UNITED STATES PATENT OFFICE.

OTTO BAITHER, OF GRIESHEIM, GERMANY, ASSIGNOR TO CHEMISCHE FABRIK GRIESHEIM ELECTRON, OF FRANKFORT ON-THE-MAIN, GERMANY.

PROCESS OF CONCENTRATING DILUTED NITRIC ACID.

No. 819,262. Specification of Letters Patent. Patented May 1, 1906.

Application filed May 22, 1905. Serial No. 261,659.

To all whom it may concern:

Be it known that I, OTTO BAITHER, a subject of the German Emperor, and a resident of Griesheim-on-the-Main, Germany, have invented certain new and useful Improvements in Concentrating Diluted Nitric Acid, of which the following is a specification.

As is well known, in many chemical operations, such as nitrations, &c., a diluted nitric acid is obtained which in itself is of no use and can be utilized only after it has been concentrated. The method hitherto employed for concentrating the diluted nitric acid consists in first mixing this liquid with concentrated sulfuric acid, then distilling this mixture, so that a strong nitric acid is drawn out and a diluted sulfuric acid is left behind, next concentrating this diluted sulfuric acid in the known apparatuses used for the concentration of the sulfuric acid, and afterward remixing the concentrated sulfuric acid so obtained with fresh diluted nitric acid. This method, however, presents the following heavy disadvantages: The concentration of the nitric acid is effected in several distinct operations and requires two sets of apparatus—viz., one for the concentration of the nitric acid and another for the concentration of the sulfuric acid. Moreover, it is to be noted that by the concentration of the sulfuric acid a most diluted sulfuric acid as a waste is invariably obtained, which can be utilized only in the manner that it is employed in some sulfuric-acid works as water to be added.

It will be seen that the described method of concentrating diluted nitric acid will prove economical only in combination with a sulfuric-acid works. This means that the concentration of the diluted nitric acid is confined to certain places and that it remains so very expensive as to render the method useless in case the nitric acid is strongly diluted.

My invention relates to a new, exceedingly cheap, and easy method of turning nitric acid diluted to any degree into concentrated nitric acid in a single operation and by means of a single apparatus.

The new method essentially consists in mixing the diluted nitric acid with a polysulfate in a still at a temperature of from 110° to 130° centigrade, so that concentrated nitric acid is extracted and a hydrate of the polysulfate is left behind. The concentrated nitric acid is discharged from the recipient into a convenient storage-vessel. The hydrate of polysulfate in the still is constant at the said temperature of from 110° to 130° centigrade and can be only decomposed or freed from the water on increasing the temperature up to 250° or 300° centigrade. After the water has been drawn out at this higher temperature either the polysulfate is allowed to cool down or it is cooled to a lower temperature by blowing air or the like through it, whereupon a fresh charge of diluted nitric acid is introduced and the whole operation is repeated. The same quantity of polysulfate may be used over and over again in the still, while its temperature is periodically changed from 110° or 130° centigrade to 250° or 300° centigrade, and vice versa. The polysulfate may be, for instance, acid sulfate of sodium—that is, $H_3Na(SO_4)_2$.

Trials have shown that the proportion of the quantities of the nitric acid, the water, and the polysulfate may be varied within certain limits; but the method will prove most economical if on every molecule of the polysulfate ($H_3Na(SO_4)_2$) half a molecule of water is taken. Then the reaction, which is the basis of this method, is expressed by the following two equations, (x being the number of molecular weights of the nitric acid and y that of the water.)

1. $(HNO_3)_x.(H_2O)_y + 2_yH_3Na(SO_4)_2 = {}_xHNO_3 + 2_y(H_3Na(SO_4)_2.\frac{1}{2}H_2O).$

2. $2_y(H_3Na(SO_4)_2.\frac{1}{2}H_2O) = 2_yH_3Na(SO_4)_2 + {}_yH_2O.$

When, for example, a diluted nitric acid of 40° Baumé is to be concentrated up to 48° Baumé, then into a still containing, say, thirteen hundred kilograms of the polysulfate at 110° to 130° centigrade a quantity of say, three hundred and twenty kilograms of nitric acid at 40° Baumé is introduced and the mixture is maintained at this temperature as long as nitric acid of 48° Baumé passes over. Then one hundred and sixty kilograms of this acid will have been extracted, which represents eighty per cent. of the whole quantity of nitric acid at 48° Baumé. This concentrated nitric acid is discharged from the recipient into some convenient stor-

age vessel. Thereupon the temperature in the still is increased, when at the beginning the missing twenty per cent. of the nitric acid will pass over in a state of 40° Baumé on the average and this quantity is put back for the next following operation. After the temperature has reached 250° to 300° centigrade the remaining water will pass over and this water extracted is allowed to run off. The polysulfate left behind is allowed to cool down to its initial temperature of from 110° to 130° centigrade and then again utilized for concentrating a fresh quantity of diluted nitric acid.

The distillation of the nitric acid may be considerably accelerated and at a considerably lower temperature than that mentioned above by blowing air through the mixture during the whole period of the operation, and the output of concentrated nitric acid will also be more favorable. After the extraction of the nitric acid the air blown through will also free the polysulfate from the water quicker, and, moreover, the air blown through will accelerate the cooling of the polysulfate so freed from the water.

The same advantages as by the blowing through of air may be obtained by rarefying the air within the distilling apparatus during the whole operation.

It will be seen that the division of the nitric acid and the water is not effected quantitatively. However, as any quantity of diluted nitric acid may be divided and this division can be obtained in a single operation and with the same apparatus at any place, while one and the same quantity of polysulfate is used over and over again and there are no by-products or wastes whatever, the new method is far superior to the hitherto known method and it presents many important advantages, so that it is of great commercial value.

It is true that in the German Patent No. 106,962 a process has been disclosed in which also a polysulfate is used for the manufacture of concentrated nitric acid; but there is an important difference between this process and that described above in that the process acccording to the said German patent is not intended for the concentration of already-existing diluted nitric acid, but solely for the manufacture of the nitric acid direct from saltpeter. Obviously this manufacture of the nitric acid is quite distinct from the concentration of diluted nitric acid. Also the materials used in both processes are different, since in the method according to the said German patent polysulfate, sulfuric acid, and Chili saltpeter are used, while in my present method polysulfate and diluted nitric acid are employed. The final products are equally different, concentrated nitric acid and water being obtained in the former process and nitric acid and acid sulfate of sodium in the latter process. For the former process a special advantage is claimed, in that the temperature does not vary, whereas in my process the temperature is periodically changed from 110° or 130° centigrade to 250° or 300° centigrade, and vice versa. Not only is my method distinct from the method described in the said German patent, but also it could not be expected at all that the division of the nitric acid and the water by the hydration of the polysulfate might be extended to such a degree that the concentration of the diluted nitric acid could be effected in an economical and profitable manner, as my trials have proved.

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

1. The method of concentrating diluted nitric acid, which consists in mixing the diluted nitric acid with a quantity of polysulfate in a still at from 110° to 130° centigrade, distilling concentrated nitric acid from the mixture at this temperature, discharging the concentrated nitric acid into a storage vessel, increasing the temperature in the still up to from 250° to 300° centigrade, so that the remaining water passes over, allowing the latter to run off, cooling the polysulfate in the still down to its initial temperature, and repeating the operation with a fresh charge of diluted nitric acid.

2. The method of concentrating diluted nitric acid, which consists in mixing the diluted nitric acid with a quantity of polysulfate ($H_2Na(SO_4)_2$) in a still at from 110° to 130° centigrade, distilling concentrated nitric acid from the mixture at this temperature, discharging the concentrated nitric acid into a storage vessel, increasing the temperature of the mass in the still up to from 250° to 300° centigrade, so that first the remaining nitric acid passes over in a diluted state and afterward the remaining water, putting back the extracted diluted nitric acid for the following operation and allowing the extracted water to run off, cooling the polysulfate ($H_3Na(SO_4)_2$) in the still down to its initial temperature, and repeating the operation with a fresh charge of diluted nitric acid.

3. The method of concentrating diluted nitric acid which consists in cooling a quantity of hot polysulfate in a still, mixing a charge of diluted nitric acid with this polysulfate, distilling concentrated nitric acid from the mixture at the same temperature, discharging the concentrated nitric acid into a storage vessel, increasing the temperature of the mass in the still up to such a point, that the remaining water passes over, allowing the latter to run off, cooling the polysulfate in the still down to its initial temperature, and repeating the operation with a fresh charge of diluted nitric acid.

4. The method of concentrating diluted nitric acid, which consists in constantly blow-

ing air through a quantity of hot polysulfate in a still, mixing a charge of diluted nitric acid with this polysulfate, distilling concentrated nitric acid from the mixture at the same temperature, discharging the concentrated nitric acid into a storage vessel, increasing the temperature of the mass in the still up to such a point, that the remaining water passes over, allowing the latter to run off, cooling the polysulfate in the still down to its initial temperature with the aid of the air blown through, and repeating the operation with a fresh charge of diluted nitric acid.

5. The method of concentrating diluted nitric acid, which consists in constantly blowing air through a quantity of hot polysulfate ($H_3Na(SO_4)_2$) in a still, mixing a charge of diluted nitric acid with this polysulfate ($H_3Na(SO_4)_2$), distilling concentrated nitric acid from the mixture at the same temperature, discharging the concentrated nitric acid into a storage vessel, increasing the temperature of the mass in the still up to such a point, that first the remaining nitric acid passes over in a diluted state and afterward the remaining water, putting back the extracted diluted nitric acid for the following operation and allowing the extracted water to run off, cooling the polysulfate ($H_3Na(SO_4)_2$) in the still down to its initial temperature with the aid of the air blown through, and repeating the operation with a fresh charge of diluted nitric acid.

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

OTTO BAITHER

Witnesses:

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ERWIN DEPPEL.