United States Patent

[11] 3,633,371

[72] Inventor **Joseph W. Davison**
Bartlesville, Okla.

[21] Appl. No. **719,032**
[22] Filed **Apr. 5, 1968**
[45] Patented **Jan. 11, 1972**

[73] Assignee **Phillips Petroleum Company**

[54] **GAS SEPARATION**
4 Claims, 1 Drawing Fig.

[52] **U.S. Cl.** **62/17,** 62/20, 62/23, 55/48, 55/84

[51] **Int. Cl.** **F25j 3/05,** F25j 3/00

[50] **Field of Search** 55/48, 86, 84; 62/17, 23, 24, 27, 28

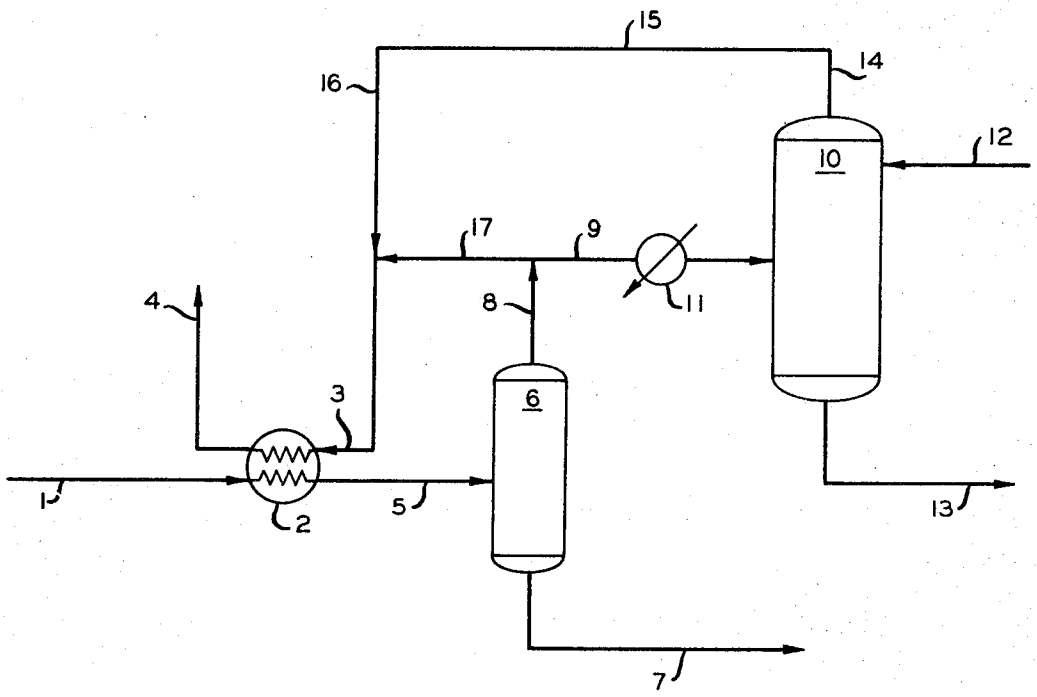
[56] **References Cited**

UNITED STATES PATENTS

2,603,310	7/1952	Gilmore	62/17
2,765,635	10/1956	Redcay	62/17
2,775,103	12/1956	Koble	62/17
2,815,650	12/1957	McIntire	55/48
2,933,901	4/1960	Davison	62/17
3,359,743	12/1967	Napoli	62/23
3,247,649	4/1966	Miller	55/48

Primary Examiner—Norman Yudkoff
Assistant Examiner—Arthur F. Purcell
Attorney—Young and Quigg

ABSTRACT: A method for recovering ethane and heavier from a normally gaseous material by partially condensing the gas by cooling to a temperature in the range of from about −70° to about −120° F., removing uncondensed gas with an absorbent at a temperature in the range of from about −70° to about −120° F.



INVENTOR.
J. W. DAVISON

BY Young & Quigg

ATTORNEYS

GAS SEPARATION

This invention relates to a new and improved method for recovering ethane from a normally gaseous material.

Heretofore cooling gases to at least partially condense same has been used as a type of a reverse distillation procedure.

According to this invention maximum recovery of ethane from a normally gaseous material at minimum cost per volume of ethane recovered is achieved by first cooling the gaseous material to a temperature in the range of from about −70° to about −120° F., said temperature being sufficient to liquefy a portion of said gaseous material and thereby produce an ethane and heavier rich liquid, separating the liquid from the remaining, uncondensed gas, and then contacting at least a portion of the remaining gas with an absorbent as hereinafter defined to remove additional ethane from the gas, the absorbing step being carried out at a temperature in the range of from about −70° to about −120° F.

By this procedure separation of ethane by liquefaction thereof is practiced to the point where maximum amounts of ethane as well as heavier hydrocarbons are liquefied and past which point the additional ethane liquefied would not be worth the cost of obtaining the necessary lower temperatures, i.e., the cost of the additional refrigeration necessary. After the cooling step the nonliquefied gaseous portion can be treated with certain absorbents while still in the temperature range of from about −70° to about −120° F. and additional ethane thereby recovered from the gas. In this manner maximum ethane recovery is achieved by using the partial condensation technique only to the extent that the amount of ethane recovered thereby justifies the refrigeration cost and thereafter still more ethane is recovered by a less expensive absorbing step. It is important to note that the absorbing step is carried out in the temperature range of from about −70° to about −120° F. since this procedure insures maximum removal of ethane by the absorbent.

By the procedure of this invention, a maximum amount of ethane is recovered from a gas in the most economic manner because the refrigerating step is employed only to the extent that it remains economical based on the amount of the ethane recovered thereby, and thereafter additional ethane is recovered by using a less expensive absorbing step. However, the absorbing step is carried out on the already refrigerated gas thereby obtaining maximum benefit of (1) the refrigeration step since the absorbent is, because of its refrigerated condition, in a state of increased capacity for ethane and also since the ethane is tending toward liquefaction, and (2) the function of the absorbent.

The ethane produce of this invention can be dehydrogenated using known procedures and the resulting ethylene employed as a monomer in known polymerization procedures to produce polyethylene. The polyethylene so produced is useful for making with known techniques such as blow molding a wide variety of articles of commerce such as bottles, e.g., baby bottles, wastepaper baskets, and the like.

Accordingly, it is an object of this invention to provide a new and improved process for separating ethane from normally gaseous materials.

It is another object of this invention to provide a new and improved process for removing a maximum amount of ethane from a gas at minimum cost by utilizing partial condensation techniques to the extent that they remain economic and thereafter using an absorbing step on the already refrigerated gases, thereby taking advantage of the increased efficiency of the refrigeration and absorbing steps at the same time in the absorbing step.

Other aspects, objects and the several advantages of this invention will become apparent from a study of the disclosure, drawing and appended claims.

The drawing shows a system which embodies this invention.

In the drawing, there is shown line **1** which carries the feed to the system.

The feed can be any conventional ethane-containing gas such as natural gas or a gasoline plant residue gas. Such gases are well known in the art. Generally, the feed gas can contain

at least 50 volume percent of methane, ethane, and propane, the remainder being essentially butane and heavier saturated and unsaturated normally gaseous hydrocarbons, nitrogen, carbon dioxide, helium, water, and the like. The amount of water present should not be sufficient to cause plugging of the apparatus by the formation of ice crystals. The methane, ethane, and propane present in the feed gas are each present in substantial amounts. For example, of a mixture of methane, ethane, and propane present in the feed gas, at least 50 volume percent can be methane, the remainder being from about 5 to about 25 volume percent each of ethane and propane.

The feed in line **1** passes to heat exchanger **2** which can be composed of one or more conventional heat exchange units and/or one or more conventional refrigeration units. The heat exchange system **2** is designed to cool the feed gas to a temperature in the range of from about −70° to about −120° F.

As shown in the drawing, recycle gas in line **3** can pass through the system. This is advantageous because it conserves refrigeration expenses by utilizing the already cooled gas after it is through the absorbing treatment, thereby cooling the feed and also heating the already treated gas before it is passed to a pipeline or other disposition by way of line **4**. It should be noted that the feed gas can be cooled in heat exchange system **2** by a combination of cooling steps such as by first being cooled by coming in heat exchange relationship with already treated and refrigerated gas in line **3**, and thereafter being further cooled by other heat exchange relationships and/or refrigeration processes which are not related to nor dependent upon the use of the gas in line **3**.

The cooling step should reduce the temperature of the feed gas into the range set forth hereinabove, and should also liquefy at least a portion of that feed gas, preferably from about 5 to about 95 volume percent of the feed gas being processed. This partial liquefaction step in the temperature range recited will produce an ethane and heavier rich liquid in contact with a methane rich gas. This gas also contains recoverable amounts of ethane.

The partially condensed feed stream then passes by way of line **5** to separator **6** wherein the uncondensed gas is allowed to separate and collect at the top of the separator and the liquified portion of the feed gas is allowed to collect at the bottom of the separator and is removed by way of line **7**.

The liquid removed by way of line **7** is rich in ethane and heavier hydrocarbons. This liquid can be subjected to a conventional demethanization distillation process to remove any methane present in the liquid, thereby leaving a liquid consisting essentially of ethane, propane, and any heavier components that happen to be in the feed gas and that boil at temperatures higher than propane. The demethanized liquid can then be subjected to another distillation step wherein substantially pure ethane is recovered as an overhead product.

The overhead product from separator **6** passes by way of line **8** and line **9** into absorbing zone **10**. It is important to maintain the refrigerated status of the overhead gas passing into absorbing zone **10** in order to make the most advantage of the prior refrigeration step when carrying out the absorbing step. If in the transfer of the overhead gas from separator **6** to absorbing zone **10** the gas happens to be heated to a temperature outside of the range of −70° to −120° F. an additional cooling system **11** can be employed to cool that gas so that the gas in the absorbing zone **10** is at a temperature in the range of from about −70° to about −120° F.

The gas in absorbing zone **10** tends to pass upwardly and in so doing is contacted countercurrently with downwardly passing absorbing liquid from line **12**.

The absorbent employed is precooled to substantially the same temperature as the gas present in the absorbing zone, and therefore is at a temperature in the range of from about −70° to about −120° F. The absorbent employed is a liquid at the temperature of the gas present in the absorbing zone and consists essentially of saturated hydrocarbons having from four to eight carbon atoms per molecule, inclusive, preferably

hexanes such as normal hexane. Mixtures of these hydrocarbons can be employed also.

The downwardly passing absorbent very readily absorbs ethane from the upwardly passing refrigerated gas because of the high capacity of the absorbent for ethane at low temperatures. Thus the contacting of the thus-refrigerated ethane with the liquid absorbent maximizes the removal of ethane from the gas into the absorbent.

The liquid absorbent rich in ethane is removed by way of line **13** for further treatment such as distillation to remove ethane therefrom as an overhead product.

The combination of ethane removed by way of lines **7** and **13** constitutes the ethane product of this invention, and will generally represent from about 5 to about 95 volume percent of the ethane present in the feed gas in line **1**. By following the procedure of this invention an ethane extraction as deep as 70 to 80 volume percent of the ethane present in the feed gas can be obtained at costs substantially less than using other techniques such as absorption or partial condensation by themselves.

Separator **6** can be operated at a pressure substantially the same as the pressure of the feed gas in line **1**, taking into account pressure drops caused by friction and the like in transporting the feed gas through the refrigeration system **2** and into separator **6**. Similarly, absorbing zone **10** can also be operated at substantially the same pressure as the pressure in separator **6** also taking into account normal pressure drop occurring in lines **8** and **9**, and cooling system **11** if present.

Alternatively, separator **6** can be operated as a flash unit in that the pressure maintained in that separator can be substantially less than the pressure of the feed gas in line **1**, thereby causing partial volatilization of the liquid passing into separator **6** and additional temperature reduction. This partial volatilization will decrease the amount of methane carried by the liquid that passes from the separator by way of line **7**. Generally, the pressure decrease in separator **6** below the pressure of the feed gas in line **1** is at least 10 p.s.i.g., preferably from about 10 to about 100 p.s.i.g. In this embodiment, the absorbing zone **10** is maintained at a pressure substantially the same as reduced pressure in separator **6**.

Apart of the overhead gas in line **8** and all of the overhead gas in line **14** can be disposed of at will such as by reheating and passing to a pipeline or other storage facility. However, for maximum economics, all of the refrigerated, overhead gas in line **14** is passed through lines **15**, **16**, and **3** into heat exchange relationship with the incoming feed gas from line **1** as discussed hereinabove. In addition, if desired, a portion of the refrigerated, overhead gas in line **8** can be passed through line **17** into line **16** and thereby through line **3** into the heat exchange system **2** to also serve to cool the incoming feed gas from line **1**. Normally, substantially all of he overhead gas in line **8** is passed through line **9** into absorbing zone **10**. However, any portion or all of the overhead gas in line **8** can be passed into line **17**. Preferably, at least 5 volume percent of the gas in line **8** passes into line **9** for treatment in absorbing zone **10**.

EXAMPLE

Two runs are made using the apparatus shown in the drawing and using different feed compositions for each run. The two different feeds are denoted herein as Feed A and Feed B, and the compositions of these feeds are given in detail hereinbelow in table I. Feed A contains about 3.4 mol percent ethane, the remainder being essentially methane, propane and heavier hydrocarbons; while Feed B contains about 12 mol percent ethane, the remainder being essentially methane, propane, and heavier hydrocarbons. The absorbent employed consists essentially of normal hexane.

Feed A is passed under pressure of 500 p.s.i.a. to cooling system **2**, cooled to a temperature of about −90° F., and thereafter passed into separation zone **6**. A portion of the overhead gas from separation zone **6** is passed into absorption zone **10** and therein is at a temperature of about −80° F. The absorbent is passed into absorption zone **10** also at a temperature of about −80° F. **Feed B is under pressure of** 500 p.s.i.a. is cooled in heat exchange system **2** to a temperature of about −70° F., and then passed to separation zone **6**. Part of the overhead gas from separation zone **6** is passed to absorption zone **10**. During this passage the gas is cooled to about −80° F. before being passed into the absorption zone. The absorbent, again consisting essentially of normal hexane, is precooled to a temperature of about −80° F. and is passed into the absorbing zone **10**.

In both runs, the pressures in separation zone **6** and absorbing zone **10** are substantially the same as the initial **500** p.s.i.a. pressure of the feed allowing of course for normal pressure drop in the pipes due to friction and the like.

The results of the runs are as follows:

TABLE I

	1 [1]	7 [1]	9 [1]	12 [1]	13 [1]	14 [1]	17 [1]
Feed A:							
Methane	252,358	3,305.5	224,147.2	0	32,364.7	191,782.5	24,905.3
Ethane	8,976	781.5	7,375.1	0	6,030.4	1,344.7	819.5
Propane +	2,666	1,265.2	1,260.7	0	1,260.7	0	140.1
n-Hexane	0	0	0	30,000	29,546	454	0
Feed B:							
Methane	224,440	16,908.9	186,778	0	26,303.3	160,474.7	20,753.1
Ethane	31,680	11,241.3	18,394.8	0	12,988.8	5,406	2,043.9
Propane +	7,920	6,360.7	1,403.3	0	1,403.3	0	155.9
n-Hexane	0	0	0	30,000	29,300.7	699.3	0

[1] Mols per day. The number heading the column corresponds to the reference number found in the drawing. For example, the column headed 1 stands for the composition of the material in line 1 of the drawing.

Reasonable variations and modifications are possible within the scope of this disclosure without departing from the spirit and scope thereof.

I claim:

1. A process comprising providing a feed gas containing at least 50 volume percent of a mixture of substantial amounts of each of methane, ethane, and propane; cooling said feed gas to a temperature in the range of from about −70° to about −120° F., said temperature and suitable pressure being sufficient to liquefy from about 5 to about 95 volume percent of said gas and thereby produce an ethane and heavier rich liquid; separating said ethane rich liquid from the remaining unliquefied gas in a separation zone; separately removing from said separation zone said unliquefied gas and said ethane rich liquid and thereafter recovering ethane from said ethane rich liquid as a product of the process and not returned thereto; passing at least a portion of the unliquefied gas which was separated from said ethane rich liquid to an absorbing zone which is maintained at a temperature in the range of from about −70° to about −120° F.; contacting said unliquefied gas in said absorbing zone with a precooled absorbent which is liquid at the temperature maintained in said absorbing zone and which consists essentially of saturated hydrocarbons having from four to eight carbon atoms per molecule, inclusive; separately removing from said absorbing zone gas rich in methane and liquid absorbent rich in ethane and thereafter separately recovering ethane from said liquid absorbent rich in ethane as a product of the process and not returned thereto.

2. The method according to claim **1** wherein said gas and liquid separating step and said absorbing step are each carried

out at substantially the same pressure as the initial pressure of said feed gas.

3. A method according to claim 1 wherein said gas and liquid separating step is carried out at a substantially lower than the initial pressure of said feed gas to cause flashing of a part of the liquefied feed gas, and said absorbing step is carried out at substantially the same pressure as said gas and liquid separation step.

4. A method according to claim 1 wherein said feed gas consists essentially of a finite amount up to 25 volume percent ethane, the remainder being essentially methane and propane; and the absorbent consists essentially of normal hexane.

* * * * *

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,633,371 Dated January 11, 1972

Inventor(s) Joseph W. Davison

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 5, line 4 (claim 3), before "substantially" insert -- pressure --.

Signed and sealed this 30th day of May 1972.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents

FORM PO-1050 (10-69)

USCOMM-DC 60376-P69
☆ U.S. GOVERNMENT PRINTING OFFICE : 1969 O—366-334