

UNITED STATES PATENT OFFICE.

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YORK, N. Y., A CORPORATION OF NEW YORK.

PROCESS FOR THE MANUFACTURE OF GLYCOLIC ACID.

No. 837,083.

Specification of Letters Patent.

Patented Nov. 27, 1906.

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To all whom it may concern:

Be it known that I, OTTO LIEBKNECHT, a
subject of the German Emperor, and a resi-
dent of Frankfort-on-the-Main, Germany,
5 have invented a certain new and useful Proc-
ess for the Manufacture of Glycolic Acid, of
which the following is a specification.

This invention relates to an economical,
easy, and effective process of manufacturing
10 glycolic acid.

The processes heretofore practiced of man-
ufacturing glycolic acid are not adapted for
the technical manufacture of the same. For
instance, it is known that glycolic acid can be
15 manufactured on a large scale from mono-
chloroacetic acid; but this process is not an
easy one and is very expensive. As a fur-
ther illustration, it is impossible to economic-
ally produce glycolic acid from oxalic acid
20 and zinc-dust because the yield is poor and
the zinc-dust very expensive.

I have discovered that oxalic acid in sul-
furic-acid or hydrochloric-acid solution is re-
duced electrolytically nearly quantitatively
25 and with a very good current yield. In prac-
ticing my process I have found it most satis-
factory in reducing the oxalic acid electro-
lytically to use electrodes having a high ca-
thodic overvoltage—as, for instance, lead
30 electrodes—and to use a diaphragm to sepa-
rate the anodic and cathodic liquors. Both
of these features are important, as well as the
use of a not too dilute acid. An electrode
not having a high cathodic overvoltage
35 would give insufficient yields, and a too di-
lute acid would cause the oxalic acid, being
highly ionized, to take too great a part in the
conductivity of the current and to cause it to
go to the anode, where it would be oxidized.

40 As an example illustrative of my process I
give the following: Seven hundred parts of
crystallized oxalic acid are dissolved in about
three thousand three hundred parts of water
and eleven hundred parts of thirty-per-cent.
45 sulfuric acid are added while stirring. This
solution forms a cathodic liquor, which it is
of advantage to keep warm during the proc-
ess of electrolyzation. The cathodic liquor
should be placed in the cathodic compart-
50 ment of a suitable electrolytic apparatus
provided with a suitable diaphragm, and the
anodic liquor, comprising a thirty-per-cent.
sulfuric acid, should be placed on the other

side of the diaphragm. The density of the
current at the cathode may vary greatly— 55
for instance, from twenty-five to two hun-
dred and fifty amperes per square meter of
surface of cathode. The presence of the dia-
phragm in connection with the use of a not
too dilute acid largely prevents the anodic 60
oxidation of the oxalic acid. On completion
of the electrolysis the anodic solution may be
used in preparing a fresh charge of the ca-
thodic liquor.

It is preferable to stir the cathodic liquor 65
during the electrolyzing, and, furthermore,
to add fresh acid in case the acid in the ca-
thodic liquor becomes too weak, a too concen-
trated solution, however, being avoided at
the cathode on account of the resulting low 70
conductivity. It is advisable not to use sul-
furic acid of a strength below fifteen parts
concentrated sulfuric acid to one hundred
parts of water, as otherwise the current yield
and the nature of the product will be affect- 75
ed. The sulfuric acid can in the examples
given be substituted for by about a twenty-
per-cent. hydrochloric acid. In place of
lead electrodes carbon or graphite can be
used; but in using carbon a proportionately 80
larger amount of current will have to be pro-
vided. Besides this other undesirable reac-
tions seem to take place simultaneously.

In order to produce glycolic acid from the
electrolyzed solution in case sulfuric acid has 85
been used, the solution is neutralized with
lime, best added while stirring, until all the
sulfuric acid and oxalic acid present is neu-
tralized by the lime. In order to remove the
last traces of sulfate of calcium, I use barium 90
carbonate and oxalic acid in the usual way.
In case hydrochloric acid has been used in-
stead of the sulfuric acid during the electroly-
sis it is only necessary to evaporate the hydro-
chloric acid in order to get glycolic acid. 95
The glycolic acid obtained by this process
may be used for technical as well as phar-
maceutic purposes.

As it is obvious that the process may be
practiced in a number of different ways with 100
considerable variation, I do not restrict my-
self to the steps or proportions described; but

What I claim, and desire to secure by Let-
ters Patent of the United States, is—

1. A process for the manufacture of gly- 105
colic acid from oxalic acid consisting in the

electrolytic reduction of oxalic acid in the cathode-compartment of an electrolytic apparatus in the presence of electrodes having a cathodic overvoltage, the oxalic acid being dissolved in a suitable dilute acid kept warm during the reduction and of such a degree of concentration that it excludes the oxalic acid from substantial participation in the conductivity.

10 2. A process for the manufacture of glycolic acid consisting in the electrolytic reduction of oxalic acid in the cathode-compartment of an electrolytic apparatus in the pres-

ence of lead electrodes, the oxalic acid being dissolved in a dilute sulfuric acid kept warm during the reduction and of such a degree of concentration that it excludes the oxalic acid from substantial participation in the conductivity. 15

In witness whereof I have hereunto signed my name in the presence of two subscribing witnesses. 20

OTTO LIEBKNECHT.

Witnesses:

HORST ZIEGLER,
JEAN GRUND.