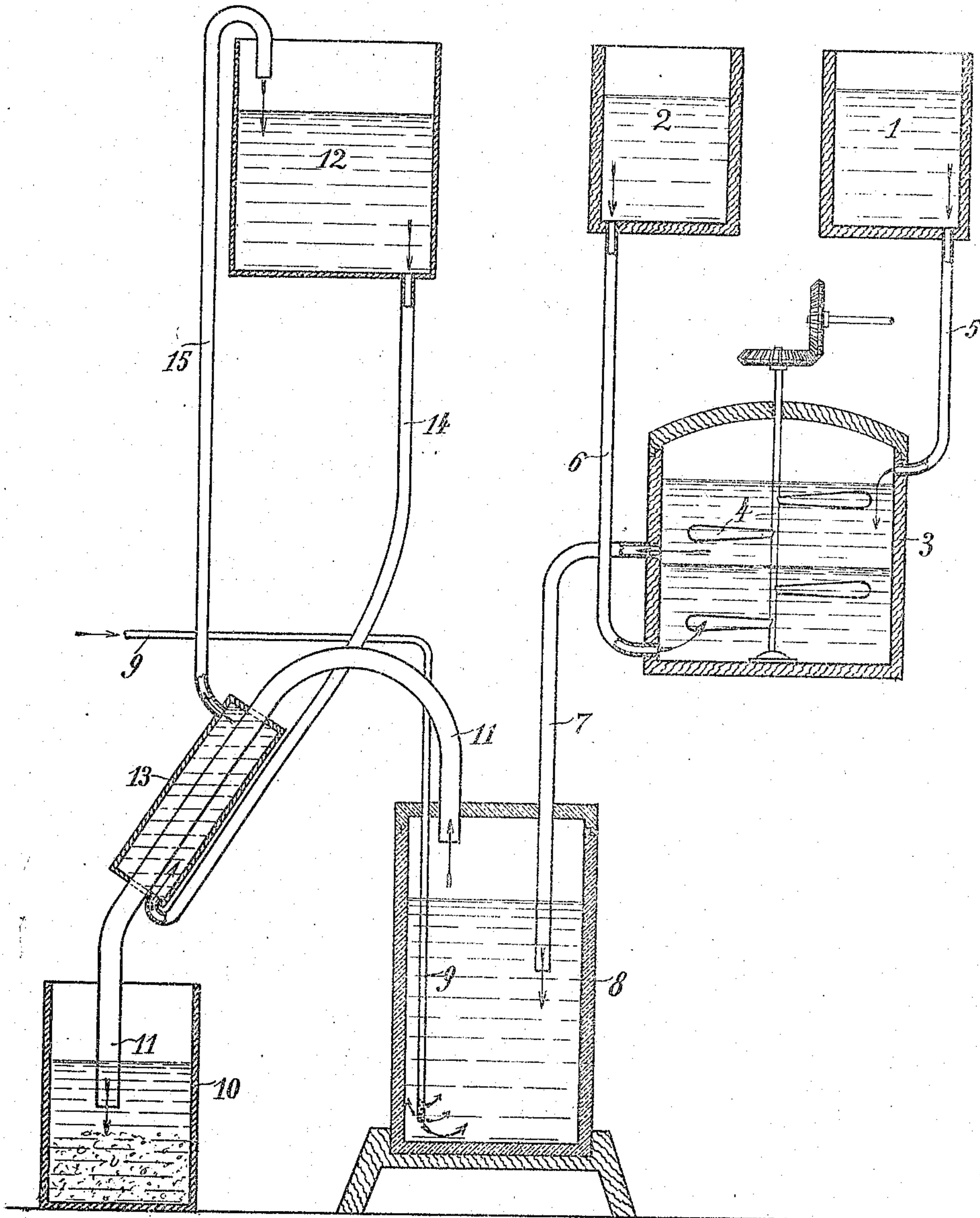


E. HERRMANN.
PROCESS OF EXTRACTING IODIN.
APPLICATION FILED MAY 9, 1906.

915,959.

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WITNESSES

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PROCESS OF EXTRACTING IODIN.

No. 915,959.

Specification of Letters Patent.

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To all whom it may concern:

Be it known that I, ERNEST HERRMANN, a citizen of the Republic of France, residing at 78 Rue d'Anjou, Paris, France, have invented new and useful Improvements in Processes of Extracting Iodin, of which the following is a specification.

This invention relates to a method by means of which iodine can be quickly and economically extracted from liquids containing iodides, iodates or organic ioduretted compounds. These liquids may be obtained by the exhaustion of fresh sea-weed, dry sea-weed, sea-weed ash or ioduretted substances of any kind. This exhaustion may be effected in any manner, according to processes already known. If it be desired to proceed quickly, methodical exhaustion with hot water (system of diffusers in sugar works) is advisable. If cold water be employed it is advisable in the case of fresh sea-weed to destroy the cells, in order that the sea-weed may more easily yield its ioduretted compounds. The treatment is either effected with a lime-milk, according to already known processes, or with an acid diluted to 2 to 3 per thousand, also according to already known processes. Sea-water may indeed be employed. It is only necessary to put about 2 grams more lime per liter in the milk of lime which may contain in that case from 5 to 7 per thousand of lime, so as to compensate the loss of lime due to the decomposition of the chlorid of magnesium which gives chlorid of calcium and insoluble magnesia which does no harm. Thus liquids are obtained which according to treatment and origin are richer in ioduretted compounds to the extent of 300 to 1000 grams of iodine per cubic meter. These liquids are then decanted, if necessary, or even filtered; they are then conveyed to wooden vats of medium size, in which stirring is easily effected, and the iodine set free by one of the known means, sulfuric acid or nitrate of soda for example. The ioduretted compounds are thus decomposed and the iodine set free. 60 liters (or approximately) of pure vaseline oil per cubic meter of liquid are then added and the liquid is agitated for a certain time so as to bring the vaseline oil well in contact with the liquid. The oil assimilates the free iodine and collects very rapidly on the surface.

The process is carried out methodically. The pure vaseline oil first treats liquids par-

tially exhausted and poor in iodine, then passes over liquid already once exhausted and finally over a fresh liquid. Thus 95% of iodine is carried off with about 60 liters of vaseline oil per cubic meter of liquid. This vaseline oil thus enriched with iodine, is poured into a stoneware or wooden receptacle and heated directly by a current of steam. This receptacle communicates with another similar receptacle through a porcelain or glass refrigerator, the latter if necessary being multitubular in order to effect adequate cooling.

The steam begins by heating the oil and the first receptacle must be rather large in order to contain the vapor condensed by this operation. Then, when the vaseline oil is hot, the steam carries off the iodine which is sublimated and becomes condensed in the refrigerator. Owing to the action of the steam the refrigerator does not become stopped. The second receptacle on the other side of the refrigerator receives the condensed steam and particles of iodine carried along in the water. When the vaseline oil has thus been deprived of its iodine, it is cooled and is employed again for treating liquids already partially exhausted. The vaseline oil may also be deprived of its iodine, if so preferred, by being directly heated or by being acted on by a current of hot air or of an inert gas.

If it be desired to proceed more quickly with the removal of the iodine from the vaseline oil so that a smaller quantity of vaseline oil will be required for the whole treatment, the iodine may be exhausted from the vaseline oil itself by stirring the oil with a liquid capable of forming a combination with the iodine. This result may be obtained very quickly with a solution of potash or of soda, but a certain time is required for decanting. The solution of sulfite of soda, while not taking more time to absorb the iodine, allows of very quick decanting.

The operation may proceed as follows:— To 600 parts of ioduretted vaseline oil, 1000 parts of water containing an excess of sulfite are added, stirring being carried on until the color due to the iodine has disappeared. The vaseline is decanted, removed and replaced by an equal quantity of ioduretted vaseline oil. Thus with the same quantity of liquid, to which when necessary sulfite is again added, it is possible to exhaust 15 to 20 volumes

of vaseline oil, thus enabling a highly concentrated iodid liquor to be obtained. The iodine of these concentrated liquors is set free by the known means, nitrites or chlorates, and it is separated by being drawn off by means of steam, by the process already described in relation to ioduretted vaseline oil, or it may be re-collected by decanting when it is precipitated.

10 The pure vaseline oil which is used in the process is obtained from the petroleum of Galicia (Austria); it constitutes a neutral uncolored liquid which distills only at a temperature above 350 degrees Celsius (660° F.).
15 This vaseline oil appertains to the chemical group of paraffins; it is not oxidized by contact with air, and it is not affected by the presence of concentrated acids.

The employment of vaseline oil insures the following advantages: The oil does not evaporate and does not ignite like benzene, petroleum or the like; it floats on the surface, thus enabling more or less clean liquids to be employed (this being impossible with solvents heavier than water, such as carbon bisulfid, chloroform or tetrachlorid of carbon), without washing and without subsequent treatment; it distills at about 345 degrees centigrade, thus enabling steam to be employed, as it does not emit any vapor before reaching its point of distillation. Finally the chemical group to which it belongs—the paraffins—are insensible to reagents and non-oxidizable in the air and thus can be employed indefinitely.
35

An apparatus for carrying out the process is shown in sectional elevation in the accompanying drawings.

40 In this Figure 1 is a receptacle of wood or other convenient material for containing the liquids from which iodine is to be extracted; 2 is a similar receptacle containing pure vaseline oil; 3 is a wooden vat provided with a stirring apparatus 4 actuated by any well

known means; 5 is a tube or pipe for supplying the liquid contained in the receptacle 1 to the vat 3; 6 is another pipe by means of which the pure vaseline oil contained in the receptacle 2 is discharged to the bottom of the vat 3. The oil enriched with iodine collects on the surface and is discharged continuously by the tube 7, into the stone-ware receptacle 8. 9 is a tube by which a current of steam is introduced directly to the bottom of the said receptacle 8, for heating the vaseline oil enriched with iodine; the outlet of the tube 9 is preferably provided with small holes so as to form a kind of sprayer for the exit of steam. The receptacle 8 communicates with the second receptacle 10 by means of a tube 11, preferably of porcelain or glass for the escape of the steam carrying off the iodine which is sublimated. The refrigeration is obtained by cold water, furnished by a tank 12 which communicates with the refrigerating jacket 13, by means of the pipes 14 and 15, arranged in the manner shown in the drawing for obtaining a continuous flow of cooling water. The arrows in the drawing show the directions in which the fluids move in the different tubes and receptacles.

Having now described my invention, what I claim as new and desire to secure by Letters Patent is:

A process of extracting iodine consisting in first liberating the iodine from iodine lyes, then mixing the iodine liquid with vaseline oil and then obtaining pure iodine directly from the resultant solution by subliming with steam.

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

ERNEST HERRMANN.

Witnesses:

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