Aug. 31, 1948. A. LATHAM, JR 2,448,491

AIR SEPARATING SYSTEM AND PROCESS

Filed Nov. 6, 1944 4 Sheets-Sheet 1

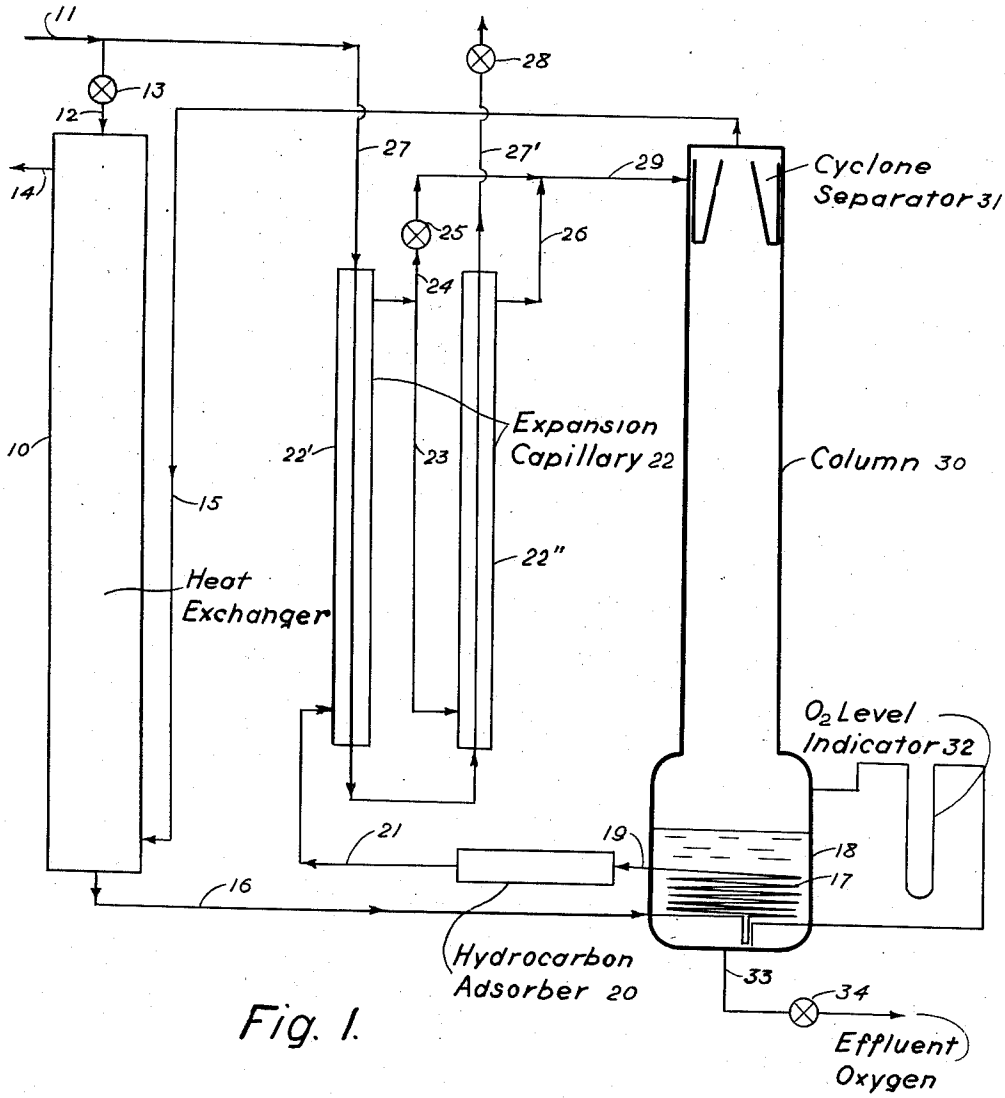


Fig. 1.

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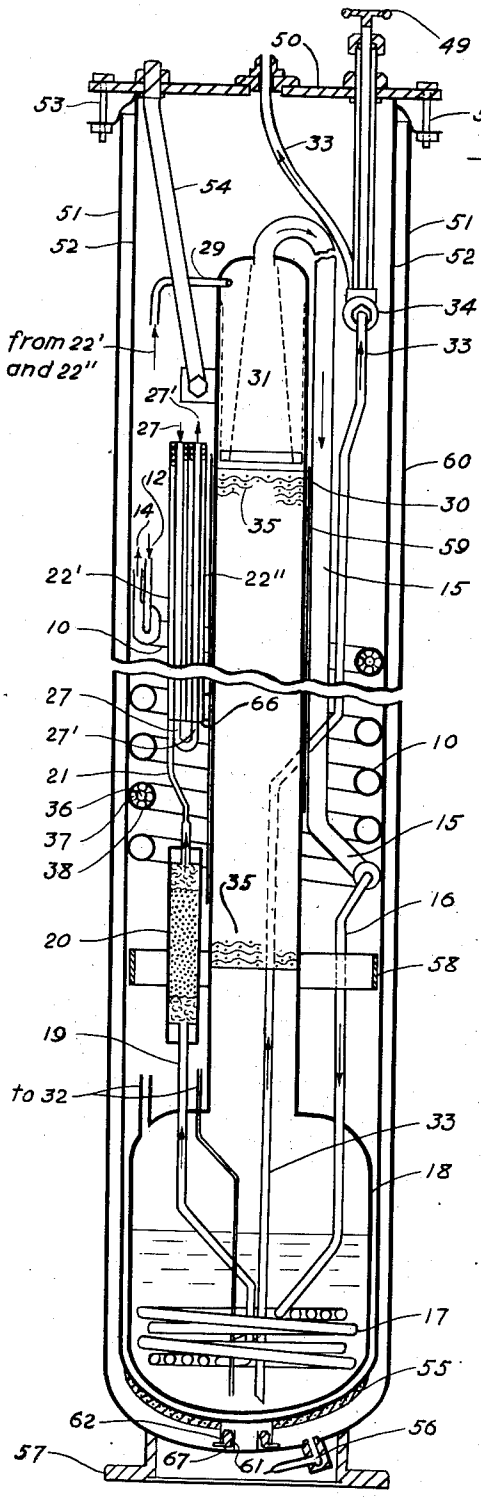


Fig. 2.

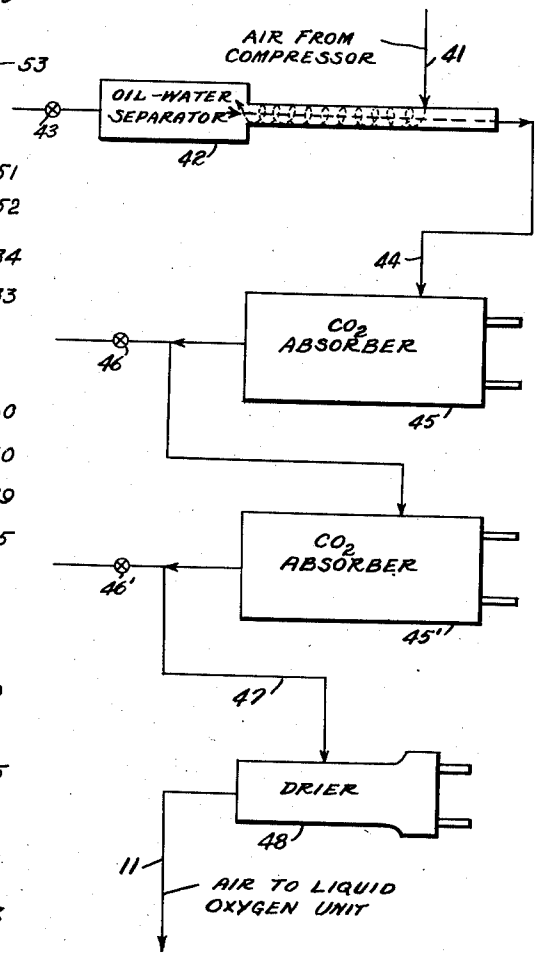


Fig. 4.

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AIR SEPARATING SYSTEM AND PROCESS

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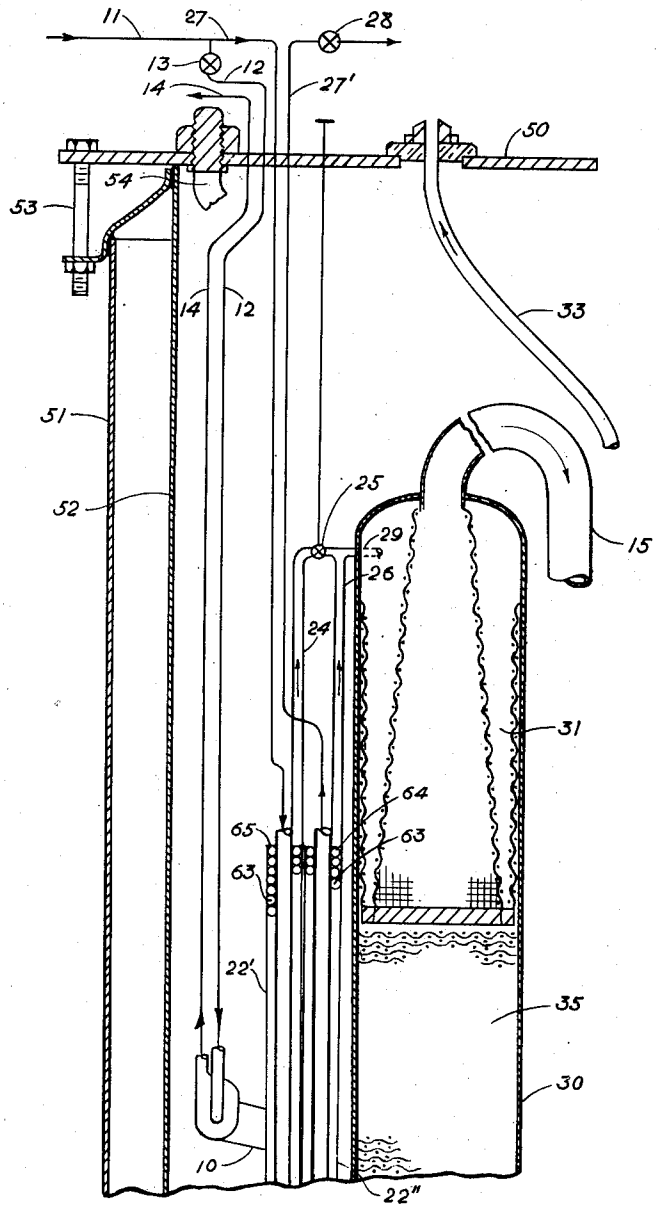


Fig. 3.

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AIR SEPARATING SYSTEM AND PROCESS

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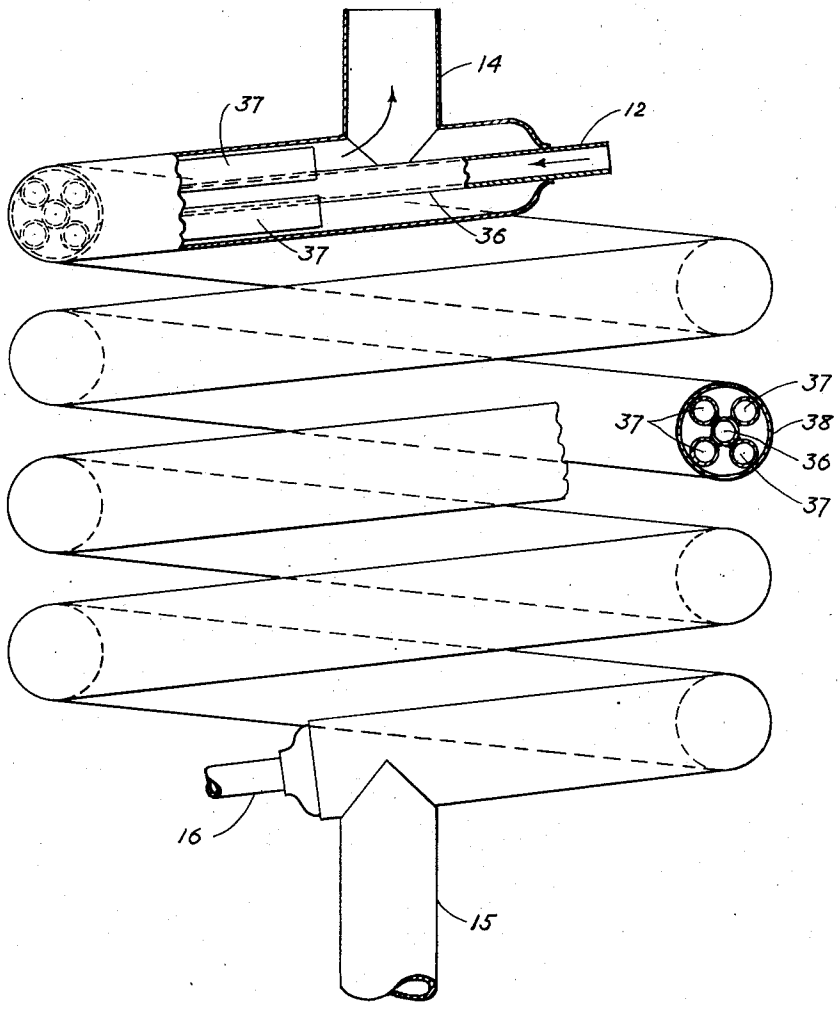


Fig. 5.

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2,448,491

AIR SEPARATING SYSTEM AND PROCESS

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6 Claims. (Cl. 62—175.5)

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This invention relates to the production of liquid oxygen and is particularly concerned with providing a portable apparatus of simple and rugged construction which can produce oxygen on a relatively small scale and can be operated by inexperienced personnel.

Objects of this invention include the provision of a device of the foregoing type which is especially compact and occupies a small volume particularly in comparison with its output; which is simple in operation with very little attention required after starting up; and which is easy to manufacture. These objects and others which will appear as the disclosure proceeds are attained by the proper arrangement and balancing of the parts and particularly by the incorporation of an improved design of heat exchanger and by the use of an expansion capillary having a warming element—all as described in detail below and with reference to the accompanying drawings in which:

Fig. 1 represents a flow sheet of the apparatus;

Fig. 2 is a side elevation of the assembled apparatus of Fig. 1, partly in section;

Fig. 3 is an enlarged portion of the upper part of Fig. 2 showing details of the piping arrangement and valves which are too small to be shown well in Fig. 2;

Fig. 4 is a flow sheet of an air purification system which may be used with the apparatus; and

Fig. 5 is a detailed elevation, partly in section, of the heat exchanger **10**.

As will be seen by reference to the drawings, especially Fig. 1, the apparatus of this invention comprises a heat exchanger **10**, a packed column **30** provided with a separator **31** at the top and a reboiler **18** at the bottom, and an expansion capillary **22** in contact with warming tube **27, 27'**. Purification of the air entering through pipe **11** may be carried out to whatever extent desired and by any suitable means of which one means is shown in Fig. 4. The hydrocarbon adsorber **20** is also a part of such a purification system.

Compact and efficient arrangement of the parts is accomplished as indicated in Figs. 2 and 3. These parts are arranged within a vacuum jacket which is generally represented by the numeral **60** having an outer shell **51** and an inner shell **52**. The space between these shells is evacuated, through a suitable lead tube **56**, as far as is reasonably possible, for example to a pressure of 10^{-6} mm. of mercury absolute. The lead tube **56** is then sealed off. Suitable insulation

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may also be provided, as by covering the outer wall of the inside shell with layers of aluminum foil alternating with glass cloth to serve as an efficient barrier to the flow of radiant heat. The two shells **51** and **52** are held concentrically with respect to each other by head plate **50** at the top, and also by positioning means at the bottom of each shell, such positioning means consisting of a plastic insulating button **67** which fits over a projection **61** attached to the inner surface of the bottom of the outer shell **51** and into a socket **62** attached to the outer surface of the bottom of the inner shell **52**.

Shells **51** and **52** will ordinarily be made of metal. Since in metal vacuum containers there is always a slow diffusion of gas from the walls, which gas if allowed to accumulate will eventually destroy the insulating properties of the jacket, provision is made of an adsorber **55** (e. g. activated charcoal) to adsorb such gas. By such means a high vacuum can be maintained in the metal container for several years. The activated charcoal or other adsorbent is conveniently contained in a shallow basket attached to the outside bottom of shell **52** as indicated in Fig. 2. This basket is provided with openings covered with glass wool filters held in place by a fine wire screen to prevent the passage of the adsorbent into the vacuum space. These windows or openings provide free communication between the body of adsorbent and the space between shells **51** and **52**.

A head plate **50** is bolted by means of bolts **53** to the top of the vacuum jacket **60** and suitable openings are provided in this head plate **50** for pipes or conduits leading the gaseous products to and from the space within the vacuum jacket **60**.

Positioned centrally within the vacuum jacket **60** is rectifying column **30** which is filled with any suitable column packing **35**, e. g. Berl Saddles. This column is supported from the head plate **50** by a series of supporting struts, one of which is indicated by numeral **54**, and is centered within the vacuum jacket by centering ring **58**. The column **30** may conveniently be surrounded by a barrier or insulation **59**, which advantageously consists of a cylindrical copper sheath surrounding column **30** and separated therefrom by glass wool, and soldered to pipe **15**. This barrier **59** helps prevent passage of heat into column **30**. In the top of the column **30** there is provided a cyclone separator **31** which may, for example, be made of fine mesh screen supported on perforated metal, or be made in any suitable con-

ventional form, and which is designed to separate the liquid portion of the air entering the column through pipe 29 and allow it to run down onto the packing 35 without being entrained by the effluent stream of gas passing out through pipe 15. The bottom of column 30 communicates directly with a reboiler 18 which also serves as a liquid reservoir. Within this reboiler 18 is located the reboiler coil 17. A liquid level indicator 32 is connected with the reboiler 18 in any suitable manner to indicate the level of the liquid within the reboiler. A water manometer is satisfactory for this purpose if suitable traps are placed in the line between it and the interior of the reboiler 18.

The expansion capillary represented generally by the numeral 22 is made up of a long length of small diameter tubing 63 wound around a warming air tube 27, 27' which communicates with the supply of entering air in pipe 11. This small diameter tube 63 which serves as an expansion device for high pressure cold air coming from reboiler coil 17 through tube 21 is conveniently wound in such a way that the air passing through it may be led off about midway of the tubing 63 thus by-passing the second half of said tubing. As shown in the drawings this is accomplished by providing a take-off line 24 about midway of the total length of tubing 63, line 24 being controlled by valve 25 and leading into line 29 into which line 26 from the end 64 of said tubing 63 is led. Specific arrangement of the expansion capillary is shown in Figs. 1, 2, and 3 wherein high pressure air is led through line 21 from reboiler coil 17 and passes through arm 22' of expansion capillary 22, which arm consists of tubing 63 wound around and soldered to warming air tube 27; thence the air, partly expanded, leaves said tubing at its upper end 65 and passes downwardly through pipe 23 into the bottom end 66 of the other arm 22'' of the expansion capillary 22, and then through the tubing 63 of said arm 22'' and out at exit end 64, thereby completing the expansion of the air in said arm 22'' out of which arm the expanded air, now mostly liquefied, passes through lines 26 and 29 to the cyclone separator 31 at the top of column 30. In pipe 23 is located take off line 24 controlled by valve 25 and leading to pipe 29. The warming air tube 27, 27' is controlled by valve 28 so that when the latter is closed no gas passes through tube 27, 27'.

Each of the two arms 22', 22'' may for example consist of about 12 feet of ⅛'' O. D. by .020'' wall tubing 63 wound on a 30'' piece of ¼'' O. D. tubing 27 and 27' and soldered in place. During normal operation, the air passes through both arms, entering through pipe 21 and leaving through pipe 26. In starting the unit, however, such an arrangement would place too much restriction on the flow of air since the relatively warm air then fed to the capillary has a much greater specific volume than the cold air fed when the unit is in full running operation. In order to pass enough air during the starting period to cool the unit in a reasonable time the by-pass valve 25 is opened. With this valve open the air passes only through arm 22' of the capillary and thus a much larger flow is obtained. The warming tube 27, 27' is arranged so that a current of warm high pressure air may be passed through it from pipe 11 by opening valve 28. In the event that the capillary tubes 63 become plugged with water, carbon dioxide, or oil the flow through the capillary stops whereupon warm

air is passed through tube 27, 27' by opening valve 28. By this means the obstruction is melted and is easily blown out. The warming air connection may be provided with a heater but this is not necessary as it is possible to remove the obstructions by using air at room temperature. This warming operation can be completed in about five minutes and the unit resumes production immediately since only a small mass of metal is warmed. For proper operation, the total length of the tubing 63 in both arms of the capillary 22 should be at least 150 times its inside diameter.

The heat exchanger 10 is shown in detail in Fig. 5. High pressure entering air from pipe 11 passes into the heat exchanger 10 through pipe 12 and out through pipe 16, these two pipes being connected by tube 36 running axially of the heat exchanger assembly and being essentially of the same diameter as pipes 12 and 16. Surrounding tube 36 are tubes 37, which are preferably of about the same diameter as tube 36, and of which four is a convenient number, which are soldered throughout their lengths to tube 36 and also to the inner wall of outside shell 38, thus insuring excellent contact and heat transfer capacity. The whole assembly is wound spirally so as to fit within the inner shell of the vacuum jacket 60. Effluent nitrogen from pipe 15 enters the space between tube 36 and shell 38 passing both inside and outside of tubes 37 and emerges at pipe 14 thus passing countercurrently to the entering air to cool same. While this cooling could be effected without the presence of tubes 37 the exchanger 10 would then have to be made very much longer to do so with the result that the compactness and efficiency of the present apparatus would be largely lost. It is thus seen that tubes 37 serve primarily to promote efficient heat transfer between the heated compressed air in tube 36 and the cold effluent nitrogen in the space between tube 36 and shell 38. In view of the metal-to-metal contact between tubes 37 and tube 36, the heat of the latter tube which is received from the heated compressed air readily passes into the walls of tubes 37, whence it is dissipated to the cold effluent nitrogen in contact with both the outside and the inside of said latter walls. The heat exchanger arrangement shown herein, wherein tubes 37 are used, therefore provides a very efficient heat exchange device of compact arrangement and small size relative to its capacity and which is also easily and reproducibly manufactured and is free from troublesome joints. Lightness in weight is achieved by using all surfaces except the outside surface of shell 38 for heat transfer and through the maintenance of high velocities throughout. The maximum wall thickness of any of the tubes 36 and 37 and shell 38 is that of the high-pressure tube 36, which is about $\frac{1}{32}$''. Since the walls of tubes 37 and shell 38 are subjected to small pressures or none at all, they are conveniently made of thinner stock than the walls of tube 36. All parts can be assembled with maintenance of uniform dimensions throughout. All pressure type joints are on the outside where they may be readily observed and repaired during leakage tests.

The air purification system shown in Fig. 4 and also including hydrocarbon adsorber 20 (Figs. 1 and 2) has for its purpose the removal of liquid oil, carbon dioxide, water, water vapor, and hydrocarbon vapors. Accordingly as these undesirable components are present in greater or lesser amounts the purification system may be

modified, added to, or more or less eliminated as may be necessary. The system shown in Fig. 4 and including hydrocarbon adsorber 20 is intended for removal of all types of undesirable components listed above. Air from the compressor is led through pipe 41 into the oil-water separator 42 which is a cyclone type separator with a trap at the bottom provided with an outlet line controlled by valve 43. From this separator 42 the air passes through line 44 to a series of carbon dioxide absorbers 45, 45' which consist of casings in which are placed canisters containing a dried granulated sodium hydroxide or other suitable carbon dioxide absorbent. These canisters are removed and replaced as required. By taking off air through the lines controlled by valves 46, 46' and bubbling it through barium hydroxide solution, the degree of exhaustion of the absorbent in the canister can be determined. From the carbon dioxide absorbers the compressed air passes through line 47 to drier 48 which also consists of a casing containing a canister of any suitable dehydrating agent which likewise may be removed and replaced as required. From drier 48 the purified air passes through pipe 11 to the main apparatus. The hydrocarbon adsorber 20 is filled with activated charcoal or other suitable adsorbent and plugged at each end except for pipes 19 and 21 communicating with its interior. It is placed just before the expansion capillary 22, rather than nearer the point where the compressed air enters the system, in order to take advantage of the greatly increased adsorptive power of activated charcoal at low temperatures. The effectiveness of the adsorber 20 can be judged by the odor of the residue left by the liquid oxygen when it evaporates. A garlic-like odor of this residue indicates the presence of hydrocarbons which have passed through the adsorber, and the necessity for removing the adsorbent and replacing it with fresh material.

Further explanation of the drawings and the various parts shown therein appear in the following description of the operation of the apparatus, wherein actual operation data are also given by way of example without however being intended as limiting.

Air from the compressor, after passing through any required purification apparatus such as that shown in Fig. 4 and with whatever cooling is required to bring it to about room temperature, is led into the apparatus of this invention through pipe 11. By way of specific example, 300 lbs. per hour of air at 2980 p. s. i. and 68° F. was so introduced through pipe 11 during normal running and the further operation of this apparatus will be described with reference to the handling of that quantity and condition of air, said air being substantially entirely free from water, water vapor, liquid oil, and carbon dioxide and in fact all ordinary contaminants except hydrocarbon vapors. The compressed air from pipe 11 passes through valve 13, which is normally in open position, into pipe 12 and thence into central tube 36 of heat exchanger 10 where it gives up heat by out-of-contact countercurrent heat exchange with effluent nitrogen which enters the heat exchanger 10 from pipe 15 and passes through the heat exchanger in the space between tube 36 and sheath 38, emerging through line 14 at a pressure of about one atmosphere and a temperature of about 55° F. and with an oxygen content of about 16.5%. The cold compressed air passing out of heat exchanger 10 through pipe 16, and at about 2890 p. s. i., has a temperature of about

—111° F. Thence it passes into the reboiler coil 17 located in a body of liquid oxygen within the reboiler 18 where it is still further cooled, emerging through pipe 19 at a temperature of about —272° F. and a pressure of about 2870 p. s. i. The cold compressed gas passing out of the reboiler coil through pipe 19 into and through absorber 20 is freed from any hydrocarbons present and passes without significant change in pressure or temperature to the entering point of the expansion capillary 22. There it enters the bottom of the coil of tubing 63 in arm 22', passes to the top of said arm at point 65 and then passes through line 23 to the bottom of the coil of tubing 63 in the second arm 22'' of the expansion capillary. After passing through this tubing it emerges at the exit end 64 at the top of the capillary, passing from there through pipes 26 and 29 into the cyclone separator 31. Valve 25 is kept closed during normal running operations. In passing through the expansion capillary 22 the air is reduced in pressure to about 42 p. s. i. and in temperature to about —300° F. At this temperature a considerable amount of the oxygen in the air has been liquefied. Separation between nitrogen and oxygen constituents takes place, according to well known principles, in the rectifying column 30; the nitrogen, in gaseous form, and containing about 16.5% of oxygen, passes out through pipe 15 into heat exchanger 10 at about 42 p. s. i. and —300° F., where it cools the incoming air as already described. Liquid oxygen from the column falls downwardly into reboiler 18, whence it passes by pressure up through pipe 33 and valve 34 out of the apparatus and to any suitable container. The oxygen so taken off is in liquid form and is about 98.5% pure; the yield is about 14 lbs. per hour. It will be noted that valve 34 is located within the vacuum jacket 60 and is controlled by a handle 49 located outside of the vacuum jacket; this is done in order to avoid, as much as possible, the loss of cold which would occur if valve 34 were placed outside the vacuum jacket 60.

As already stated, operation of the apparatus is very simple and can be readily carried on by inexperienced personnel. During operation it is merely necessary to keep track of the liquid level in reboiler 18 as shown by the liquid level indicator 32 and to see that the capillary 22 is not plugged, as indicated by stoppage in flow of effluent nitrogen from pipe 14—in addition to changing the purifying elements as they become exhausted. The liquid level in reboiler 18 is controlled by valve 34 which may be closed if the level falls too low. Plugging of the capillary 22 is cleared by opening valve 28 to let the warming air pass through tube 27, 27' as already described.

The warming air is expanded (by opening valve 28) after passing through tube 27, 27' rather than before as it is obvious that the cooling due to such expansion reduces the temperature of such air so much as to make it unsuitable as a warming medium.

To start up the apparatus the procedure already described is followed in all essential particulars except that valve 25 is open and valve 34 is closed, while valve 13 remains open and valve 28 closed as in normal running. The opening of valve 25 by-passes the second arm 22'' of expansion capillary 22 in the manner and for the reasons already set forth. Valve 34 is kept closed until sufficient liquid oxygen is accumulated in

reboiler 18 as shown by the liquid level indicator 32.

An apparatus capable of handling the volume of air and products referred to in the foregoing illustration and also including the purification system shown in Fig. 4 can readily be constructed to weigh less than 500 lbs., occupying a floor space 2' by 2½' and with a height of 5 to 6'.

I claim:

1. An apparatus for separating air into constituents, comprising a heat exchanger, an expansion capillary, a rectifier, means for introducing compressed air into said heat exchanger, means for passing said compressed air from said heat exchanger into said capillary, means for passing said air, after expansion in said capillary, into said rectifier for separation into liquid and gaseous components, and means for passing said gaseous components through said heat exchanger: said heat exchanger comprising two concentric tubes and a plurality of additional tubes each having an axis substantially parallel to those of said concentric tubes and each being mechanically attached to each of said concentric tubes, and said capillary having a length of at least 150 times its inside diameter and being associated with means for passing relatively warm air periodically in out-of-contact heat-exchange relationship with the expanding air in said capillary to melt any solidified obstructions to the passage of air through said capillary.

2. In an apparatus for separating air into constituents, wherein cooled compressed air is expanded to further cool the same, a capillary expansion element for expanding said compressed air, said element having a length of at least 150 times its inside diameter, and means associated with said element for passing relatively warm air periodically in out-of-contact heat-exchange relationship with the expanding air in said element to melt any solidified obstructions to the passage of said expanding air through said element.

3. Apparatus according to claim 2, wherein said means for passing relatively warm air is in the form of a tube, about which said capillary expansion element is wound and to which said element is mechanically attached.

4. Apparatus according to claim 2, wherein said capillary expansion element is provided with a by-pass intermediate its ends whereby air expanding in said element may be taken off without completing passage through the entire length of said element.

5. In a process for treating air, the steps which comprise cooling compressed air by heat exchange with the products of rectification, further cooling said cooled compressed air by expansion through a capillary of suitable length and diameter to reduce said air to nearly atmospheric pressure and to a temperature below the boiling point of oxygen, rectifying said further cooled air into a gaseous portion which is mostly nitrogen and a liquid portion which is substantially pure oxygen, bringing said portions separately into said heat exchange with said incoming compressed air, continuing said steps until such operation is substantially prevented by the accumulation of congealed obstructions in said capillary, and then passing a portion of said incoming compressed air into out-of-contact heat-exchange relationship with the contents of said capillary to melt said obstructions.

6. In a process for the treating of gases by expansion through a capillary expansion element, the steps which comprise first, passing compressed gas through said element to cool and expand said gas until substantial obstruction to the passage of said gas is created by the accumulation of congealed matter in said element, and second, passing a portion of said compressed gas into out-of-contact heat-exchange relationship with the contents of said element for a sufficient time to melt said congealed matter, and then alternately repeating said first and second steps as often as required.

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REFERENCES CITED

The following references are of record in the file of this patent:

UNITED ST .TES PATENTS

Number	Name	Date
1,919,500	Carpenter	July 25, 1933
1,968,518	Fraser	July 31, 1934
2,000,906	Turner	May 14, 1935
2,089,543	De Baufre	Aug. 10, 1937
2,137,260	Boles	Nov. 22, 1938

FOREIGN PATENTS

Number	Country	Date
559,109	France	June 7, 1923