

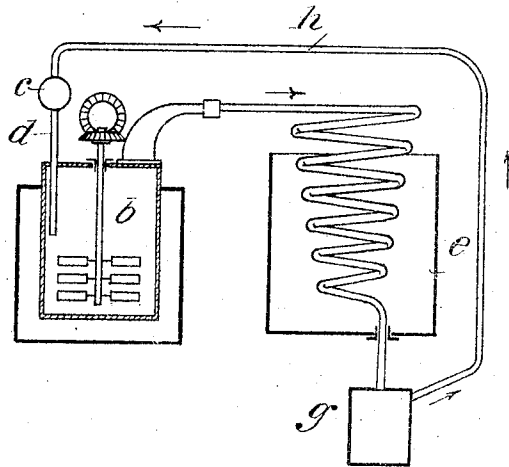
J. WEISE.

PROCESS FOR THE PREPARATION OF FORMIC ACID FROM THE REACTION MIXTURE OF
FORMATES AND MINERAL ACID.

APPLICATION FILED JULY 10, 1912.

1,043,985.

Patented Nov. 12, 1912.



Witness:
L. H. Cadwall.
C. D. Brown.

Inventor
Johannes Weise
by Foster Freeman Watson & Co.
Attys

UNITED STATES PATENT OFFICE.

JULIUS WEISE, OF WIESBADEN, GERMANY, ASSIGNOR TO THE FIRM OF RUDOLPH KOEPP & CO., OF OESTRICH-ON-THE-RHINE, GERMANY.

PROCESS FOR THE PREPARATION OF FORMIC ACID FROM THE REACTION MIXTURE OF FORMATES AND MINERAL ACID.

1,043,985.

Specification of Letters Patent.

Patented Nov. 12, 1912.

Application filed July 10, 1912. Serial No. 708,653.

To all whom it may concern:

Be it known that I, JULIUS WEISE, a subject of the German Emperor, and resident of Wiesbaden, Germany, have invented certain new and useful Improvements in Processes for the Preparation of Formic Acid from the Reaction Mixture of Formates and Mineral Acid, of which the following is a specification.

10 The object of the present invention is a process for the preparation of formic acid with a yield which could not be attained hitherto.

It is well-known that formic acid is prepared by mixing sodium formate with sulfuric acid. In this process a very thick pulp or paste is formed consisting of pulverulent sodium sulfate and liquid formic acid. If the latter is distilled off, then under heating the pulp or paste the distillation is fairly rapid at first. Soon, however, the mass breaks up into a moist powder, and then the process does not proceed any further. In order to carry over the still remaining formic acid into the receiver hitherto a vacuum has been made use of. This however has essential drawbacks, because with the use of a vacuum when manufacturing on a large scale it is, as is well-known, not possible to so close the apparatus that air does not continually get into the vacuum through leaks, so that the air pump has to be continually kept working. This is of little consequence for ordinary working processes and only requires a greater consumption of power, viz., for keeping the air pump continually going. However, when preparing formic acid this is different. Formic acid has such an affinity for air that it can be only separated from same with difficulty, and that such a separation in an efficient manner is even practically impossible. In consequence of this when using a vacuum a very considerable amount of formic acid is carried away by the air pump with the evacuated air. This amount is so considerable that the preparation of formic acid as hitherto effected was very unsatisfactory economically, but nevertheless the industry knew no other means for obtaining a more economical yield.

In consequence of the above-mentioned resistance which formic acid obtained in a thick pulp opposes to the distillation and

of the failure of a vacuum for this purpose, 55 there remains no other means for the workman-manager than the well-known method of effecting the distillation with the use of a strong current of a volatile substance which mechanically carries with it the body to be 60 distilled off.

The employment of a mechanically acting current of steam, used in similar cases is entirely excluded in the present case, as the use of such current of steam would dilute 65 the formic acid, in a most undesirable manner. It suggests itself to use for the purpose in question instead of a current of steam, a strong current of air. Against this, however, stands the great obstacle of the 70 already mentioned affinity of formic acid for air, which renders impossible a subsequent separation of the air from the formic acid carried therewith and thus leads to large losses of formic acid. Now it has been 75 found that this obstacle can be effectively overcome, and that it is possible to carry out the preparation of formic acid from formates and mineral acids with an almost theoretical yield, by inserting in a manner 80 already known in other industries the whole distillation apparatus: reaction vessel, cooling coil, receiver for the liquid formic acid, into the closed circuit of one and the same air current constantly kept in motion by a 85 ventilator or the like.

The air current is for instance driven by the ventilator into the reaction vessel, from there through the cooling coil, and then into the receiver from there to return to the ventilator, and to assume its new course through the apparatus. In this case the circulating air acts in such manner that it dissolves the formic acid adhering as moisture to the sodium sulfate and always takes the same up 95 again to the saturation point, then to give it off in the cooling device. In the latter it naturally only gives off that part of the acid which the hot saturated air-acid-mixture issuing from the distilling vessel can 100 no longer retain at the saturation temperature prevailing in the cooling vessel, but this does not adversely effect the ultimate yield of acid, in consequence of the continuous cyclic motion of the mixture. It is 105 evident that always the same amount of air is driven through the apparatus and the formic acid carried with the air but not

condensed in the cooling vessel, is always led back into the reaction vessel. By suitably closing off the various vessels against the atmosphere care is taken that outer air does not get to the circulating current of air.

In the drawing the apparatus described is represented as far as this is necessary for understanding same.

10 Above the reaction vessel *b* provided with a steam jacket and stirring device the ventilator *c* is arranged. The latter stands in communication with the interior of the reaction vessel by the tube *d*. The air delivered by the ventilator flows from *b* to the cooling coil *e* carrying with it the gaseous formic acid formed and then flows to the receiver *g* from where it returns through the suction tube *h* to the ventilator *c* and thence into the reaction chamber *b*. This arrangement has given very satisfactory results in practice. The distillation is effected in a surprisingly rapid manner, a loss is prevented, as any acid carried away from the receiver is led back into the reaction chamber as already explained. Instead of air naturally other gases can be employed which are indifferent to the distillate.

30 In the literature already processes have become known according to which the distillation of certain substances is effected in a cyclic process with the use of inert gases. However, it has not become known to employ such a process for the distillation of

formic acid and thereby to avoid the difficulties which hitherto were connected with the preparation of formic acid and which have been described above.

As already mentioned it is not new to obtain in distillation processes an acceleration by leading an indifferent gas or the like through the apparatus in a closed circuit. However, the industries in which this method of working has become known are so far removed from the preparation of formic acid from formates that the application of this method of working for overcoming the difficulties inherent to the preparation of formic acid was in no way obvious.

What I claim as new and desire to secure by Letters Patent is:—

A process for the preparation of formic acid from the reaction mixture of formates and mineral acid by distillation, consisting in leading a current of a gas indifferent to the reaction mixture and its final product, in a cyclic current through the whole distillation apparatus and the reaction chamber connected therewith which are both closed off against the access of outer air.

In testimony whereof I have signed my name to this specification in the presence of two subscribing witnesses.

JULIUS WEISE.

Witnesses:

ERNST JOSEF WESTHOPF,
ING LUDWIG HUCKERT.