(No Model.)

E. D. KENDALL.

PROCESS OF RECOVERING SULPHURIC ANHYDRIDE.

No. 323,583. Patented Aug. 4, 1885.

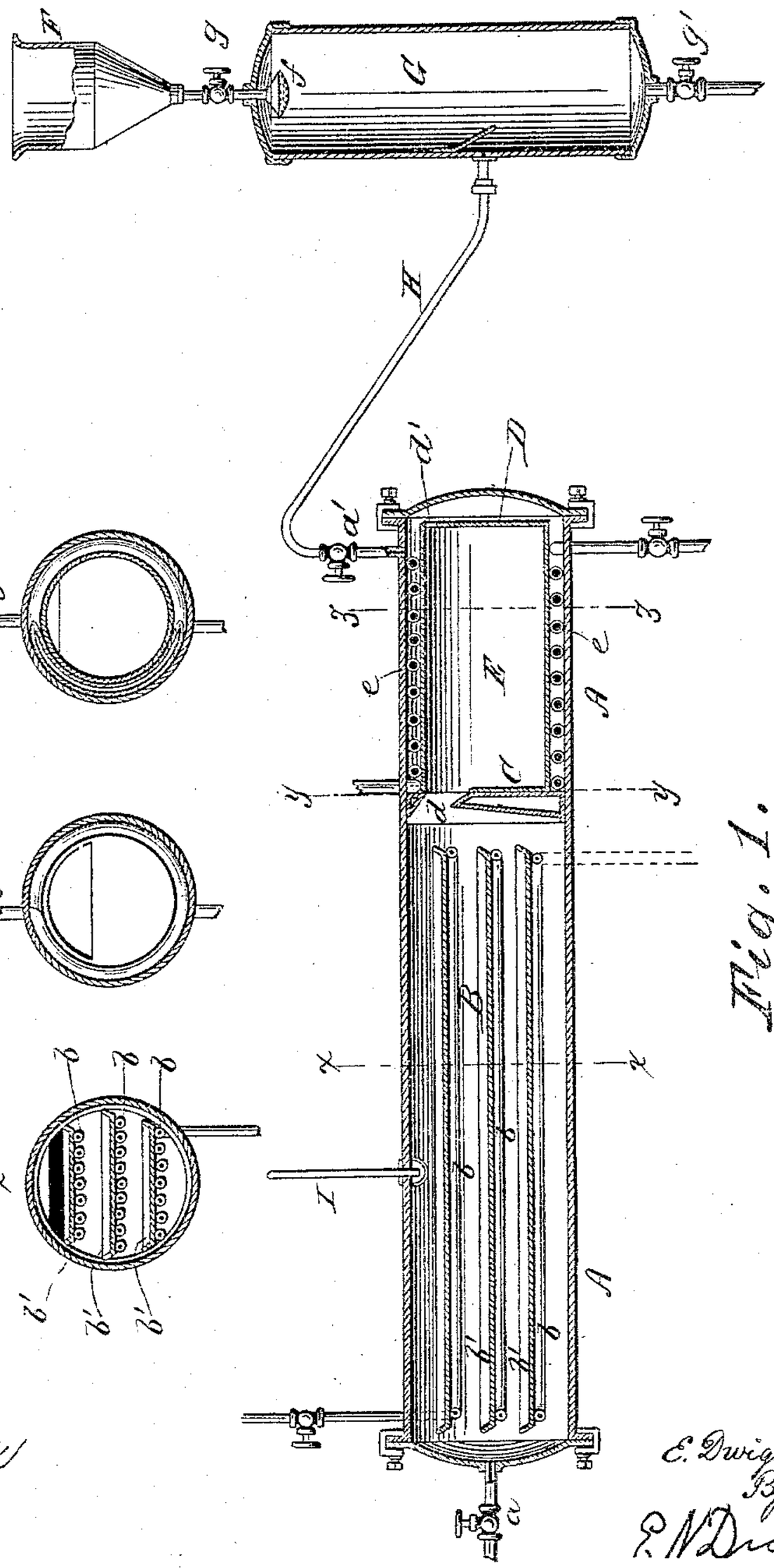

Fig. 1.

Fig. 4.

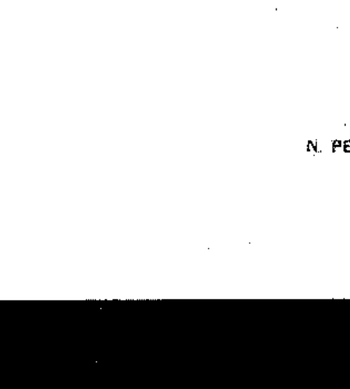

Fig. 3.

Fig. 2.

Witnesses:
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PROCESS OF RECOVERING SULPHURIC ANHYDRIDE.

SPECIFICATION forming part of Letters Patent No. 323,583, dated August 4, 1885.

Application filed May 28, 1884. (No model.)

To all whom it may concern:

Be it known that I, EDWARD D. KENDALL, of the city of Brooklyn, county of Kings, and State of New York, have invented a new and useful Process of Recovering Sulphuric Anhydride, of which the following is a full, true, and exact description, reference being had to the accompanying drawings.

It is well known that sulphuric anhydride, as contained in varying proportions in fuming sulphuric acid, or so-called "anhydrous sulphuric acid," is used in large quantities in the preparation of various coloring-matters, especially in the preparation of alizarine and of acid magenta. In making such preparations a surplus of the anhydride is necessarily used in order to accomplish the results desired. This surplus has generally not been recoverable, because if an attempt were made to distil off the anhydride under the pressure of the atmosphere, so much heat would be necessarily employed as to destroy or injure the coloring-matter.

The object of my invention is to recover the sulphuric anhydride from any body in which it may exist in excess by distilling the same in a partial vacuum.

My process consists in subjecting the mixture of fuming sulphuric acid and carbon compound to a moderate degree of heat, not sufficient to affect the character nor impair the quality of the desired carbon-sulpho-acid product, within a part of an apparatus from which air has been wholly or partially exhausted, whereby vapor of sulphuric anhydride is liberated at a comparatively low temperature, another part of the apparatus being arranged to condense and retain the anhydride, the necessary vacuum being produced by peculiar means in a part of the apparatus adapted to that purpose.

In my drawings I exhibit a form of apparatus, which, however, is no part of my invention as here claimed, nor do I limit myself to its use. It is, however, suitable for the purposes to be accomplished.

In my drawings, similar letters refer to similar parts.

Figure 1 is a view, partly in section, of my entire apparatus. Fig. 2 shows a section through Fig. 1 on the line *x x;* Fig. 3, a section through Fig. 1 on the line *y y;* Fig. 4, a section through Fig. 1 on the line *z z*.

In Fig. 1, A A is a horizontal cylinder made of boiler-iron, the ends of which are provided with removable covers made air-tight by suitable packing. One cover is furnished with a cock or valve, *a*, closing air-tight, and near the cover at the other end of the cylinder is a similar cock, *a'*. The interior of the said cylinder is divided into two chambers, B and E, by a thick or double-walled diaphragm, C, which chambers communicate with each other by an opening through the upper part of the diaphragm. Within the chamber B steam-pipes are arranged so as to form shelves *b b b*. (Also shown in cross-section in Fig. 2.) Steam may be transmitted through the said pipes by means of suitable connections passing air-tight through the sides of the cylinder A A. Shallow trays *b' b' b'* are represented resting on the said shelves. Within the chamber E is a coil of pipe, *e e*, arranged for the circulation of cold water by means of suitable connections passing air-tight through the top and bottom of the cylinder. Resting within the said coil and in close contact therewith is a cylindrical vessel, D, partially open at each end, as shown at *d* and *d'*, and also shown in Fig. 3. The end *d* of the vessel D fits closely against the flat surface of the diaphragm C.

G is a closed cylinder, provided at each end with a cock, *g* and *g'*, closing air-tight.

F represents a small tank or receptacle for liquid, and *f* is a perforated hollow ball or "rose" for delivering the liquid, as a shower, to the interior of the cylinder G.

H is a pipe and suitable coupling for connecting the interiors of the horizontal cylinder A A and the upright cylinder G.

I is a barometrical tube the mercury of which is in communication with the interior of the cylinder A A.

My process of recovering sulphuric anhydride and the operation of the above-described apparatus are as follows: The covers are removed from the ends of the cylinder A A. The vessel D is placed in position within the cold-water coil in the chamber E. The shallow trays *b' b' b'*, being nearly filled with the before-mentioned mixture of carbon compound and excess of fuming sulphuric acid,

are placed on the shelves *b b b*, and the covers are to be replaced air-tight on the ends of the cylinder A A, which cylinder is then to be connected with the cylinder G by the pipe and coupling H. The cock *g'* being closed and the cocks *a a'* and *g* open, dry carbon dioxide (known also as "carbonic-acid gas") is to be transmitted through the said cylinders. The gas enters through the cock *a* and finds exit through the cock *g* and receptacle F. A lighted taper being instantly extinguished when plunged into the receptacle indicates that the cylinders are full of carbon dioxide. The cocks *a* and *g* are now to be closed, and the receptacle F is to be filled with an aqueous solution of caustic alkali—say a solution of commercial caustic soda of about 1.15 specific gravity. On reopening the cock *g* the alkaline solution, under pressure of the atmosphere, descends with force in a shower from the rose *f*, rapidly absorbing carbon dioxide and creating a vacuum within the cylinders A A and G. The degree of exhaustion is indicated by the barometer-tube I. When this is sufficient, and while a portion of the alkaline solution still remains in the receptacle F, the cock *a'* is to be closed. A vacuum having thus been produced in the chambers B and E, a current of cold water is to be sent through the coils *e e*, and steam through the pipes that form the shelves *b b b*. Under these circumstances sulphuric anhydride is freely evolved from the excess of fuming or so-called "anhydrous sulphuric acid" contained in the trays *b' b' b'*, and condenses as a crystalline sublimate within the cooled vessel D. When this precipitation is completed, the apparatus may be opened by removing the covers, and the carbon compound contained on the trays *b' b' b'*, now despoiled of its surplus sulphuric anhydride may be removed, while this anhydride, being deposited in the vessel D, may be withdrawn by withdrawing said vessel from the other end of the apparatus.

I have shown the apparatus in these drawings as a convenient one for the purpose desired; but it must be observed that many other forms could be employed and still be within the limits of the invention—as, for instance, a mechanical vacuum produced by an air-pump might, in certain cases, be utilized by exhausting a large chamber and putting it suddenly in connection with the fuming sulphuric acid to be evaporated, the essential part of my invention consisting in removing and condensing sulphuric anhydride or fuming sulphuric acid by subjecting the compounds containing them to a low temperature and a partial vacuum, whereby the injury to the compounds which might result from subjecting them to a high temperature is avoided.

What I claim as my invention, and desire to secure by Letters Patent, is—

1. The process of recovering sulphuric anhydride from a compound containing an excess of fuming sulphuric acid, which consists in heating the compound in a vacuum or under a pressure less than that of the atmosphere, and of then condensing the volatilized sulphuric anhydride in a suitable chamber, substantially as described.

2. The process of recovering sulphuric anhydride from a compound containing an excess of fuming sulphuric acid, heating the compound in a vacuum or under a pressure less than that of the atmosphere in condensing the volatilized sulphuric anhydride, and depositing it in a chamber artificially cooled, substantially as described.

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Witnesses:
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