C. BLAGBURN.

PROCESS OF OBTAINING SUBSTANTIALLY PURE NITROGEN FROM THE AIR.

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1,036,788. Patented Aug. 27, 1912.

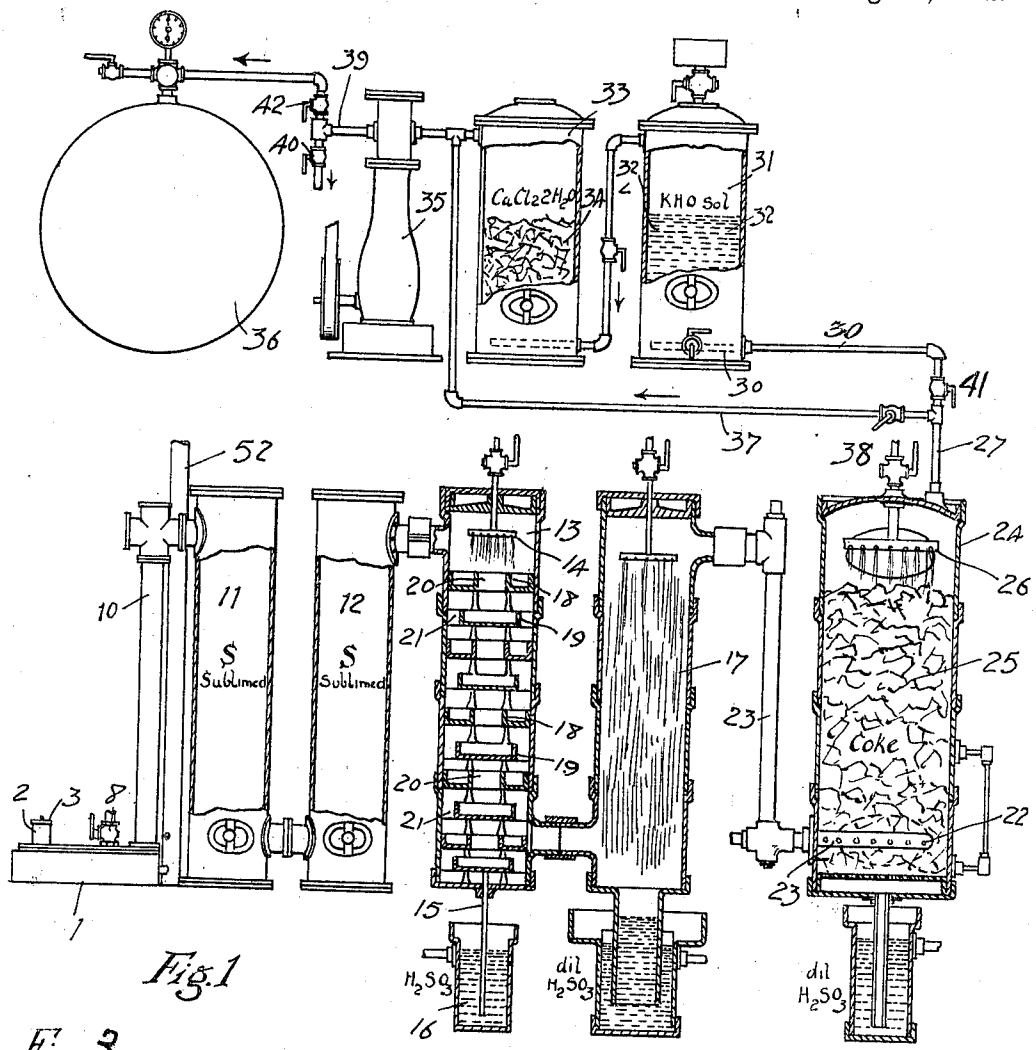
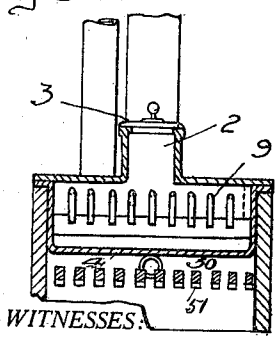


Fig. 1

Fig 3



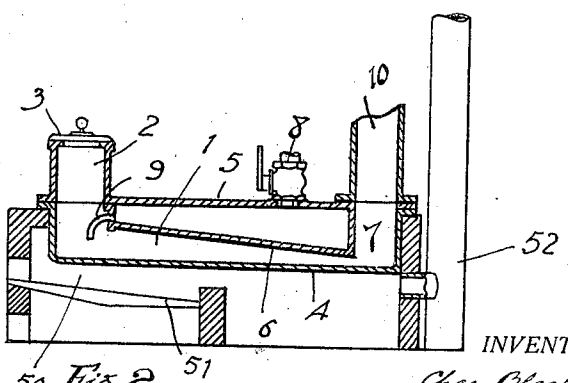


Fig. 2

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PROCESS OF OBTAINING SUBSTANTIALLY PURE NITROGEN FROM THE AIR.

1,036,788. Specification of Letters Patent. Patented Aug. 27, 1912.

Application filed November 27, 1907. Serial No. 404,120.

To all whom it may concern:

Be it known that I, CHARLES BLAGBURN, a subject of the King of Great Britain, residing at Antioch, in the county of Contra Costa and State of California, have invented new and useful Improvements in Processes of Obtaining Substantially Pure Nitrogen from the Air, of which the following is a specification.

The object of the present invention is to provide a cheap process of obtaining from the air practically pure nitrogen.

The problem of obtaining nitrogen from the air at a low cost has become in recent times extremely important; first, on account of the greatly increased demand for nitrate fertilizers to reënrich poor or exhausted soils; second, on account of the extensive and increasing use of cyanids in mining and the arts; and, third, on account of the use of nitrogen gas as a preservative medium for preserving edibles and other perishable articles in air-tight receptacles.

I have discovered a process, by means of which sulfur may be so burned on a commercial scale as to remove all of the oxygen from the stream of air supplied for its combustion, thus solving the "nitrogen" problem, since this process, on account of the cheapness of sulfur, enables the nitrogen to be obtained at quite a low cost.

The process herein claimed generically was disclosed in my prior application No. 296,160, filed January 16, 1906.

In the accompanying drawing, Figure 1 is a diagrammatic sectional view of the apparatus for carrying out my improved process; Fig. 2 is a longitudinal section of the furnace; Fig. 3 is a transverse section thereof.

1 indicates a combustion chamber, which is very shallow in proportion to its length and width, in order to expose a large surface of sulfur to the air of combustion. To said combustion chamber sulfur is fed by means of a charging passage 2 which can be closed when desired by means of a suitable cover 3. Beneath said combustion chamber is a fire-box 50, having a grate 51 and a flue 52, in which a fire can be kindled to heat the sulfur initially, or, if desired, suitable fuel can be burned during the process, although I have not found this to be necessary. The bottom 4 of the combustion chamber 1 slopes very slightly, (the slope being exaggerated in the drawing) from front to rear, to insure the melted sulfur flowing over the entire surface of said bottom. In said combustion chamber and extending across its entire width is a heating chamber 5, which has a bottom 6, which thus forms the top of the combustion chamber. It slopes downward from a point near the charging end of the combustion chamber to the outlet 7 thereof. Into said heating chamber air is drawn by suction, as hereinafter described, through a pipe 8, and impinges upon the sloping bottom thereof. The air in this heating chamber, being highly heated by the combustion of sulfur in the chamber beneath, emerges from the heating chamber through downwardly extending pipes 9, by which means it is fed to the sulfur in the bottom of the combustion chamber. However, while it is thus desirable in many cases, as for making nitrogen on a very large scale, to heat the air before supplying it to the sulfur, I am enabled to carry out my process by burning the sulfur with air not heated before being introduced into the combustion chamber, this air being supplied through the charging opening 2, the cover 3 being then removed.

In order to successfully practise this invention, two main conditions are necessary. First, the furnace must be so constructed that the air of combustion is compelled, before escaping from the combustion chamber, to pass into close proximity to the sulfur, which, near the point of exit, would be in a molten condition with sulfur vapors arising therefrom; and, secondly, the sulfur must be in excess, or at any rate no more air must be supplied to the sulfur than is necessary to supply the oxygen for combustion thereof.

As will hereinafter appear, the gases from the furnace have to pass through a long series of purifying chambers, and, therefore, in order to supply any air to the furnace, a suction pump is necessary at the end of said series. Now, for a given rate of feeding the sulfur, the suction pump must be so operated that the current of air supplied thereby is never more than necessary, as above mentioned. So long as sulfur vapor passes off unconsumed, as evidenced by the formation of sublimed sulfur in the condensing chambers, the process is being properly carried out.

I realize that the apparatus itself, that is, the furnace, which is the essential element

thereof, may be greatly varied in form and construction, and still satisfy the other essential condition, namely, that the air be compelled to pass into close proximity to the highly heated sulfur, so that no oxygen can escape, and all such variations I regard as within the scope of my invention.

From the outlet 7 of the furnace, the gases pass upward by a pipe 10. Should any oxygen have been unconsumed by the sulfur before reaching the pipe 10, it will certainly combine with the excess sulfur vapor therein, the pipe 10 being intensely hot. From the pipe 10 the gases, consisting of nitrogen, sulfurous anhydrid, sulfur vapor, and a small amount of steam, due to the presence of moisture in the atmospheric air supplied to the furnace, then pass in succession through two subliming chambers 11, 12, which are sufficient to condense and precipitate in the form of sublimed sulfur nearly all the excess of sulfur which has passed off in the form of vapor.

From the top of the second subliming chamber 12, the nitrogen and sulfurous anhydrid pass into the top of a sulfurous acid tower 13, in the top of which is arranged a spraying device 14 which sprays water thereinto. In said tower are placed one above the other alternating trays 18, 19, of two series, the trays 18 of one series having a central passage 20, and those of the other having an annular space 21 between the side of the tray and the side of the tower, through which the gases can pass. The water is compelled to flow from one tray to the next below it either through a central opening 20, or through one of the annular passages 21. This water, if not too much, passing through the nitrogen and sulfurous anhydrid gases, forms from the latter strong sulfurous acid, which is collected by a pipe 15 from a liquid seal 16 at the bottom of said tower. The nitrogen and part of the sulfurous anhydrid gases then pass into the bottom of a second tower 17, into the top of which water is also sprayed, the amount of water supplied in this tower being greater than that in the first tower, so that sulfurous acid in much larger quantity, but greatly diluted, is collected therefrom.

From the tower 17 the gases pass through perforations 22 of a supply pipe 23 entering the bottom of a wash vessel 24 and pass upward between pieces of some porous substance 25, such as coke, which exposes a great extent of surface, upon which water is sprayed by means of a sprayer 26, so that the sulfurous anhydrid gas coming in contact with the moist surface forms with the water diluted sulfurous acid, which is collected at the bottom in the same manner as before.

From the top of the wash vessel 24 the gases pass through a perforated pipe 30 into the bottom of a wash vessel 31, about half filled with a solution 32 of caustic potash, which serves to effectually arrest any trace of sulfurous acid or carbonic acid gas, and forming sulfite of potassium, which is valuable in the arts. From the top of the vessel 31 the gases pass into a vessel 33 which is about half filled with calcium chlorid 34 to take up the moisture. This vessel is used when dry nitrogen is required for use as a preservative, but in other cases, when the nitrogen is not so used, the calcium chlorid may be omitted. From the top of the latter vessel the gas passes to a suction pump 35, which creates the current which draws the air into the furnace and the gases through the series of chambers, and which likewise forces the nitrogen gas into a suitable receiver 36, from which it may be drawn out as required for use.

By using a suction pump to draw the gases through the furnace I insure that the products of combustion shall have a greater velocity than, or a velocity at least as great as, the air that flows into their place. From this it results that the sulfurous fumes are carried off as quickly as formed, and the air can have free access to the surface of the burning sulfur.

In order to economize in respect of the chemical reagents in the vessels 31, 33, there is provided a bypass pipe 37, having a valve 38, which is opened in commencing the process, and is closed as soon as the gas is sufficiently free from oxygen. On the other side of the pump is a discharge pipe 39 having a valve 40 therein. A valve 41 is interposed between the vessels 24 and 31, and a valve 42 between the suction pump and the receiver. The valves 38 and 40 are opened and the valves 41 and 42 are closed on commencing the process, and the gas emerging through the pipe 39 is tested until it is found to be sufficiently free from oxygen, and then said valves are closed and the valves 41, 42, are opened, and the gas is passed into the receiver 36.

It will be understood that in all cases the vessels are composed of suitable material, such as earthenware, or, in certain cases, of iron lined with lead, to withstand the action of the acids passing therethrough. It is also important that said vessels are made perfectly air-tight.

I have found that by the above process nitrogen of great purity can be obtained from the atmosphere. In practising this process, it has been my custom to reject all nitrogen gas which does not reach a purity of 99.6 to 99.8 per cent. Generally speaking, it is only retained at the latter degree of purity. By placing iron filings in the receiver for a few days, even this small percentage of oxygen, .2 per cent, is found to

be absent, and the receiver then contains absolutely pure nitrogen.

The cost of obtaining nitrogen by the above process is very much less than those by processes heretofore attempted commercially, so far as my knowledge extends.

I make no claim herein to the apparatus disclosed in this application as said apparatus forms the subject of a separate application, Serial No. 544,212, the same being a division of this application, said application having matured into Patent No. 993,017.

I claim:—

1. A continuous process of obtaining substantially pure nitrogen from atmospheric air which consists in causing molten sulfur to flow in a stream, feeding sulfur and supplying air at one end of said stream while restricting the amount of air supplied so that no more oxygen will be present than is sufficient to combine with the sulfur from said stream, compelling the whole of the air to flow, in its whole course, close to the surface of the burning sulfur, conducting away, while confining, the sulfurous acid, unburned sulfur, and nitrogen, depositing the unburned sulfur, washing out the sulfurous acid, and collecting the nitrogen, substantially as described.

2. The process of obtaining substantially pure nitrogen from atmospheric air which consists in directing the whole of a stream of air downward into close proximity to the surface of burning sulfur, restricting the magnitude of said stream so that no more oxygen will be present than suffices to combine with the sulfur arising therefrom, reducing the height of said stream as it advances, while increasing its velocity, conducting away the sulfurous acid, unburned sulfur, and nitrogen, and removing therefrom the unburned sulfur and sulfurous acid.

In testimony whereof I have hereunto set my hand in the presence of two subscribing witnesses.

CHARLES BLAGBURN.

Witnesses:

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D. B. RICHARDS.