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Fig. 1.

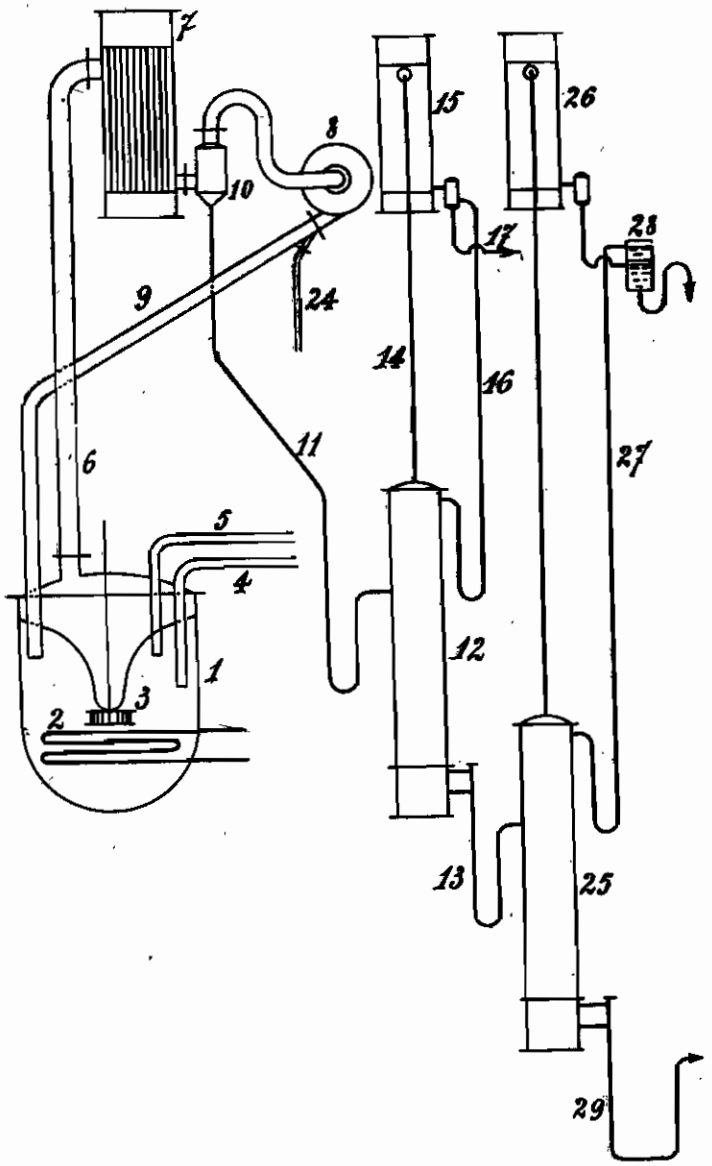

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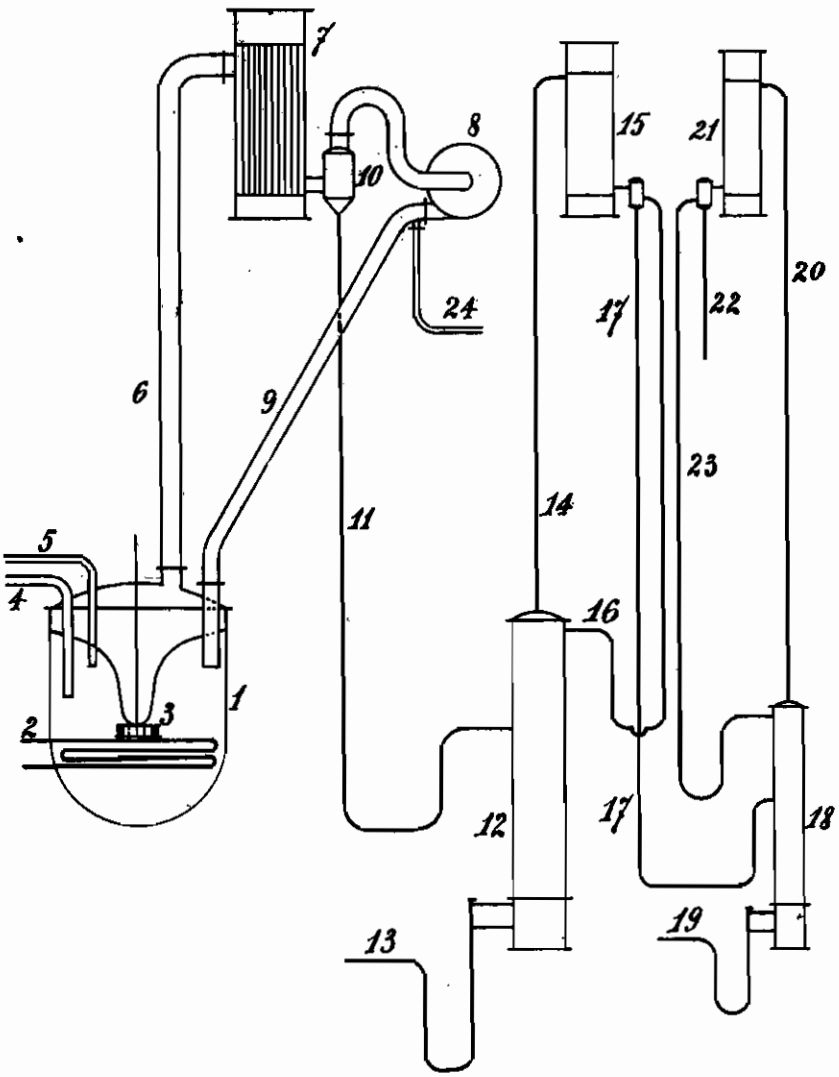
Fig. 2



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ALIEN PROPERTY CUSTODIAN

MANUFACTURE OF ALIPHATIC ACIDS

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It is known to oxidise acetaldehyde, previously diluted with acetic acid, by means of a current of air or inert gases containing oxygen in more or less considerable quantity. The reaction is generally activated by catalysts, such as salts of manganese, cobalt, copper, cerium and the like.

The operation is mostly carried on in a continuous way, aldehyde being caused to arrive simultaneously with the gas containing oxygen into a reaction vessel provided with stirring means for finely emulsifying such gas and the liquid under treatment.

Generally, the products obtained from the oxidation of aldehyde are separated by distillation from the liquid which is caused to escape by overflow of the reaction vessel into which the air or gas of reaction and aldehyde to oxidise are introduced.

This way of operating presents inconveniences which consist in withdrawing from the reaction medium a certain quantity of catalyst and complicating the distillation by reason of residual deposits formed in the evaporation system and containing peroxidised derivatives which deteriorate metals forming such system.

The present invention has for its object a process which permits not only avoiding the inconveniences referred to, but also improving operative yields hitherto generally obtained.

The invention essentially consists in carrying the acid manufactured in the reaction vessel with a current of gas circulating between this vessel and a well cooled condenser. The intensity of the gaseous current is adjusted in such manner as to cause the quantity of acid carried therewith and condensed to correspond to the quantity manufactured. In this way the volume of the liquid within the reaction vessel remains substantially constant, and the catalyst is not carried away from the vessel, whereby its proportion may be increased to attain the favorable effect desired and the catalyst introduced may last indefinitely.

The condensed product is then separated down to its elements and dehydrated by distillation, eventually in accordance with the principles of azeotropic distillation.

It is also known to be advantageous to effect the reaction of oxidation of acetaldehyde diluted with acetic acid by operating at a moderate temperature, for example between 30 and 60° centigrade. Above such temperature the quantity of carbon dioxide formed by the combustion of acetaldehyde will increase very rapidly and the respective yields will decrease.

When the operation is carried on at such temperature a current of air containing oxygen necessary to the oxidation of aldehyde is not capable of carrying away with it the quantity of acetic acid vapours corresponding to that which has been formed. It is for this reason that, according to the present invention, the gaseous mixture grown poor in oxygen and having been deprived of products of the oxidising reaction is caused to circulate in the vessel of reaction. In this way there is brought about the complete removal of acetic acid formed by the oxidation of acetaldehyde in the bath.

The process of this invention is also utilisable in the case of aliphatic aldehydes containing more than 2 carbon atoms in the molecule.

In order to show how the invention may readily be carried into practical effect, the following examples are given for the purpose of illustration, but not of limitation, with reference to the accompanying drawings in which:

Figure 1 is a schematic representation of a plant for the manufacture of acetic acid according to the invention, and

Figure 2 is a schematic representation of a plant for the manufacture of propionic acid according to the invention.

EXAMPLE I.—*Manufacture of acetic acid.*

1 metric ton of crystallizable acetic acid containing 25 kgs of acetaldehyde and 1 kg. of acetate of manganese is introduced into a reaction vessel 1 (Fig. 1) provided with a pipe coil 2 for heating or cooling and a turbine member 3 for finely emulsifying the gas and liquid within the vessel 1. The liquid in the latter is heated by means of the coil 2 to the temperature of 60° Centigrade and then there are introduced into the liquid through a pipe 4 a current of air at the rate of 270 cubic meters per hour and through a pipe 5 acetaldehyde at the rate of 150 kgs per hour. The reaction of oxidation sets in and soon it becomes necessary to cool by introducing cold water into the coil 2 in order to maintain the temperature within the vessel 1 at about 50°C.

Hot gases leaving the oxidiser 1 through a conduit 6 are cooled in a condenser 7 at the outlet end of which gases grown poor in oxygen and separated in a separation head 10 are taken up by a fan or blower 8 by which they are blown through a conduit 9 back into the reaction vessel 1. The blower 8 is so adjusted as to discharge 800 cubic meters per hour, such discharge being sufficient to extract out all of acid formed in the reaction at the temperature of 50°C.

In the separation head 10 the condensed liquid, which is constituted by a mixture of

	Per cent
Acetic acid	97
Acetaldehyde	2
Water	1

is separated from the poor gases taken up by the blower 8 and flows thereout through a pipe 11 at the rate of 135 kgs per hour.

This liquid is introduced into the median portion of a distillation column 12 ahead of which through a pipe 17 there is withdrawn acetaldehyde which is reintroduced into the reaction vessel 1. At the base of said columin there flows out through a pipe 13 hydrated acetic acid at a concentration of about 98% which is then dehydrated azeotropically in a second distillation column 25 once for all charged with a suitable quantity of a carrier such as ethyl acetate.

The azeotropic mixture ethyl-acetate-water, after condensation at 26, is decanted at 28. The aqueous layer is drawn off, while the upper layer mainly constituted by ethyl acetate is continuously returned through a pipe 27 to the top portion of the column 25. At the base of the latter there is collected at 28 anhydrous acetic acid.

The total efficiency of the oxidation is 98,6%.

Through a pipe 24 connected with the discharge end of the blower 8 flows off a gas including still 5 to 6% of oxygen, from which acetaldehyde and acetic acid vapours contained therein are removed by simple washing or by other known means.

EXAMPLE II.—*Manufacture of propionic acid*

The reaction vessel 1 (Fig. 2) is charged with 1000 kgs of propionic acid containing 50 kgs of propionaldehyde and 5 kgs of propionate of manganese.

The mixture being heated up to 75° C., there is introduced into the vessel 1, on one part, propionaldehyde at the rate of 125 kgs per hour and, on the other part, an air current at the rate of 200 cubic meters per hour.

As soon as the reaction sets in, the temperature of the liquid mixture in vessel 1 is adjusted to 60° C. and the formed products are caused to be eliminated by means of the gaseous current produced by the blower 8 which is adjusted to discharge 750 cubic meters per hour.

The product condensed at 7 and constituted by propionic acid including propionaldehyde and a small quantity of water is introduced into a distillation column 12 at the base of which there is collected at 13 anhydrous propionic acid.

At the top of column 12 through a pipe 14 escapes a mixture of propionaldehyde, water and propionic acid, which mixture is condensed at 15 and partially returned through a pipe 16 to the top portion of column 12. The remainder of this condensation is introduced through a pipe 17 into the median portion of a distillation column 18 at the base of which there is collected at 19 anhydrous propionic acid, while ahead of this column at 22 is drawn off a mixture of propionaldehyde and water, which is separated by known means down to its elements, propionaldehyde being reintroduced into the reaction vessel 1.

The total efficiency of the oxidation is 98,7%.

HENRI MARTIN GUINOT.