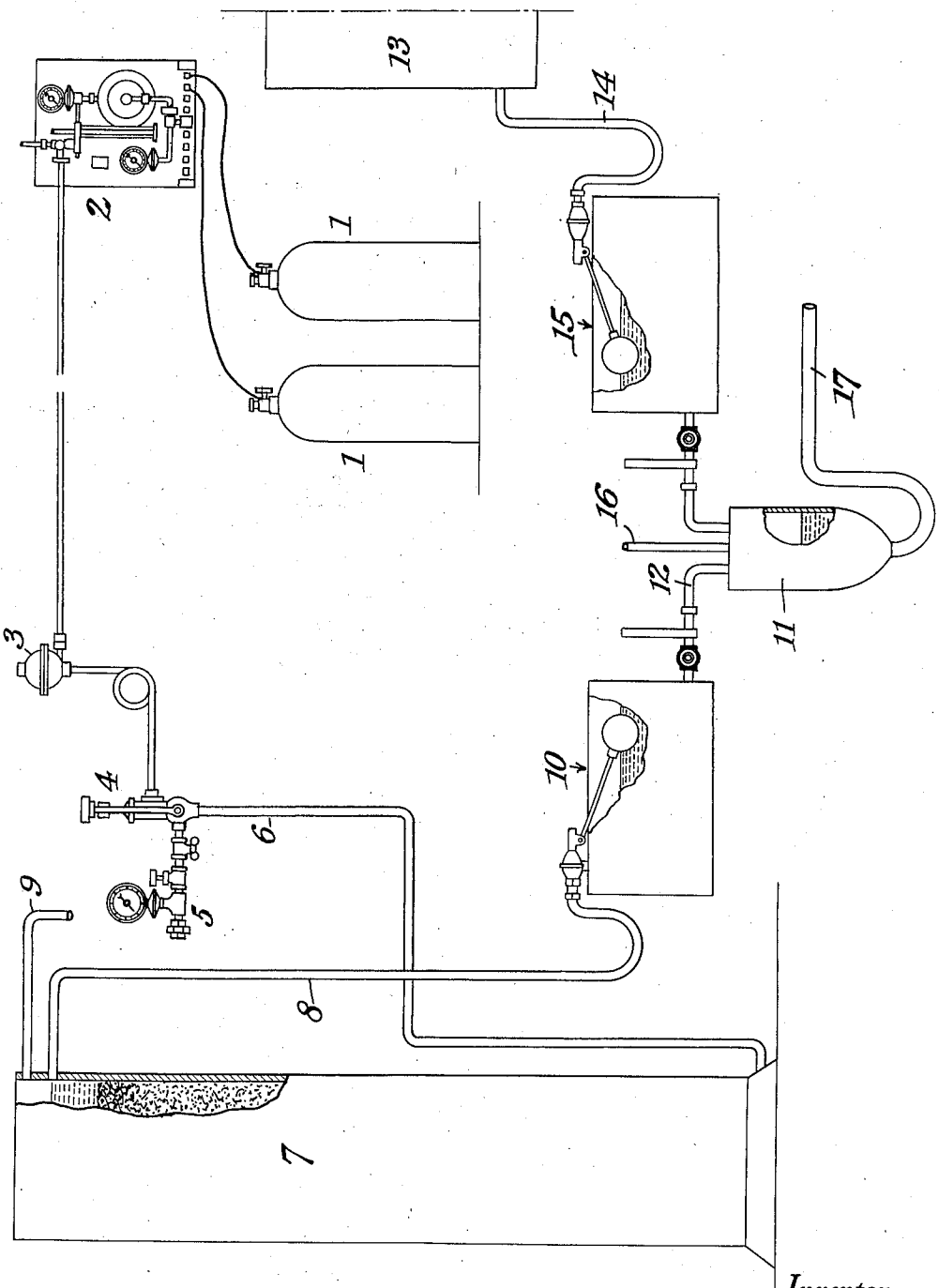
J. C. BAKER.

PROCESS OF STERILIZING.

APPLICATION FILED SEPT. 13, 1919.

1,378,644. Patented May 17, 1921.



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UNITED STATES PATENT OFFICE.

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PROCESS OF STERILIZING.

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Application filed September 13, 1919. Serial No. 323,650.

To all whom it may concern:

Be it known that I, JOHN C. BAKER, a citizen of the United States of America, residing at Ridgefield Park, Bergen county, State of New Jersey, have invented certain new and useful Improvements in Processes of Sterilizing, of which the following is a full, clear, and exact description.

It has been proposed and experiments have heretofore been conducted for utilizing nitrogen trichlorid (NCl_3) as a sterilizing agent, but the processes followed and the results obtained have led to a very generally accepted belief in its uselessness for this purpose. This conclusion, I now have reason to believe, was largely based upon a failure to produce real nitrogen tri-chlorid and a failure to produce what purported to be this compound in a condition in which it could be practically applied and used for the purpose.

Nitrogen tri-chlorid as a chemical compound, has been long known, but because of the difficulty of producing it, and because of its explosive character it has not been practicable to make and preserve it for any useful purpose. I have discovered, however, that as a sterilizing or disinfecting agent it has remarkable and valuable properties, being nearly as rapid in its action as chlorin, and in cases where a large or more than the usual amount of chlorin is required to effect sterilization or disinfection, that is to say over 0.1 to 0.3 p. p. m., it may be used in its stead with great economy and success. In certain work under my supervision, for example, at Ottawa, Canada, 1.8 p. p. m. of chlorin were required for sterilization, but I accomplished the same result with 0.5 p. p. m. of nitrogen tri-chlorid, thus securing marked economy of operation, and I have further found that in certain liquids such as sewage or other chlorin consuming liquids, sterilization may be effected with a much less proportion of nitrogen tri-chlorid than would be required of chlorin.

My discovery that nitrogen tri-chlorid is a valuable and efficient sterilizing agent owes its value and importance, moreover, to a process or method which I have devised for producing and using it, which process involves not only a novel manner of making the compound, but its immediate application or use for sterilizing, as it may not be practically accumulated or stored, and according to my invention I proceed as follows:

I dissolve chlorin gas in water in the proportion of 0.1 to 0.5% of chlorin to water, and pass this aqueous solution through a body of crushed limestone until the chlorin is all converted to hypochlorous acid and the hydrochloric acid (HCl) all neutralized. The reaction may be thus expressed:

(1) $Cl_2 + H_2O \rightleftarrows HOCl + HCl$

(2) $2HOCl + 2HCl + CaCO_3 \rightarrow 2HOCl + CaCl_2 + H_2CO_3$

The first named reaction is reversible, and using the quantities specified does not go to completion, but may be completed by the neutralization of the hydrochloric acid by the action of the limestone. This acid after conversion to weak carbonic acid (H_2CO_3) will become finally converted by prolonged contact with the limestone to calcium bicarbonate ($Ca(HCO_3)_2$).

The active material in the resulting solution is hypochlorous acid (HOCl), which is thus obtained free from chlorin (Cl_2) or hydrochloric acid (HCl), and in concentration sufficient for economical working conditions. The solution resulting from this action of the limestone is then mixed in measured quantity with ammonia or any solution containing a known quantity of ammonia or ammonium compounds such as ammonium hydroxid, chlorid or sulfate or other compound, and the mixture results in the formation of nitrogen tri-chlorid (NCl_3), as shown by the following equation:

$$NH_4OH + 3HOCl \rightarrow NCl_3 + 4H_2O$$

or

$$NH_4Cl + 3HOCl \rightarrow NCl_3 + 3H_2O + HCl$$

In these equations the proportion by weight of available chlorin to ammonia (NH_3) is 12.5 to 1, and the resulting product may be used under proper condition, as I shall hereinafter more fully set forth as a highly useful and efficient sterilizing agent for many purposes.

In order that my process may be successfully carried out, I have annexed hereto a drawing which illustrates diagrammatically an apparatus by means of which the invention may be practised.

I use containers 1, 1 of chlorin which passes as may be desired or controlled to a chlorin meter 2. From there the gas passes through a check valve 3 to an injector 4, by

means of which it is passed into a regulated flow of water entering through a valve controlled inlet 5 in a pipe line 6 when it combines with the water to form a chlorin solution.

This solution is introduced into a tower or extended chamber or receptacle 7 containing crushed limestone, and by the action of which it is converted into hypochlorous acid. This latter solution is taken off by a pipe 8, an overflow pipe 9 being provided for use in case of need, and by such pipe it is delivered into a constant level box or chamber 10.

From this latter it is passed in regulated quantity into a mixing chamber 11 by a pipe 12, provided with valves and a gage, and entering the top of said chamber.

I also use an ammonia tank 13 for containing the ammonia or ammonium solution and connect this tank by a pipe 14 with a constant level box or receptacle 15 from which a pipe with valves and a gage to regulate the flow of ammonia leads into the top of the mixing chamber.

To facilitate the mixture of the two ingredients a pipe 16 with a water jet enters the mixing chamber and the resulting solution is led off by a pipe 17 to the water, sewage or other material which is to be sterilized in the ordinary or any well-known manner.

All parts of this apparatus are of well-known character and are widely used in other processes, particularly those involving the use of chlorid as a sterilizing agent.

The resulting solution, if the rates of supply of the hypochlorous acid and ammonia be properly determined and regulated, will contain nitrogen tri-chlorid in an aqueous solution, and it may be used for sterilizing water, milk, sewage, or other articles or materials. The compound is exceedingly volatile and by spraying or otherwise subdividing the solution it may be driven off as a gas, and utilized in this condition as a sterilizing agent, especially for solid articles, such as fruits and the like.

I have not shown any special mechanism or appliances for sterilizing and none are necessary. The mere mixture of the solution with a fluid in definite proportions effects its sterilization, or the mere exposure of articles to the gas effects the same result.

What I claim is:

1. The process of sterilizing herein described, which consists in bringing together continuously during the period and at the rate of the use of the product the materials necessary for the production of an aqueous solution of nitrogen trichlorid, and utilizing the same as it is produced as a sterilizing agent.

2. The process herein described of sterilizing which consists in producing hypochlorous acid, mixing therewith ammonia or ammonium compounds in measured proportions and utilizing the solution of nitrogen-tri-chlorid resulting therefrom immediately as produced as a sterilizing agent.

3. The process of sterilizing herein described, which consists in continuously bringing together during the period and at the rate of the use of the product the materials necessary for producing an aqueous solution of nitrogen trichlorid, forcing air through the solution as produced and applying the gas as the sterilizing agent.

4. The process of sterilizing herein described which consists in mixing in measured quantities hypochlorous acid and ammonia or ammonium compounds and utilizing the nitrogen tri-chlorid resulting from such mixture immediately as produced as the sterilizing agent.

In testimony whereof I hereunto affix my signature.

JOHN C. BAKER.