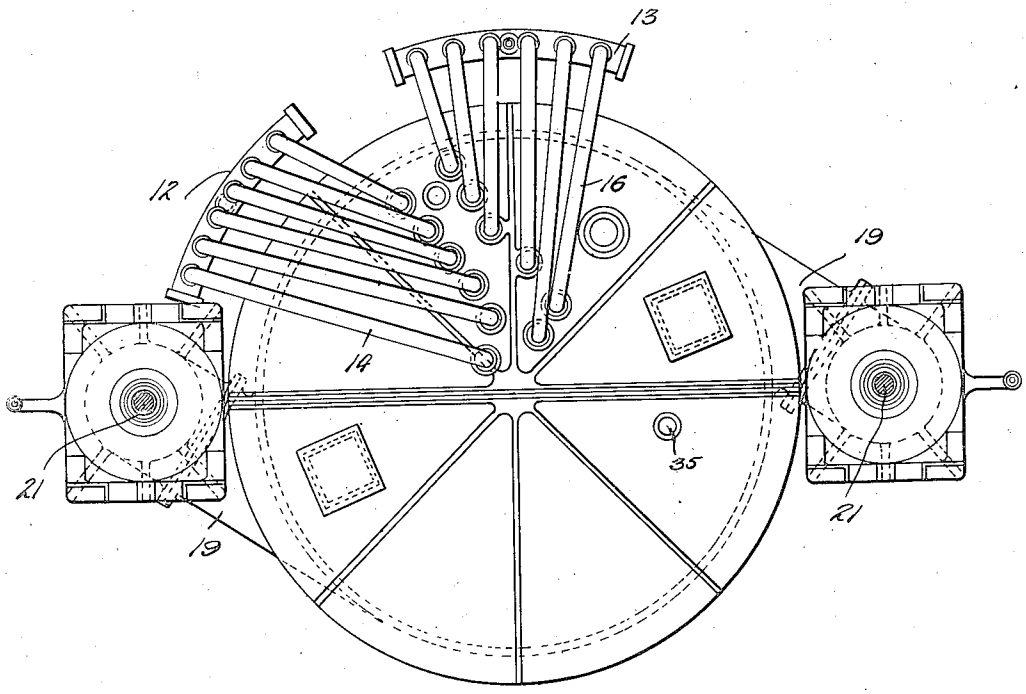
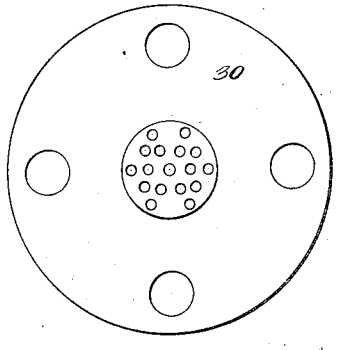


Fig. 4.



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4 SHEETS—SHEET 4.

Fig. 3.

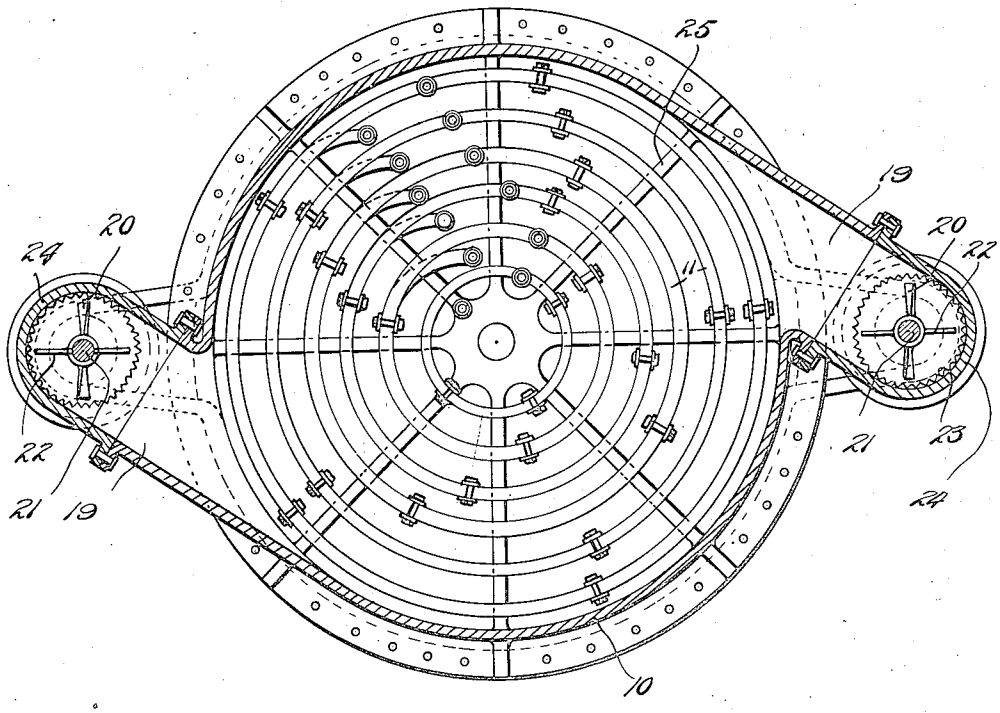
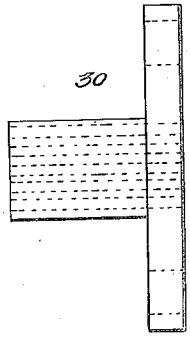


Fig. 5.



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UNITED STATES PATENT OFFICE.

ARTHUR HOUGH, OF NEW YORK, N. Y.

APPARATUS FOR NITRATION OF ORGANIC LIQUIDS.

1,312,119. Specification of Letters Patent. Patented Aug. 5, 1919.

Application filed May 18, 1918. Serial No. 235,369.

To all whom it may concern:

Be it known that I, ARTHUR HOUGH, subject of the King of Great Britain, residing at New York, in the county of New York and State of New York, have invented certain new and useful Improvements in Apparatus for Nitration of Organic Liquids, of which the following is a specification.

The present invention relates to an apparatus for nitration of organic liquid, such as toluol, benzol, glycerin and the like and the object of the invention is to produce an apparatus for the purpose specified which will be positive and dependable in its action, which will perform the nitration in a safe and expeditious manner, and which will be durable, and which will regulate the inlet of the organic material, in such a way as to prevent liability of undesirable decomposition. Other objects will be apparent from the description of the apparatus and process.

In the accompanying drawing:

Figures 1, and 1ª taken together show a central vertical section of an improved form of nitration apparatus falling within the scope of my invention;

Fig. 2 is a top plan view of the apparatus and showing the inlet and outlet for cooling agent;

Fig. 3 is a horizontal section of the tank and certain of its connections, said section being taken on the line 3—3 of Figs. 1 and 1ª;

Figs. 4 and 5 show respectively a rear end view and a side view of a nozzle suitable for injecting the organic liquid to be nitrated.

Referring more particularly to said drawings, 10 is a tank made preferably of cast iron, and provided with numerous cooling coils 11, through which a cooling liquid is circulated or caused to flow, the liquid being allowed to run in from the header 12, and being drawn off preferably by suction through the header 15. The pipes 14, leading from the header 12, pass directly downwardly to near the bottom of the tank, then being bent around to form the coils 11, from the upper end 15 of which, suitable risers 16 connect with the outlet header 13. The normal liquid level in the apparatus is slightly above the top of the coils, and the pipes 14 and 16, from about the level of the top of the coil upwardly to the top of the tank are provided with suitable sleeves, which may be made of steel if desired, for the purpose of preventing attack of the pipes themselves by the nitric acid condensed by the cold pipes thereupon. When these sleeves become attacked to a considerable extent they may be removed and new sleeves substituted. It is not necessary to similarly incase the pipes forming the coils themselves which are submerged within the liquid, since the liquid mixed acids will only slightly attack the pipes.

The tank 10 is initially filled, at least up to the bottoms of the tangential inlet passages 19, with nitrating mixture of any suitable composition, depending upon the particular material to be nitrated. This may conveniently consist of a mixture of nitric and surfuric acids.

Leading from the lower end of the tank 10 is an outlet 17, to a circulating system comprising the riser 18, and the tangential inlet 19, through which the liquid circulated again enters the nitrating tank. The liquid is forced upwardly through the riser 18, by the screw propeller 20, carried by the shaft 21, which shaft also carries suitable stirring arms 22, of which there may be any suitable number. The portion of the pipe 18, in which are mounted the propeller 20 and the arms 22 is internally fluted as shown at 23, for the purpose of preventing rapid rotation of the liquid passing therethrough, and thereby causing the power applied to the propeller 22 to have a greater lifting effect, whereby a greater amount of circulation of the liquid is produced. It will be noted that the circulating system consisting of the outlet 17, the lower portion 18 of the riser, the upper portion 24, and the inlet 19 are each of successively increasing diameters, and the portion of the riser adjacent the propeller 20 is of a larger diameter than the part 18 or the part 24 thereof. The reason for this is to produce a minimum amount of friction in the circulating system, whereby a given amount of power will produce a maximum amount of circulation.

The coils 11 do not rest upon the bottom of the tank, but upon a spider 25, thereby leaving a free space in the bottom of the tank in communication with the outlet 17.

The shaft 21 is rotated by suitable gearing 26, and is connected to a governor 27, which governor controls the compound valve 28, through which the liquid to be nitrated or air enters the pipe 29 and are injected through the nozzle 30 into the riser at a point below the stirring arms 22 and also

preferably below the screw propeller 20. The valve 28, when fuly opened for the passage of liquid, allows the passage through the pipe 29 of an amount of liquid to be nitrated, approximately equal to .1% of the amount of liquid flowing through the riser, or in other words for each volume of liquid entered through the pipe 29 and nozzle 30 there are about 1000 volumes of liquid passing through the circulating system. By the action of the screw 20 and the blades 22, the nitrating acid and the liquid to be nitrated are very thoroughly mixed, and the amount of heat given off is so small that this is readily absorbed by the large quantity of acid, without any considerable (or even any appreciable) rise in temperature. The governor is so arranged that as soon as the speed of the shaft 21 falls below a predetermined rate, the valve from pipe 32, through which the organic liquid enters will be immediately closed, and the valve from pipe 33 (connected to a source of compressed air) will be opened, thereby forcing out of the pipe 29, the quantity of liquid contained therein, through the nozzle 30 and into the riser, which air also produces a desired amount of agitation in the riser. When the valve from pipe 32 closes, and the valve from pipe 33 opens, the change of location of the parts of the governor, caused by the speeding up the shaft 21 first opens the valve from 32 and subsequently closes the valve from 33. To carefully watch the temperature, the thermometers are located at 34, in proximity to the liquid inlet, and also at 35 in the upper portion of the tank just below the normal liquid level therein. On account of the large surface of the cooling coils 11, it is easy to reguate the temperature of the mixed acid and reacting materials in the tank and circulating system. It will be noted from the drawings that two circulating systems are shown, although obviously a larger number could be provided if desired.

When the nitration operation is completed, the reaction products can be withdrawn in any convenient manner, for example, through the pipe 39, (Fig. 1[a]).

From the foregoing it will be seen that my invention involves a novel method of nitrating, as well as an apparatus therefor, said method involving the introduction of the liquid to be nitrated into a tunnel in which a rapidly flowing stream of nitrating acid is immediately mixed with the organic liquid so introduced, whereby the heat is given out in such a manner as to not appreciably raise the temperature of the mixed acid, whereby the nitration process is made perfectly safe.

It will be obvious that the method and apparatus are not restricted to the nitration of any one particular organic liquid, but that the same is applicable to various liquids. While I have referred to air being introduced through the pipe 33, any other inert gas could obviously be substituted.

It is obvious that the invention is not restricted to the specific embodiment hereinbefore described in detail, but that various changes within the scope of the appended claims are contemplated.

What I claim is:

1. An apparatus for nitrating organic liquids, comprising a tank, a circulating connection between the lower portion and the upper portion thereof, cooling coils in said tank and means for passing a cooling fluid therethrough, said circulating connection being provided with propelling means adapted to cause the liquid to flow upwardly through said connection, and with stirring means adapted to completely mix the materials passing therethrough, an inlet for the organic liquid to be nitrated located below said stirring means, and a tangential inlet from said circulating connection to said tank.

2. A nitration apparatus comprising a tank provided with cooling means, a circulating system comprising an outlet from the lower part of said tank, a riser and a tangential inlet to said tank at a point above the outlet thereof, a propeller and mixer in said riser, an inlet to said riser at a point below said mixer, for material to be nitrated, and a controlling means for said inlet regulated by the speed of the said mixer.

3. A nitration apparatus comprising a tank having cooling means, a circulating system comprising an outlet, a tunnel and a tangential inlet, propelling and stirring means in said circulating system, an inlet for liquid to be nitrated to said circulating system at a point in advance of said stirring means, and coöperating means whereby the stopping of the propelling means cuts off the inlet of liquid and forces in an inert gas.

4. A circulatory system for a nitration tank comprising a tunnel, a rotary propeller, and stirring arms rotated therewith, said propeller and arms being carried by a rotary shaft, means for applying power for rotating said shaft, an inlet in said tunnel for material to be nitrated, a regulating valve for said inlet, a governor connected to said rotary shaft, and a connection between said governor and said regulating valve, for closing said valve when the speed of rotation of said shaft falls below a predetermined limit.

5. A circulating system connected to a nitrating tank, said system comprising a tunnel having a rotary propeller therein, and flutings on the interior surface of said tunnel in the vicinity of the propellers to prevent excessive rotation of the liquid passing

through said tunnel and being forced along by said propeller.

6. A nitration tank having cooling coils therein, the portion of said coils which are above the normal liquid level being provided with a sleeve to prevent excessive corrosion of that portion of the said coils by acid vapors.

7. A method of nitrating organic liquids which comprises providing and maintaining a body of nitration acid in contact with cooling means, continuously drawing out of said body from its lower part, a stream of such cooled acid, raising the same and reintroducing the same tangentially into the main body of acid at a level above that at which it was withdrawn, introducing into said circulating stream of acid an amount of material to be nitrated which is not greater than one one-thousandth of the volume of the stream of acid so circulated, and thoroughly mixing the said material with the said acid immediately after being so introduced and before the said acid again comes into contact with the main body of nitrating acid, while controlling the inlet of said material in a predetermined ratio to the speed of circulation of said stream of acid.

In testimony whereof I affix my signature in presence of two witnesses.

ARTHUR HOUGH.

Witnesses:
A. G. SIMONS,
SADIE E. LETHAM.