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(54) SEPARATION OF ETHYLENE FROM ETHANE

(71) We, SNAMPROGETTI SPA, an Italian company, of Corso Venezia 16, Milan, Italy, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process for separating ethylene from ethane. More particularly, the present invention relates to a process for separating ethylene from ethane when both ethylene and ethane are in admixture with methane, hydrogen, carbon monoxide, propane, propylene, propyne, propadiene and small amounts of C_4 hydrocarbons and heavier hydrocarbons, i.e. (C_5 and C_5^+). All hydrocarbons with C_4, C_5 and C_5^+ are generally indicated by C_3^+ in the present specification.

Such a mixture is generally known as "cracking gas" and may contain up to 10% by weight of C_4, C_5, C_5^+, although usually their total contents is much less. The cracking gas when used in the method according to the present invention is substantially free from acetylene since it has been previously hydrogenated and is substantially devoid of acidic components such as hydrogen sulphide and carbon dioxide and water, these having previously been stripped. Also a fraction of the C_4, C_5 and C_5^+ hydrocarbons may be eliminated prior to feeding the initial mixture to the process according to this invention.

A few processes are already known for the separation of ethylene from ethane when both ethylene and ethane are in admixture with the above enumerated other compounds. The plants for effecting two such known processes are shown schematically in Figures 1 and 2 of the accompanying drawings.

Referring firstly to Figure 1, a hydrocarbon mixture which contains ethylene and ethane is fed in a pipe 1 to a exchanger 2 where it is cooled to a temperature of about minus 40°C, after which it is fed to a separator 3 from which a gaseous fraction in a pipe 5 and a liquid fraction in a pipe 4 are withdrawn. The gaseous fraction is richer in hydrogen and carbon monoxide than the original mixture.

The gaseous fraction is conveyed in pipe 5 to a heat exchanger 6 where it is cooled to a temperature of about minus 80°C and it is then fed to a separator 8 for separating a liquid phase withdrawn in a pipe 7 and a gaseous phase which is withdrawn in a pipe 9 and is even richer in hydrogen and carbon monoxide. The gaseous phase in pipe 9 is further cooled, in a heat exchanger 10, to a temperature of about 100°C and the cooled product is separated in a separator 11 into a liquid phase withdrawn in a pipe 12 and a gaseous phase withdrawn in a pipe 13. The gaseous phase in pipe 13 is subjected to a further cooling in a cooler 14 to separate off hydrogen which is withdrawn in a pipe 15 and contaminated substantially only by methane and carbon monoxide, and a liquid phase which is withdrawn through a pipe 16 and which contains substantially only methane.

The liquid phases in pipes 4, 7, 12 and 16, obtained from the abovementioned four cooling steps, are fed to a distillation column 17 from which a head product is obtained in a pipe 18, which is composed substantially of methane, and a tail product, in a pipe 19, composed of all the other hydrocarbons.

The tail product in pipe 19 is fed to a distillation column 20 wherein the heavy products, C_3 and C_3^+, are separated off as tail products, while the head product is a stream in a pipe 21, which is essentially composed of ethylene and ethane.

This stream is fed to a rectification column 22 from which ethylene is obtained in a pipe 23 as the head product and ethane is obtained as the tail product in a pipe 24. A modification of such a diagram as outlined above is the one shown in Figure 2.

According to the known process effected in a plant as shown in Figure 2 of the accompanying drawings, the hydrocarbon stream in pipe 1, which contains ethylene and ethane, is fed, at the outset, to a distillation column having the same function as the distillation column 20 shown in Figure 1 as regards the removal of the C_3 and C_3^+ hydrocarbons. Thus the C_3 and C_3^+ hydrocarbons are removed first, as tail products.

The head product is then cooled through four consecutive cooling stages similarly to what has been described with reference to Figure 1, the first cooling step being carried out at a temperature of about minus 60°C, i.e. slightly below that used in the plant of Figure 1.

The four liquid streams which are obtained (in pipes 3, 4, 5 and 6) are fed, similarly to the previous pattern, to a distillation column 7 which corresponds to the column 17 in Figure 1, and from which is obtained, as the head product in a pipe 8, essentially methane and, as the tail product in a pipe 9, a stream which is mainly composed of ethylene and ethane.

The known processes described above involve a high initial cost and high consumption.

According to the present invention; there is provided a process for separating ethylene from ethane when ethylene and ethane are originally in admixture with methane, hydrogen, carbon monoxide, propane, propylene, propyne, propadiene and small amounts of hydrocarbons containing four or more carbon atoms, the process comprising the following steps:—

(a) subjecting the original mixture to at least one of distillation, stripping and flashing, to produce a relatively light product which contains all or substantially all the hydrogen and carbon monoxide present in the original mixture, substantially all of the methane originally present, from 20% to 80% of each of the ethylene and ethane originally present and traces of hydrocarbons having 3 or more carbon atoms, and a relatively heavy product composed of the residual hydrocarbons;

(b) cooling in one or more stages the relatively light product to a temperature below minus 100°C, thereby separating a gaseous phase composed mainly of hydrogen with minor amounts of methane and carbon monoxide and with traces of ethylene and ethane, and one or more liquid phases composed substantially of methane, ethylene and ethane with minor amounts of hydrocarbons having three or more hydrocarbons;

(c) feeding the liquid phase(s) produced in step (b) to, respectively, one or more points of a distillation apparatus, with or without a head condenser, thereby obtaining at the head a fraction which contains methane, hydrogen, and small amounts of carbon monoxide and ethylene, and at the bottom a mixture of ethylene and ethane in which the ratio by weight of ethylene to ethane is greater than the corresponding ratio in the original mixture;

(d) feeding the relatively heavy product of step (a) composed of C_2, C_3, C_4 and heavier hydrocarbons, to a distillation column, thereby separating at the bottom the C_3, C_4 and heavier hydrocarbons and, at the head, a mixture of ethylene and ethane in which the ratio by weight of ethane to ethylene is greater than the corresponding ratio in the initial mixture;

(e) feeding the two mixtures of ethylene and ethane produced in steps (c) and (d) to a rectification column, the mixture in which the weight ratio of ethylene to ethane is greater than that in the original mixture being fed at a point above that at which the other mixture is fed, to produce a head product composed of ethylene and a bottom product composed of ethane.

One convenient embodiment of the process of the present invention is that wherein the relatively light product is obtained by distillation or stripping in step (a) and is cooled in three stages, the first stage involving cooling to a temperature below −10°C to separate a liquid hydrocarbonaceous phase and a gaseous phase composed of hydrogen, carbon monoxide, methane, ethylene and ethane, the second stage involving cooling the gaseous phase from the first stage to a temperature below −80°C, to separate a liquid phase essentially composed of methane, ethylene and ethane and a gaseous phase composed of hydrogen, carbon monoxide, methane, ethylene and ethane, with the combined ethylene and ethane contents being up to 5% by volume of the gaseous phase, the third stage involving cooling the gaseous stream from the second stage to a temperature below −100°C, to separate a gas phase composed of hydrogen with carbon monoxide and methane in minor amounts, and a liquid phase composed of ethylene, ethane and methane, the three liquid phases from the three stages being fed, in step (c), to three points of the distillation apparatus.

Preferably, in this embodiment the first and second stages involve cooling to approximately −80°C and approximately −100°C, respectively.

Preferably the pressure obtaining during the distillation or stripping of step (a) is in the range from 20 to 50 atmospheres and the head temperature is in the range from 0°C to −60°C.

Another convenient embodiment of the process of the present invention is that wherein the relatively light product obtained by flashing in step (a) and is subjected to three consecutive cooling steps with separation of three liquid phases at approximately −100°C, approximately −120°C, and below −120°C, respectively.

Preferably the pressure obtaining during the flashing of step (a) is in the range from 7 to 15 atmospheres and the temperature is in the range from −40°C to −80°C.

Conveniently the relatively light and relatively heavy products in step (a) and the mixture of ethylene and ethane produced in step (d) are obtained in a single distillation

column, the relatively light product being obtained as a head product, the relatively heavy product being obtained as a bottom product, and the ethylene and ethane mixture being obtained as a lateral cut, respectively.

For a better understanding of the present invention and to show how the same may be carried into effect, reference will now be made, by way of example, to Figures 3 to 6 of the accompanying drawings in which each of Figures 3 to 6 represents diagrammatically a different plant for carrying out a different embodiment of the process of the present invention.

Referring firstly to Figure 3, a hydrocarbon stream which contains ethylene and ethane (in fact, cracking gas at a temperature of −50°C or below) is fed in a pipe 1 to a distillation column 2 which is operated under a pressure in the range from 20 to 50 atmospheres and at a head temperature of from 0°C to −60°C. From column 2 are withdrawn in a pipe 3 a bottom product composed of ethylene, ethane and all the C_3 and C_3^+ hydrocarbons, and in a pipe 4 a head product composed of carbon monoxide, hydrogen, methane, a portion of ethylene and ethane and traces of the C_3 hydrocarbon.

The head product in pipe 4 is cooled below −60°C in a heat exchanger 5 and is separated in a separator 6 into a liquid phase which is composed of the heavier hydrocarbons and is withdrawn in a pipe 7, and a gaseous phase which is withdrawn in a pipe 8, cooled in a heat exchanger 9 and separated in a separator 10 into a liquid phase withdrawn in a pipe 11 and a gaseous phase which is withdrawn in a pipe 12 and essentially composed of hydrogen, carbon monoxide and methane with minor amounts of ethylene and ethane.

The gaseous feed in pipe 12 is cooled in a cooler 13 at a temperature below −100°C, and from the cooler 13 are discharged, in a pipe 14, hydrogen and traces of methane and carbon monoxide, and, in a pipe 15, a liquid phase essentially composed of methane, ethylene and ethane.

The liquid phases in pipes 7, 11 and 15 are fed to a distillation column 16 from the head of which there is discharged in a pipe 17 a head product essentially composed of methane with traces of hydrogen and carbon monoxide, and from the bottom of which is discharged in a pipe 18 a stream of ethylene and ethane in which the weight ratio of ethylene to ethane is greater than that in the stream in pipe 1.

The tail product in pipe 3 is fed to a distillation column 19, from which is discharged in a pipe 20 all the C_3 and C_3^+ hydrocarbons, and from the head of which is obtained, in a pipe 21, a stream of ethane and ethylene in which the weight ratio of ethane to ethylene is greater than that in the stream fed in pipe 1.

The two streams each containing ethylene and ethane are fed separately to a rectification column 22, the stream in pipe 18 being fed at a point above the stream in pipe 21. From the rectification column 22 there is withdrawn, as an overhead in a pipe 23, ethylene of the required purity, and there is also withdrawn, as a tail product in a pipe 24, ethane of the required purity.

Referring now to Figure 4 of the drawings, the plant as shown therein is similar to that of the plant shown in Figure 3 except that the distillation column 2 has been replaced by a stripping column 2. Otherwise the components present and the operating conditions are the same.

The plant illustrated in Figure 5 of the accompanying drawings is similar to that illustrated in Figure 3, the only exception being that the distillation column 2 of Figure 3 is replaced by a separator 2 in which the initial hydrocarbon stream (which is fed in via pipe 1) can be flashed. The general technique described with reference to Figure 3 can be employed in relation to the plant of Figure 5 with particular advantage when the initial hydrocarbon stream in pipe 1 has a low pressure (from 7 to 15 atmospheres).

In such a case the mixture in pipe 1 is cooled to a temperature in the range from −40°C to −80°C and then flashed in the separator 2. The remaining steps can be the same as described with regard to Figure 3. In such a case, however, it might become necessary in the column 22 to withdraw ethylene, not from the head of column 22 (as shown in pipe 23 in Figure 3 and as shown by solid lines in Figure 5) but through a lateral discharge pipe shown as a broken line in Figure 5, if the quantity of methane present in column 22 is sufficiently large. In this event, the methane could be removed as the head product.

When operating the plant illustrated in Figure 5, the cooling effected in the heat exchangers 5 and 9 and in the cooler 13 preferably achieve a reduction in temperature to approximately −100°C, approximately −120°C and below −120°C respectively.

Figure 6 illustrates a plant which is a further modification of that illustrated in Figure 3. Except where otherwise stated, the operating conditions can be the same for the plant of Figure 6 as for the plant of Figure 3.

In the embodiment illustrated in Figure 6, the column 19 of Figure 3 and the column 2 of Figure 3 have been combined into a new column 2 to which the initial mixture is fed in a pipe 1. In the new column 2 the feed is split into a light fraction which is withdrawn through a pipe 4 and which is identical to the fraction withdrawn through the pipe 4 of the plant of Figure 3, and bottom stream which is withdrawn through a pipe 20 similar to the stream contained in the pipe 20 shown in Figure 3, and a stream withdrawn through a

lateral pipe 21 which has a composition similar to that of the stream in the pipe 21 of Figure 3. The stream in the pipe 21 of Figure 6 is fed to a rectification column similar to the column 22 shown in Figure 3.

The process according to the present invention affords the following advantages.

(i) The loss of ethylene in the methane fraction withdrawn through pipe 17 can be reduced.

(ii) The methane-stripping column 16 of Figure 3 can be reduced in size compared to a similar column in a plant of the prior art, because it handles from 20 to 80 percent less than the corresponding component in the prior art plants illustrated in Figures 1 and 2.

(iii) The reboiler of the column 16 shown in Figure 3 can be operated at a lower temperature, which can save energy.

(iv) The ethane-stripping operation, which is effected in the column 19 shown in Figure 3 is carried out on smaller quantities of hydrocarbons having two carbon atoms than in the illustrated prior art plants and, therefore, that column can be reduced in size and corresponding savings can be made on its reboiler.

(v) The feeding of one ethylene and ethane mixture to the column 22 (of Figure 3) which is richer in ethylene than the original feed and the feeding of another ethylene and ethane mixture which is richer in ethane than the original feed can result in a reduction of the reflux required for column 22, and in certain cases this reduction can be as significant as 20%.

(vi) The splitting of the feed mixture into two streams, in column 2 shown in Figure 3, has the advantage of removing methane from the heavier bottom product of that column.

For the purpose of comparison, there is set out in Table 1 the flow rate (expressed in normal cubic meters per hour) of various compounds in different components of the plant of Figure 1, forming part of an installation having an annual output of 500,000 metric tons of ethylene. Table 2 is similar but relates to the flow rate when carried out in the plant of Figure 4. By comparing the data in tables 1 and 2 it can be seen that there is a reduction of 59% of the ethylene lost in the methane fraction, in the case of the plant according to Figure 4.

Also, the quantity of hydrogen recovered in the plant of Figure 4 is greater than that recovered in the plant of Figure 1.

TABLE 1

Streams	1	5	4	9	7	13	12	16	18
H_2	51415	50120	1295	49111	1009	49000	111	49	2464
CO	535	498	37	461	37	448	13	14	101
C_1	7820	6612	1208	4645	1967	4283	362	928	4460
C_2^-	49970	27432	22538	5305	22127	2241	3064	2003	130
C_2	40594	17707	22887	1955	15752	486	1469	450	19
C_3^-	1294	224	1070	4	220	0	4	0	0
C_3 and C_3^+	1008	50	958	0	50	0	0	0	0
TOTALS	152636	102643	49993	61481	41162	56458	5023	3444	7274

TABLE 2

Streams	1	4	3	8	7	12	11	15	17
H_2	51415	51415	0	50897	518	50758	139	87	744
CO	535	535	0	512	23	503	9	20	52
C_1	7820	7815	5	6663	1152	6111	552	1704	3405
C_2^-	49970	16080	33890	5972	10108	2454	3518	2211	94
C_2	40594	8897	31697	2302	6595	739	1563	702	7
C_3^-	1284	70	1224	1	69	0	1	0	0
C_3 and C_3^+	1008	23	985	0	23	0	0	0	0
TOTAL	152636	84835	67801	66347	18488	60565	5782	4724	4302

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Our copending Patent Application No. 2415/77 (Serial No. 1,539,292) describes and claims a process for separating by distillation ethylene from ethane, starting from a C_2 cut, which process comprises subjecting to the distillation first and second discrete fractions obtained from the C_2 cut by means of at least one fractionation of the C_2 cut, the first discrete fraction having a weight ratio of ethylene to ethane greater than that in the initial C_2 cut, and the second discrete fraction having weight ratio of ethane to ethylene greater than that in the initial C_2 cut.

WHAT WE CLAIM IS:—

1. A process for separating ethylene from ethane when ethylene and ethane are originally in admixture with methane, hydrogen, carbon monoxide, propane, propylene propyne, propadiene and small amounts of hydrocarbons containing four or more carbon atoms, the process comprising the folowing steps:—

(a) subjecting the original mixture to at least one of distillation, stripping and flashing, to produce a relatively light product which contains all or substantially all the hydrogen and carbon monoxide present in the original mixture, substantially all of the methane originally present, from 20% to 80% of each of the ethylene and ethane originally present and traces of hydrocarbons having 3 or more carbon atoms, and a relatively heavy product composed of the residual hydrocarbons;

(b) cooling in one or more stages the relatively light product to a temperature below minus 100°C, thereby separating a gaseous phase composed mainly of hydrogen with minor amounts of methane and carbon monoxide and with traces of ethylene and ethane, and one or more liquid phases composed substantially of methane, ethylene and ethane with minor amounts of hydrocarbons having three or more hydrocarbons;

(c) feeding the liquid phase(s) produced in step (b) to, respectively, one or more points of a distillation apparatus, with or without a head condenser, thereby obtaining at the head a fraction which contains methane, hydrogen, and small amounts of carbon monoxide and ethylene, and at the bottom a mixture of ethylene and ethane in which the ratio by weight of ethylene to ethane is greater than the corresponding ratio in the original mixture;

(d) feeding the relatively heavy product of step (a), composed of C_2, C_3, C_4 and heavier hydrocarbons, to a distillation column, thereby separating at the bottom the C_3, C_4 and heavier hydrocarbons and, at the head, a mixture of ethylene and ethane in which the ratio by weight of ethane to ethylene is greater than the coresponding ratio in the initial mixture;

(e) feeding the two mixtures of ethylene and ethane produced in steps (c) and (d) to a rectification column, the mixture in which the weight ratio of ethylene to ethane is greater than that in the original mixture being fed at a point above that at which the other mixture is fed, to produce a head product composed of ethylene and a bottom product composed of ethane.

2. A process according to claim 1, wherein the relatively light product is obtained by distillation or stripping in step (a) and is cooled in three stages, the first stage involving cooling to a temperature below −10°C to separate a liquid hydrocarbonaceous phase and a gaseous phase composed of hydrogen, carbon monoxide, methane, ethylene and ethane, the second stage involving cooling the gaseous phase from the first stage to a temperature below −80°C to separate a liquid phase essentially composed of methane, ethylene and ethane and a gaseous phase composed of hydrogen, carbon monoxide, methane, ethylene and ethane, with the combined ethylene and ethane contents being up to 5% by volume of the gaseous phase, the third stage involving cooling the gaseous stream from the second stage to a temperature below −100°C to separate a gas phase composed of hydrogen with carbon monoxide and methane in minor amounts, and a liquid phase composed of ethylene, ethane and methane, the three liquid phases from the three stages being fed, in step (c), to three points of the distillation apparatus.

3. A process according to claim 2, wherein the first and second stages involve cooling to approximately −80°C and approximately −100°C respectively.

4. A process according to claim 1, 2 or 3, wherein the pressure obtaining during the distillation or stripping of step (a) is in the range from 20 to 50 atmospheres and the head temperature is in the range from 0°C to −60°C.

5. A process according to claim 1, wherein the relatively light product is obtained by flashing in step (a) and is subjected to three consecutive cooling steps with separation of three liquid phases at approximately −100°C, approximately −120°C, and below −120°C, respectively.

6. A process according to claim 5, wherein the pressure obtaining during the flashing of step (a) is in the range from 7 to 15 atmospheres and the temperature is in the range from −40°C to −80°C.

7. A process according to any preceding claim, wherein the relatively light and relatively heavy products in step (a) and the mixture of ethylene and ethane produced in

Our copending Patent Application No. 2415/77 (Serial No. 1,539,292) describes and claims a process for separating by distillation ethylene from ethane, starting from a C_2 cut, which process comprises subjecting to the distillation first and second discrete fractions obtained from the C_2 cut by means of at least one fractionation of the C_2 cut, the first discrete fraction having a weight ratio of ethylene to ethane greater than that in the initial C_2 cut, and the second discrete fraction having weight ratio of ethane to ethylene greater than that in the initial C_2 cut.

WHAT WE CLAIM IS:—

1. A process for separating ethylene from ethane when ethylene and ethane are originally in admixture with methane, hydrogen, carbon monoxide, propane, propylene propyne, propadiene and small amounts of hydrocarbons containing four or more carbon atoms, the process comprising the folowing steps:—

(a) subjecting the original mixture to at least one of distillation, stripping and flashing, to produce a relatively light product which contains all or substantially all the hydrogen and carbon monoxide present in the original mixture, substantially all of the methane originally present, from 20% to 80% of each of the ethylene and ethane originally present and traces of hydrocarbons having 3 or more carbon atoms, and a relatively heavy product composed of the residual hydrocarbons;

(b) cooling in one or more stages the relatively light product to a temperature below minus 100°C, thereby separating a gaseous phase composed mainly of hydrogen with minor amounts of methane and carbon monoxide and with traces of ethylene and ethane, and one or more liquid phases composed substantially of methane, ethylene and ethane with minor amounts of hydrocarbons having three or more hydrocarbons;

(c) feeding the liquid phase(s) produced in step (b) to, respectively, one or more points of a distillation apparatus, with or without a head condenser, thereby obtaining at the head a fraction which contains methane, hydrogen, and small amounts of carbon monoxide and ethylene, and at the bottom a mixture of ethylene and ethane in which the ratio by weight of ethylene to ethane is greater than the corresponding ratio in the original mixture;

(d) feeding the relatively heavy product of step (a), composed of C_2, C_3, C_4 and heavier hydrocarbons, to a distillation column, thereby separating at the bottom the C_3, C_4 and heavier hydrocarbons and, at the head, a mixture of ethylene and ethane in which the ratio by weight of ethane to ethylene is greater than the coresponding ratio in the initial mixture;

(e) feeding the two mixtures of ethylene and ethane produced in steps (c) and (d) to a rectification column, the mixture in which the weight ratio of ethylene to ethane is greater than that in the original mixture being fed at a point above that at which the other mixture is fed, to produce a head product composed of ethylene and a bottom product composed of ethane.

2. A process according to claim 1, wherein the relatively light product is obtained by distillation or stripping in step (a) and is cooled in three stages, the first stage involving cooling to a temperature below −10°C to separate a liquid hydrocarbonaceous phase and a gaseous phase composed of hydrogen, carbon monoxide, methane, ethylene and ethane, the second stage involving cooling the gaseous phase from the first stage to a temperature below −80°C to separate a liquid phase essentially composed of methane, ethylene and ethane and a gaseous phase composed of hydrogen, carbon monoxide, methane, ethylene and ethane, with the combined ethylene and ethane contents being up to 5% by volume of the gaseous phase, the third stage involving cooling the gaseous stream from the second stage to a temperature below −100°C to separate a gas phase composed of hydrogen with carbon monoxide and methane in minor amounts, and a liquid phase composed of ethylene, ethane and methane, the three liquid phases from the three stages being fed, in step (c), to three points of the distillation apparatus.

3. A process according to claim 2, wherein the first and second stages involve cooling to approximately −80°C and approximately −100°C respectively.

4. A process according to claim 1, 2 or 3, wherein the pressure obtaining during the distillation or stripping of step (a) is in the range from 20 to 50 atmospheres and the head temperature is in the range from 0°C to −60°C.

5. A process according to claim 1, wherein the relatively light product is obtained by flashing in step (a) and is subjected to three consecutive cooling steps with separation of three liquid phases at approximately −100°C, approximately −120°C, and below −120°C, respectively.

6. A process according to claim 5, wherein the pressure obtaining during the flashing of step (a) is in the range from 7 to 15 atmospheres and the temperature is in the range from −40°C to −80°C.

7. A process according to any preceding claim, wherein the relatively light and relatively heavy products in step (a) and the mixture of ethylene and ethane produced in

step (d) are obtained in a single distillation column, the relatively light product being obtained as a head product, the relatively heavy product being obtained as a bottom product, and the ethylene and ethane mixture being obtained as a lateral cut, respectively.

8. A process substantially as hereinbefore described with reference to any one of Figures 3 to 6 of the accompanying drawings.

9. Ethane whenever separated by a process according to any preceding claim.

10. Ethylene whenever separated by a process according to any one of claims 1 to 8.

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