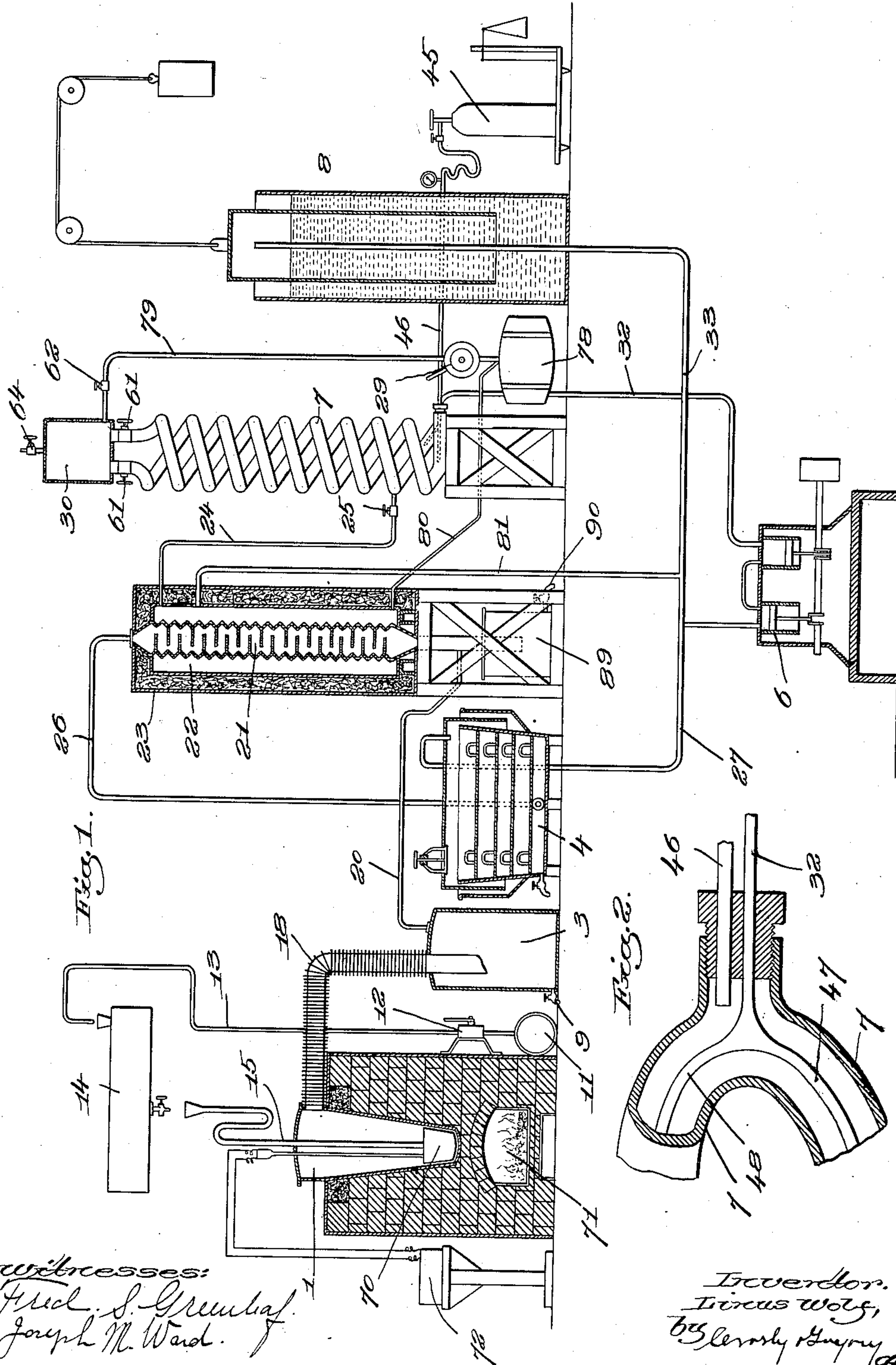


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AND METHOD OF MAKING SAID GAS.  
APPLICATION FILED OCT. 1, 1908.

928,729.

Patented July 20, 1909.





# UNITED STATES PATENT OFFICE.

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## LIQUID GAS SUITABLE FOR ILLUMINATING AND HEATING PURPOSES AND METHOD OF MAKING SAID GAS.

No. 928,729.

Specification of Letters Patent.

Patented July 20, 1909.

Application filed October 1, 1908. Serial No. 455,707.

*To all whom it may concern:*

Be it known that I, LINUS WOLF, a subject of the Emperor of Germany, residing at Boston, county of Suffolk, and State of Massachusetts, have invented an Improvement in Liquid Gas Suitable for Illuminating and Heating Purposes and Methods of Making said Gas, of which the following description, in connection with the accompanying drawing, is a specification, like figures on the drawing representing like parts.

This invention relates to the production of an illuminating and heating gas in liquefied form.

One of the objects of the invention is to provide a new liquefied gas which is capable of being manufactured from crude oil, petroleum, bitumen, natural gas, and other raw material; which when put under a pressure of three or four or less atmospheres at normal temperature will rapidly assume the gaseous form without leaving any liquid residue; which can be safely transported in its liquefied form; and the vapor of which is non-poisonous and can be safely used for either illuminating or heating purposes.

Another object of the invention is to provide a novel and inexpensive method of manufacturing such a gas from raw material.

The distillation vapors produced by the dry distillation of crude oil, petroleum, bitumen, and similar raw material, contain hydrogen and hydrocarbons of the methane, ethylene, acetylene, and other series beginning with those above mentioned members and rising to those of a higher carbon content and a correspondingly higher boiling point. These hydrocarbons may be for convenience roughly divided into the following four classes:

Class 1. Those substances which are in solids or in liquid form at ordinary temperatures and the boiling point of which is well above ordinary temperature. This class is represented by most of the petroleum products of commerce, such as kerosene, lubricating oils, vaseline, paraffin, tar, etc.

Class 2. Those substances boiling at or near ordinary temperature, including those that are readily liquefied by moderate cooling or slight pressure. Examples of this

class are gasolene, benzin, naphtha, and other easily-vaporizable liquid hydrocarbons.

Class 3. Those substances which are gaseous at ordinary temperature and pressures but which can be liquefied by vigorous refrigeration or by moderate refrigeration, and the application of pressure. Examples of this class are acetylene, propane, pentane etc.

Class 4. Those gases which can be liquefied only by extremely high pressure in connection with excessively high refrigeration. Examples of this class are hydrogen, methane, etc., gases which because of the difficulty with which they can be liquefied are commonly referred to as permanent gases.

It should be remembered that the above is merely a rough classification and that there is no sharp line between the different classes, but that, on the other hand, they graduate imperceptibly into each other.

My new liquefied gas is one secured, first, by removing as completely as possible from the distillation products of crude oil petroleum, bitumen, or other suitable raw materials, or from natural gas, those substances belonging to Classes 1 and 2 as above described; second, by liquefying as completely as possible those substances belonging to Class 3; third, by condensing the remaining unliquefied portions of Class 3 and causing them to be mixed with the liquefied gas; and lastly, separating the excess of the substances of Class 4. The new product which I thus obtain is a true liquefied gas which, if at ordinary temperature, the pressure upon it be lessened to not more than a few atmospheres (four to five) will rapidly and completely assume the gaseous form without leaving any liquid residue.

In manufacturing a liquefied gas of this character from raw material, such, for instance, as heavy oil, paraffin oil, crude petroleum, bitumen, etc., I first distil such raw material to obtain therefrom distillation gases, and after separating or condensing therefrom those substances, such as tar, belonging generally to Class 1 above described, the distillation gases are treated to eliminate all those constituents which belong generally to Class 2 and which comprise the easily-vaporizable liquid hydrocarbons. This step in the process can conveniently be done by chilling or cooling the gases to a marked de-



gree, as would be the case if they were cooled to  $-25^{\circ}\text{C}$ . to  $-30^{\circ}\text{C}$ ., the chilling of the gases to this point resulting in converting into liquid form the constituents represented by Class 2. The gas remaining after eliminating the above-described liquefiable constituents is then further treated so as to liquefy those constituents represented by Class 3, and the excess permanent gases are then separated from the product, thus leaving a liquefied gas embodying the invention.

While it is possible to secure the separation of the permanent gases from the liquefied gas in various ways, I prefer to accomplish this by forcing the gas while at a low temperature into a body of gasoline, for I find that where this procedure is adopted, the permanent gases are more easily separated from the liquefied gas and at the same time any heavy liquefiable hydrocarbons which would naturally be carried off with the permanent gases are washed out from the gases by the gasoline and are thus liquefied and added to the body of liquefied gas from which the permanent gases are separated. During this latter process some small amount of the gasoline into which the liquefied gas is forced becomes mixed with the product but the amount of gasoline thus mixed is so small that it is gaseous at ordinary temperatures even under several atmospheres pressure and therefore the presence of this small amount of gasoline does not result in the production of any liquid residue when the liquefied gas is vaporized. It is possible to treat natural gas according to this same method and to produce therefrom a liquefied gas capable of being used successfully in heating and illuminating.

My invention can be explained more in detail by referring to the accompanying drawings which are illustrative of the character of an apparatus which may conveniently be adopted to carry out my method, it being understood that the method is not confined to the use of the apparatus herein shown, but may be carried out by other apparatus, and also that the device herein illustrated is not essential for the production of the resultant product. In the apparatus herein shown the desired low temperature of the gas is secured either by the expansion of some of the highly-compressed or liquefied gas, or by the expansion of some of the gasoline which is under a heavy pressure. The gas is cooled in this way partly because a greater cooling effect can be produced than is possible with ordinary water-cooling processes, and partly because it is more economical to cool the gas in this way than by other known processes.

Referring now to the drawings wherein I have shown for the purpose of illustration an apparatus capable of carrying out the process, Figure 1 is a diagrammatic view of

the apparatus as a whole, and Fig. 2 is a detail hereinafter referred to.

The apparatus herein shown includes a distilling device which is used where the raw products to be used in making the liquefied gas are crude oil, bitumen, etc. When the liquefied gas is to be made from natural gas, this distilling device will not be necessary. The distilling device comprises a retort 1 having therein a pan or receptacle 70 in which the oil or other product to be vaporized is received. This retort is arranged to be heated by a suitable furnace 71 and the temperature of the furnace may conveniently be controlled by any usual or suitable thermostat which is shown more or less diagrammatically at 72. The retort is supplied with oil in any usual way as, for instance, by providing a pipe 15 leading into the retort, which in turn is supplied with oil from a tank 14. A pump 12 connected to the tank by a supply pipe 13 serves to keep the tank filled with oil from a main supply 11. The retort has connected thereto a pipe 18 through which the oil vapor or distillation gas formed in the retort is delivered to a tank 3 in which the tar and other similar heavy products belonging to Class 1 above referred to are condensed and accumulate. Such products may be drawn off from time to time through the valve 9. The tank 3 has connected thereto a pipe 20 leading to a gas-cooling apparatus which is capable of cooling the gas to a marked degree. In fact, I have found from experiments that the gas enters the cooling apparatus at about  $125^{\circ}\text{C}$ . and emerges from the cooling apparatus at about  $-25^{\circ}\text{C}$ . This cooling apparatus comprises a central chamber 21 into the lower end of which the pipe 20 leads and which is inclosed within a casing 22 that is surrounded by a covering 23 of insulating material. The central chamber 21 is open at its bottom and communicates with a closed tank or chamber 89 which is provided with the valve 90. The distillation gases passing up through the chamber 21 are rapidly cooled to a comparatively low temperature, as above specified, and during such cooling process those hydrocarbon constituents of the distillation gases which belong to Class 2 as above defined, and which comprise the easily-vaporizable liquid hydrocarbons are condensed or liquefied and accumulate in the tank 89 from which they may be removed from time to time through the valve 90. The upper end of the chamber 21 is connected by a pipe 26 with a gas purifier 4 which is for the purpose of removing from the gas any sulfureted hydrogen, and other impurities. The purifier is connected by a pipe 27 with a compressor 6 and also by a pipe 33 with a gasometer 8, this gasometer acting as a balance in usual manner. The compressor is also connected by a pipe 32



with the gas-separating apparatus in which the permanent gases, or those belonging to Class 4 as above defined, are separated from the mass of liquefied distillation gas. This gas-separating apparatus is in the form of a closed chamber adapted to contain gasoline, petroleum, or other oil, and while the chamber may have any appropriate shape, I have herein shown it in the form of two spiral or serpentine coils 7 which are connected together at the bottom and which are both connected to a chamber 30 at the top. The pipe 32 leads into the lower ends of the coils, as shown in Fig. 2, and is branched within the coils, as at 47, 48, the two branches extending a short distance above the bottom of the coils. The lower end of the coils is connected by a pipe 46 with the tank 45 into which the liquefied gas is to be stored.

78 is a tank or chamber adapted to hold gasoline, petroleum or other oil which is used in the coil 7, said tank being connected to the chamber 30 by means of a pipe 79 having a valve 62 therein.

29 is a pump arranged to force the gasoline from the tank 78 into the chamber 30. Communication between the chamber 30 and the coils 7 can be controlled by means of two valves 61.

My improved process is carried out by means of the above-described apparatus in the following manner: The distillation gases which are produced in the retort 1, or the natural gas from which the product is to be made, are first carried over through the pipe 18 into the tank 3, at which point the tar and other similar heavy products belonging to Class 1 as above defined are condensed and removed from the gas. The gas passes from the tank 3 to the chamber 21 of the cooling apparatus where its temperature is reduced to approximately  $-25^{\circ}$  C. to  $-30^{\circ}$  C. As stated above, during this cooling process, those constituents of the distillation gas which belong generally to Class 2 as above defined, that is, those constituents which will liquefy at atmospheric pressure and a temperature to which the gas is cooled, become liquefied and drain into the tank 89, and thus become separated from the other constituents belonging generally to Classes 3 and 4 as above defined. The constituents thus separated have the general characteristics of gasoline and include all the easily-vaporizable liquid hydrocarbons. The remaining cooled gas passes while still at its low temperature into and through the purifier 4 wherein the noxious and poisonous gases, such as sulfureted hydrogen, etc. are removed, without, however, appreciably increasing the temperature of the gas. The gas while still cool passes to and through the compressor 6 which is arranged to compress it to approximately 1500 pounds to the square inch. The heat of compression gen-

erated in the compressor may be removed in any suitable way, as by water cooling the compressor. The compressed gases are then delivered to the closed chamber which has previously been partially filled with gasoline or some other petroleum oil, the gasoline preferably filling the coils 7 substantially to the top thereof. During the operation the valves 62 and 64 are closed. The compression of the cooled gas to 1500 pounds results in liquefying substantially all the constituents except the permanent gases, such as methane and hydrogen, that is, such compression results in liquefying substantially all the constituents of Class 3 as above defined, while leaving the constituents belonging to Class 4 still in gaseous form. When liquefied gas thus obtained is at a low temperature and under a high pressure, as above specified, it has a greater specific gravity than gasoline, and consequently it settles to the bottom of the chamber 7 while the gasoline floats on top. The permanent gases which are not liquefied bubble up through the gasoline and pass into the chamber 30. These permanent gases, however, tend to carry with them a certain proportion of the heavy hydrocarbon constituents belonging to Class 3, and which are capable of being liquefied, and as such permanent gases with the liquefiable hydrocarbon constituents pass up through the gasoline, petroleum, or other oil in the chamber 7, such hydro-carbon constituents are washed out of the permanent gases by the gasoline, petroleum or other oil, and unite with the liquefied constituents at the bottom of the coils 7. During this washing process possibly a certain small proportion of the gasoline or petroleum mixes with the hydrocarbons that are washed out of the permanent gases, and such small proportion of the gasoline becomes mixed with the liquefied gas. The liquefied gas which accumulates at the bottom of the coils is drawn off into the tank 45 through the pipe 46 in usual manner. The product which is drawn off in the tank 45 is a liquefied gas formed by liquefying those constituents of distillation gas, or natural gas, which are left after the more easily liquefiable constituents belonging to Class 2 and the permanent gases belonging to Class 4 are removed. The liquefied gas thus obtained comprises principally ethane and ethylene and the next higher homologues of ethane, although a careful analysis of the gas shows that it has a small trace of methane and sometimes a small trace of benzol. The liquefied gas is of such a nature that when it is allowed to flow out from the tank at atmospheric pressure and normal temperature the entire body of liquid in the tank becomes vaporized and no liquid residue remains in the tank after the vapors have escaped. This is an exceedingly important feature because the presence of a



liquid residue in the tank is a source of great danger owing to the fact that such liquid residue will gradually vaporize and thus form a very explosive mixture. The vapor  
 5 formed from the liquefied gas above described is capable of developing an extremely high heat when burned under proper conditions and is also an extremely good illuminant. The gas produced from the vaporization of the liquid is non-poisonous and  
 10 is much less likely to form an explosive mixture with air than is acetylene or ordinary illuminating gas.

My improved method also includes a  
 15 novel way of cooling the gases before they pass to the purifier by which the extremely low temperature of  $-25^{\circ}\text{C}$ . to  $-30^{\circ}\text{C}$ . can be readily obtained at a comparatively slight expense. This is done by utilizing for this  
 20 purpose the cooling effect of the expansion and vaporization of the highly-compressed gasolene or other oil in the coils 7. As shown in the drawings the upper end of the casing  
 22 is connected by a pipe 24 to the coil 7 at  
 25 a point above the lower end thereof, said pipe having therein an ordinary reducing valve 25. Another pipe 80 connects the lower end of the casing 22 with the tank 78  
 and still another pipe 81 connects the casing  
 30 22 at a point below its upper end with the pipes 27 and 33. The pipe 24 is preferably connected to the coil 7 at a point slightly above the division line between the liquid  
 gas and the gasolene or petroleum. It will  
 35 be understood that during the operation of the apparatus the gasolene in the coil 7 is subjected to the high pressure developed by the compressor and a portion of this compressed  
 gasolene which has some of the liquefied  
 40 gas constituents associated therewith escapes through the reducing valve 25 and expands and vaporizes within the casing 22, thus cooling the chamber 21 and the gases  
 passing up therethrough. The cooling effect  
 45 which can thus be produced by the expanding gas is sufficient to reduce the temperature of the distillation gases to approximately  $-25^{\circ}\text{C}$ . to  $-30^{\circ}\text{C}$ . The vapors formed  
 by this expansion of the gas are returned  
 50 to the compressor through the pipe 81 while the gasolene liquid which is not vaporized or which is formed from the condensation  
 of the vapor in the casing 22 is returned to  
 55 the tank 78 through the pipe 80. This manner of cooling the gas I consider as an im-

portant feature of the process as it enables me to secure rapidly and at a minimum expense the extremely low temperature desired.

While the apparatus shown is such as could be used in liquefying distillation gases,  
 60 the process can also be used for producing my improved liquefied gas from natural gas.

Having fully described my invention, what I claim as new and desire to secure by Letters Patent is:—  
 65

1. The process of producing a liquefied gas which consists in producing distillation gases, liquefying all such constituents thereof as will liquefy at atmospheric pressure and at a temperature of approximately  
 70  $-25^{\circ}\text{C}$ . and which in liquid form include the easily-vaporizable liquid hydrocarbons, separating all the liquid thus obtained from the remaining constituents and liquefying all said remaining constituents except the  
 75 permanent gases.

2. The process of producing a liquid gas from distillation gases which consists in cooling the distillation gases to approximately  
 80  $-25^{\circ}\text{C}$ ., thereby to liquefy all those constituents which comprise the easily-vaporizable liquid hydro-carbons, separating the liquid thus obtained from the remaining constituents, and subjecting the remaining  
 constituents to a pressure sufficient to liquefy  
 85 all the gases except the permanent gases.

3. The process of producing a liquid gas from distillation gases which consists in cooling the distillation gases to approximately  
 90  $-25^{\circ}\text{C}$ ., thereby to liquefy all those constituents which comprise the easily vaporizable liquid hydro-carbons, separating the liquid thus obtained from the remaining constituents, and forcing all the remaining  
 constituents into a chamber filled with gaso-  
 95 lene under a pressure of approximately 1500 pounds.

4. A liquid gas which comprises liquefied hydrocarbon-constituents of distillation gases which are entirely free from those constitu-  
 100 ents which can be liquefied at atmospheric pressure and at a temperature of approximately  $-25^{\circ}\text{C}$ .

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

LINUS WOLF.

Witnesses:

BERTHA F. HEUSER,  
 FREDERICK S. GREENLEAF.