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PROCESS FOR THE SEPARATION OF HF VIA AZEOTROPIC DISTILLATION

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(56) Prior Art Documents
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(57) Claim

1. A process for the separation of a mixture comprising hydrogen fluoride and at least one of 2,2-dichloro-1,1,1-trifluoroethane and 2-chloro-1,1,1,2-tetrafluoroethane and minor amounts of halogenated materials using a combination of phase separation and distillation, said process comprises the steps of:

condensing the mixture to a temperature from -80 °C, to 40 °C, and a pressure from 0.10 MPa to 3.55 MPa, in a separation zone so that an organic phase comprising at least 85 mole percent of at least one of 2,2-dichloro-1,1,1,-trifluoroethane and 2-chloro-1,1,1,2-tetrafluoroethane and less than 15 mole percent of HF is formed as the bottom layer in the separation zone and an acid phase comprising at least 93 mole percent of HF and less than 7 mole percent of at least one of 2,2-dichloro-1,1,1-trifluoroethane, and

2-chloro-1,1,1,2-tetrafluoroethane is formed as the top layer in the separation zone; and

either (i) removing the acid phase from the top of the separation zone and distilling it in a distillation column to produce a substantially pure HF fraction, or (ii) removing the organic phase from the bottom of the separation zone and distilling it in a distillation column to produce a substantially pure organic fraction, or both (i) and (ii).

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Complete Specification for the invention entitled: PROCESS FOR THE SEPARATION OF HF VIA AZEOTROPIC DISTILLATION

The following statement is a full description of this invention, including the best method of performing it known to me:—*

***Note: The description is to be typed in double spacing, pica type face, in an area not exceeding 250 mm in depth and 160 mm in width, on tough white paper of good quality and it is to be inserted inside this form.**

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C. J. Thompson, Commonwealth Government Printer, Canberra

CR-8635

~~TITLE~~

PROCESS FOR THE SEPARATION OF HF VIA PHASE SEPARATION AND DISTILLATION

FIELD OF INVENTION

Process for the separation of mixtures comprising hydrogen fluoride (HF), 2,2-dichloro-1,1,1-trifluoroethane (FC-123), and/or 2-chloro-1,1,1,2-tetrafluoroethane (FC-124) by subjecting the mixture to phase separation.

BACKGROUND OF THE INVENTION

The efficient utilization of HF is important from both economic and process operability viewpoints. Techniques to effect the separation and recovery of HF from fluorocarbon process streams have been disclosed.

U.S. 2,478,362 discloses the use of a continuous separation zone to separate an organic phase from HF and then recycling the latter to the reactor feed system.

U.S. 3,406,099 discloses an azeotropic system useful for separation of CF_3COCF_3, HF or CCl_2FCClF_2 from mixtures containing one or more of these materials.

U.S. 3,873,629 discloses a continuous process for separating mixtures of HF and $ClCHF_2$ by countercurrent contact of a gaseous mixture of the two components with H_2SO_4.

U.S. 3,947,558 discloses a process for the separation of HF from the reaction products generated by fluorinating a 1-3 carbon chlorinated hydrocarbon by first separating HCl, followed by cooling to form an HF-rich layer and an HCl-free organic layer. This latter layer is mixed with a liquid 2 to 8 carbon glycol; after which an HF-enriched glycol layer is separated from the halocarbon layer. HF is recovered from the glycol by distillation.

U.S. 3,976,447 discloses the separation of HF from gaseous mixtures by treatment with dry particles of $CaCl_2$, $BaCl_2$, or $SrCl_2$, after which the HF is desorbed.

U.S. 4,209,470 discloses a process for the separation of HF from its liquid mixtures with 1-chloro-1,1-difluoroethane by adding an auxiliary solvent to enhance the HF composition of a liquid inorganic phase in a separation zone. The HF is then separated from the inorganic phase by distillation.

EP 98,341 discloses a process for the separation of HF and 1-chloro-1,1-difluoroethane which does not require an auxiliary solvent even though the feed stream to the separation zone contains pentafluorobutane which the disclosure states should contribute to the mutual solubility of HF and 1-chloro-1,1-difluoroethane; and therefore, should hinder a phase separation process. The separation is done without the use of auxiliary solvents by avoiding contamination and exercising good temperature control.

The need to produce alternate fluorocarbons useful as refrigerants and blowing agents or as intermediates in the production of other fluorocarbons useful as refrigerants and blowing agents has spurred an interest in processes for the preparation of FC-123 and FC-124. These are useful themselves as blowing agents, refrigerants and intermediates in the preparation of 1,1,1,2-tetrafluoroethane (FC-134a), a highly useful fluorocarbon refrigerant.

One process for the preparation of FC-123 and FC-124, described in U.S. 4,766,260 involves vapor phase hydrofluorination of halogenated alkenes with excess HF. This process produces a reaction mixture effluent consisting essentially of HF, FC-123, FC-124, tetrachloroethylene, HC1 and minor (less than 5 mole percent) amounts of other halogenated products, such as 1,2,2,-trichloro-1,1-difluoroethane (FC-122) and pentafluoroethane (FC-125). Removal of HC1 and FC-125 can be easily accomplished by simple distillation, leaving a mixture consisting of hydrogen fluoride (HF), FC-123, FC-124, tetrachloroethylene, and minor (e.g., less than 5 mol percent) amounts of other halogenated products. One method for separating the reaction mixture, described in U.S. 4,944,846 utilizes the formation of HF azeotropes with FC-123 and FC-124 under conditions of controlled HF/FC-123 ratios.

The instant invention provides an alternate route to separation of HF from FC-123 and FC-124 utilizing phase separation and distillation.

SUMMARY OF THE INVENTION

The present invention provides a process for the separation of a mixture comprising hydrogen fluoride (HF) and at least one of 2,2-dichloro-1,1,1-trifluoroethane (FC-123) and 2-chloro-1,1,1,2-tetrafluoroethane (FC-124) and minor amounts of halogenated materials using a combination of phase separation and distillation. The process comprises the steps of:

condensing the mixture to a temperature from -80°C, to 40°C, and a pressure from 0.10 MPa to 3.55 MPa, in a separation zone so that an organic phase comprising at least 85 mole percent of at least one of 2,2-dichloro-1,1,1,-trifluoroethane (FC-123) and 2-chloro-1,1,1,2-tetrafluoroethane (FC-124) and less than 15 mole percent of HF is formed as the bottom layer in the separation zone and an acid phase comprising at least 93 mole percent of HF and less than 7 mole percent of at least one of 2,2-dichloro-1,1,1-trifluoroethane (FC-123), and

2-chloro-1,1,1,2-tetrafluoroethane (FC-124) is formed as the top layer in the separation zone; and

either (i) removing the acid phase from the top of the separation zone and distilling it in a distillation column to produce a substantially pure HF fraction, or (ii) removing the organic phase from the bottom of the separation zone and distilling it in a distillation column to produce a substantially pure organic fraction, or both (i) and (ii).

The distillate comprising HF can be recovered from the top of the distillation column and recycled to the separation zone, if desired, for reasons set forth above. Other organic compounds which may be present include tetrachloroethylene and minor (e.g., less than 5 mole percent) amounts of other halogenated products.

The invention provides for the substantially complete separation of HF from the organic components utilizing phase separation and distillation techniques. It capitalizes on the discovery that HF, FC-123 and FC-124 form azeotropes that are more volatile than their individual components. Further, it depends on the formation of these azeotropes in the distillative stripping of HF substantially completely from the organic phase and in the distillative stripping of the organic components substantially completely from the acid phase, thereby enabling the separate recovery of substantially pure HF and organic fractions.

The invention also takes advantage of the further discovery that the proportions of HF in the azeotropic compositions (recovered in the distillative stripping of the acid and the organic phases) are greater than exist in the liquid organic phase of the two-phase system formed on condensing the original feed mixture. Likewise the proportions of organics in the azeotropic compositions are greater than exist in the liquid acid phase of the two-phase system. Thus recycling the azeotropic compositions to the feed mixture condensation step results in an overall enhanced degree of separation and eventually complete separation of HF and the organics, one from the other.

DESCRIPTION OF THE DRAWINGS

Figure 1 is a schematic flow diagram of one embodiment of the process of the invention.

Figure 2 is a schematic diagram of a second embodiment of the invention.

DETAILS OF THE INVENTION

While the feed mixture treated in accordance with the instant invention can be obtained from a variety of sources, an advantageous use of the instant invention resides in treating the effluent mixture from the preparation of FC-123 and FC-124 by reaction of tetrachloroethylene with HF, so that products from the reaction can be withdrawn or recycled as desired. The effluent mixture from this reaction generally consists essentially of HF, FC-123, FC-124, tetrachloroethylene, HCl, and minor amounts of halogenated products, such as 1,2,2-trichloro-1,1-difluoroethane (FC-122) and pentafluoroethane (FC-125). Low boiling components, such as HCl and FC-125, can be conventionally removed, leaving a mixture consisting essentially of hydrogen fluoride (HF), FC-123, FC-124, tetrachloroethylene, and minor (less than 5 mol percent) amounts of other halogenated products, which can advantageously be treated in accordance with the instant invention.

Applicants have found that two phases are formed at a variety of temperatures and pressures. At atmospheric pressure and -40°C, HF and FC-123 form two phases: an acid phase, consisting essentially of 98.3 mole percent (88.5 weight percent) HF and 1.7 mole percent (11.5 weight percent) FC-123, and an organic phase consisting essentially of 98.6 mole percent (99.8 weight percent) FC-123 and 1.4 mole percent (0.2 weight percent) HF. At the same temperature and pressure HF and FC-124 also form two phases: an acid phase, consisting essentially of 84.9 mole percent (45.2 weight percent) HF and 15.1 mole percent (54.8 weight percent) FC-124, and an organic phase consisting essentially of 97.6 mole percent (99.6 weight percent) FC-124 and 2.4 mole percent (0.4 weight percent) HF.

Utilizing this information applicants discovered that subjecting mixtures, comprising HF, FC-123, FC-124, and optionally, other organic compounds, to a temperature from about -80°C to 40°C, preferably from about -40°C to about 0°C, and a pressure from about 0.10 MPa to about 3.55 MPa, preferably from about 0.10 MPa to about 1.83 MPa in a separation zone results in the formation of an organic phase comprising less than 15 mole percent HF and an acid phase comprising at least 93 mole percent of HF.

The organic phase forms at the bottom of the separation zone and consists essentially of less than 15 mole percent HF and at least 85 mole percent of at least one of FC-123, FC-124 and other organic compounds which may be present. The acid phase forms at the top of the separation zone and consists essentially of at least 93 mole percent HF and less than 7 mole percent of at least one of FC-123, FC-124 and other organic compounds which may be present. The exact amounts of the various components of the organic and acid phases within the ranges specified are dependent on the amounts of these components in the original mixture and the mutual solubilities of the phases at operating temperature and pressure.

Preferred embodiments of the invention are illustrated by Figures 1 and 2. Referring to the Figures, a mixture 1 of Fig. 1 and Fig. 2 consisting essentially of HF, FC-123, FC-124 and minor amounts of other halogenated products is passed through a cooler 2 of Fig. 1 and Fig. 2 at a temperature from about -80°C to about 40°C and pressure from about 0.10 MPa to about 3.55 MPa, the cooled mixture 3 of Fig. 1 and Fig. 2 is sent to separation zone 4 of Fig. 1 and Fig. 2, maintained at a temperature from about -80°C to 40°C and pressure from about 0.10 MPa to about 3.55 MPa where an organic phase 5 of Fig. 1 and Fig. 2 containing less than 15 mol percent HF and an acid phase 6 of Fig. 1 and Fig. 2 containing at least 93 mol percent HF are formed. The acid phase 6 is removed from the top of the separation zone 4 and passed through heat exchanger 7 and pressure adjuster 8 of Fig. 1 and Fig. 2, and the resulting acid phase 9 of Fig. 1 and Fig. 2 is fed to a multiple-plate distillation column 10 of Fig. 1 and Fig. 2. The temperature at the top of column 10 is preferably from about -4°C to about 133°C, and more preferably from about -4°C to 97°C, and the pressure preferably from about 0.10 MPa to about 3.55 MPa, and more preferably from about 0.10 MPa to about 1.83 MPa, the exact temperature being dependent on the pressure. Essentially pure HF 11 of Fig. 1 and Fig. 2 can be removed from the bottom of column 10. Reboiler 12, of Figs. 1 and 2, provides heat input to the column by revaporizing a portion of mixture 11. A mixture of HF, FC-123 and FC-124, as well as minor amounts of other halogenated products can be removed from the top 13, of the column 10 of Fig. 1 and Fig. 2, passed through a condenser 14 of Fig. 1 and Fig. 2. The resulting mixture 15 of Fig. 1 and Fig. 2 can then be recycled through cooler 2 to separation zone 4.

In a further embodiment the organic phase 5 can be sent to a second multiple-plate distillation column 16 of Fig. 2 having a temperature at the top of from about -15°C to about 120°C, and a pressure from about 0.10 MPa to about 3.55 MPa, preferably about 0.69 MPa to about 1.83 MPa. A mixture of FC-123, FC-124 and other organic compounds which may be present, 17 of Fig. 2 can be removed from the bottom of column 16 or recycled through reboiler 18 of Fig. 2, to column 16. A mixture of FC-123 and FC-124 with HF, 19 of Fig. 2, can be removed from the top of column 16, cooled in condenser 20, and, passed directly, 21 of Fig. 2, to cooler 2 for recycle to the separation zone 4. Condenser 20 also provides reflux liquid for column 16.

EXAMPLES

In the following illustrative examples, all values for the compounds are in moles, temperatures are in Celsius. All the data were obtained by calculation using measured and calculated thermodynamic properties. The numbers at the top of the columns refer to Fig. 1 for Tables 1-8 and to Fig. 2 for Tables 9-14.

EXAMPLE 1

The purification system with a separation zone temperature of -40°C and one multiple plate distillation column is being operated as shown in Table 1. The mole percent of HF in the acid phase is 97.4 and in the organic phase 1.5.

TABLE 1

Compound	1 Feed Mix.	3 Decant Feed	6 Acid Phase	5 Organic Phase	11 Pure HF	15 Top Prod.	9 Col. Feed
HF	53.84	55.10	54.38	0.72	53.11	1.26	54.37
FC-123	38.30	39.30	1.00	38.30	0.00	1.00	1.00
FC-124	7.79	8.23	0.44	7.79	0.00	0.44	0.44
Temp °C	101	-40	-40	-40	20	-3	0
Press. MPa	1.83	1.83	1.83	1.83	0.10	0.10	0.10

EXAMPLE 2

The effect of varying the temperature of the separation zone is shown in Tables 2, 3, 4 and 5.

TABLE 2

Compound	3 Decant Feed	6 Acid Phase	5 Organic Phase
HF	53.65	46.49	7.16
FC-123	38.27	2.09	36.18
FC-124	8.00	0.76	7.24
Mole % HF		94.2	14.2
Temp °C	40	40	40
Press. MPa	1.83	1.83	1.83

TABLE 3

Compound	3 Decant Feed	6 Acid Phase	5 Organic Phase
HF	53.64	51.31	2.33
FC-123	38.27	1.47	36.80
FC-124	8.00	0.59	7.41
Mole % HF		96.1	5.0
Temp °C	0	0	0
Press. MPa	1.83	1.83	1.83

TABLE 4

Compound	3 Decant Feed	6 Acid Phase	5 Organic Phase
HF	53.64	52.94	0.70
FC-123	38.27	0.98	37.29
FC-124	8.00	0.42	7.58
Mole % HF		97.4	1.5
Temp °C	-40	-40	-40
Press. MPa	1.83	1.83	1.83

TABLE 5

Compound	3 Decant Feed	6 Acid Phase	5 Organic Phase
HF	53.65	53.46	0.19
FC-123	38.27	0.56	37.71
FC-124	8.00	0.27	7.73
Mole % HF		98.5	0.4
Temp °C	-80	-80	-80
Press. MPa	1.83	1.83	1.83

EXAMPLE 3

The effect of operating the distillation column at different pressures and temperatures is shown in Tables 6-8.

TABLE 6

Compound	11 Pure HF	15 Top Product	9 Column Feed
HF	93.51	3.89	97.40
FC-123		1.80	1.80
FC-124		0.78	0.78
Temp °C	20	-4	0
Press. MPa	0.10	0.10	0.10

TABLE 7

Compound	11 Pure HF	15 Top Product	9 Column Feed
HF	93.51	3.89	97.40
FC-123		1.80	1.80
FC-124		0.78	0.78
Temp °C	132	97	0
Press. MPa	1.83	1.83	1.83

TABLE 8

Compound	11 Pure HF	15 Top Product	9 Column Feed
HF	93.51	3.89	97.40
FC-123		1.80	1.80
FC-124		0.78	0.78
Temp °C	171	133	0
Press. MPa	3.55	3.55	3.55

EXAMPLE 4

If the purification system contains two multiple plate distillation columns (Fig. 2), then it is being operated under the conditions shown in Tables 9-14.

TABLE 9

Compound	1 Feed Mix.	3 Decant Feed	6 Acid Phase	5 Organic Phase	11 Pure HF	15 Top Prod.	21 Top Prod.
HF	53.87	64.68	56.46	8.22	53.87	2.59	8.22
FC-123	38.83	41.83	2.49	39.34	0.00	2.49	0.51
FC-124	7.80	11.48	1.19	10.29	0.00	1.19	2.49
Mole % HF			93.9	14.2			
Temp °C	101	40	40	40	85	56	43
Press. MPa	1.83	1.83	1.83	1.83	0.69	0.69	0.69

Compound	9 Column Feed	17 Organic Product
HF	56.46	0.00
FC-123	2.49	38.83
FC-124	1.19	7.80
Temp °C	70	83
Press. MPa	0.69	0.69

TABLE 10

Compound	1 Feed Mix.	3 Decant Feed	6 Acid Phase	5 Organic Phase	11 Pure HF	15 Top Prod.	21 Top Prod.
HF	53.87	59.14	56.46	2.68	53.87	2.59	2.68
FC-123	38.33	40.42	1.58	38.84	0.00	1.58	0.51
FC-124	7.80	11.12	0.83	10.29	0.00	0.83	2.50
Mole % HF			95.9	5.1			
Temp °C	101	0	0	0	85	56	43
Press. MPa	1.83	1.83	1.83	1.83	0.69	0.69	0.69

Compound	9 Column Feed	17 Organic Product
HF	56.46	0.00
FC-123	1.58	38.33
FC-124	0.83	7.78
Temp °C	70	83
Press. MPa	0.69	0.69

TABLE 11

Compound	1 Feed Mix.	3 Decant Feed	6 Acid Phase	5 Organic Phase	11 Pure HF	15 Top Prod.	21 Top Prod.
HF	53.87	57.29	56.47	0.82	53.88	2.59	0.82
FC-123	38.33	39.86	1.02	38.84	0.00	1.02	0.51
FC-124	7.80	10.90	0.57	10.32	0.00	0.57	2.51
Mole % HF			97.3	1.6			
Temp °C	101	-40	-40	-40	85	56	43
Press. MPa	1.83	1.83	1.83	1.83	0.69	0.69	0.69

Compound	9 Column Feed	17 Organic Product
HF	56.[illegible]8	0.00
FC-123	1.02	38.33
FC-124	0.57	7.81
Temp °C	70	83
Press. MPa	0.69	0.69

TABLE 12

Compound	1 Feed Mix.	3 Decant Feed	6 Acid Phase	5 Organic Phase	11 Pure HF	15 Top Prod.	21 Top Prod.
HF	53.87	56.69	56.47	0.22	53.88	2.59	0.22
FC-123	38.33	39.42	0.58	38.84	0.00	0.58	0.51
FC-124	7.80	10.67	0.35	10.32	0.00	0.35	2.51
Mole % HF			98.4	0.4			
Temp °C	101	-80	-80	-80	85	56	43
Press. MPa	1.83	1.83	1.83	1.83	0.69	0.69	0.69

Compound	9 Column Feed	17 Organic Product
HF	56.47	0.00
FC-123	0.58	38.33
FC-124	0.35	7.81
Temp °C	70	83
Press. MPa	0.69	0.69

TABLE 13

Compound	1 Feed Mix.	3 Decant Feed	6 Acid Phase	5 Organic Phase	11 Pure HF	15 Top Prod.	21 Top Prod.
HF	62.02	63.64	63.26	0.38	62.02	1.23	0.38
FC-122	0.34	0.34	t	0.34	0.00	t	t
FC-123	19.65	20.54	0.61	19.92	0.00	0.61	0.27
TCE*	14.80	14.82	0.02	14.80	0.00	0.02	t
FC-124	2.92	3.56	0.24	3.32	0.00	0.24	0.41
Mole % HF			98.6	1.0			
Temp °C	111	-20	-20	-20	85	57	48
Press. MPa	1.83	1.83	1.83	1.83	0.69	0.69	0.69

Compound	9 Column Feed	17 Organic Product
HF	63.26	0.00
FC-122	t	0.34
FC-123	0.61	19.65
TCE*	0.02	14.80
FC-124	0.24	2.92
Temp °C	70	104
Press. MPa	0.69	0.69

*Tetrachloroethylene
Trace

TABLE 14

Compound	1 Feed Mix.	3 Decant Feed	6 Acid Phase	5 Organic Phase	11 Pure HF	15 Top Prod.	21 Top Prod.
HF	128.96	152.09	141.16	10.94	128.96	12.20	10.94
FC-123	69.44	84.95	6.57	78.39	0.00	6.57	8.95
Mole % HF			95.6	12.2			
Temp °C	101.40	40.00	40.00	40.00	85.38	64.05	64.09
Press. MPa	1.83	1.83	1.83	1.83	0.69	0.69	0.69

Compound	9 Column Feed	17 Organic Product
HF	141.16	0.00
FC-123	6.57	69.44
Temp °C	70.00	91.88
Press. MPa	0.69	0.69

The claims defining the invention are as follows:-

1. A process for the separation of a mixture comprising hydrogen fluoride and at least one of 2,2-dichloro-1,1,1-trifluoroethane and 2-chloro-1,1,1,2-tetrafluoroethane and minor amounts of halogenated materials using a combination of phase separation and distillation, said process comprises the steps of:

condensing the mixture to a temperature from -80°C, to 40°C, and a pressure from 0.10 MPa to 3.55 MPa, in a separation zone so that an organic phase comprising at least 85 mole percent of at least one of 2,2-dichloro-1,1,1,-trifluoroethane and 2-chloro-1,1,1,2-tetrafluoroethane and less than 15 mole percent of HF is formed as the bottom layer in the separation zone and an acid phase comprising at least 93 mole percent of HF and less than 7 mole percent of at least one of 2,2-dichloro-1,1,1-trifluoroethane, and

2-chloro-1,1,1,2-tetrafluoroethane is formed as the top layer in the separation zone; and

either (i) removing the acid phase from the top of the separation zone and distilling it in a distillation column to produce a substantially pure HF fraction, or (ii) removing the organic phase from the bottom of the separation zone and distilling it in a distillation column to produce a substantially pure organic fraction, or both (i) and (ii).

2. The process of claim 1 wherein the temperature is from -40°C to 0°C and the pressure is from 0.10 MPa to 1.83 MPa.

3. The process of claim 1 or 2 comprising the additional steps of

(a) removing the acid phase from the top of the separation zone,

(b) feeding the acid phase from (a) to a distillation column at a temperature from -4°C to 133°C and a pressure from 0.10 MPa to 3.55 MPa to separate the HF component from the acid phase, and

(c) recovering the HF component of (b) from the bottom of the distillation column.

4. The process of claim 3 wherein an azeotrope containing HF is formed at the top of the distillation column.

5. The process of claim 4 wherein the azeotrope is recycled to the separation zone.

6. The process of any one of claims 3 to 5 comprising the additional steps of

(d) recovering the distillate comprising HF from the top of the distillation column, and

(e) recycling the distillate of (d) to the separation zone.

7. The process of any one of claims 1 to 6 comprising the additional steps of

(a) removing the organic phase from the bottom of the separation zone,

(b) feeding the organic phase from (a) to a distillation column at a temperature from -15°C to 120°C and a pressure from 0.10 MPa to 3.55 MPa to separate the HF component from the organic phase, and

(c) recovering the organic phase of (b) from the bottom of the distillation column.

8. The process of claim 7 wherein an azeotrope containing HF is formed at the top of the distillation column.

9. The process of claim 8 wherein the azeotrope is recycled to the separation zone.

10. The process of any one of claims 7 to 9 comprising the additional steps of

(d) recovering the distillate comprising HF from the top of the distillation column, and

(e) recycling the distillate of (d) to the separation zone.

11. A process for the separation of a mixture comprising hydrogen fluoride, 2,2-dichloro-1,1,1-trifluoroethane, and/or 2-chloro-1,1,1,2-tetrafluoroethane, substantially as hereinbefore described with reference to the Examples and drawings.

12. The product when produced by the process claimed in any one of the preceding claims.

DATED this 7 day of November 1991.

E.I. DU PONT DE NEMOURS AND COMPANY
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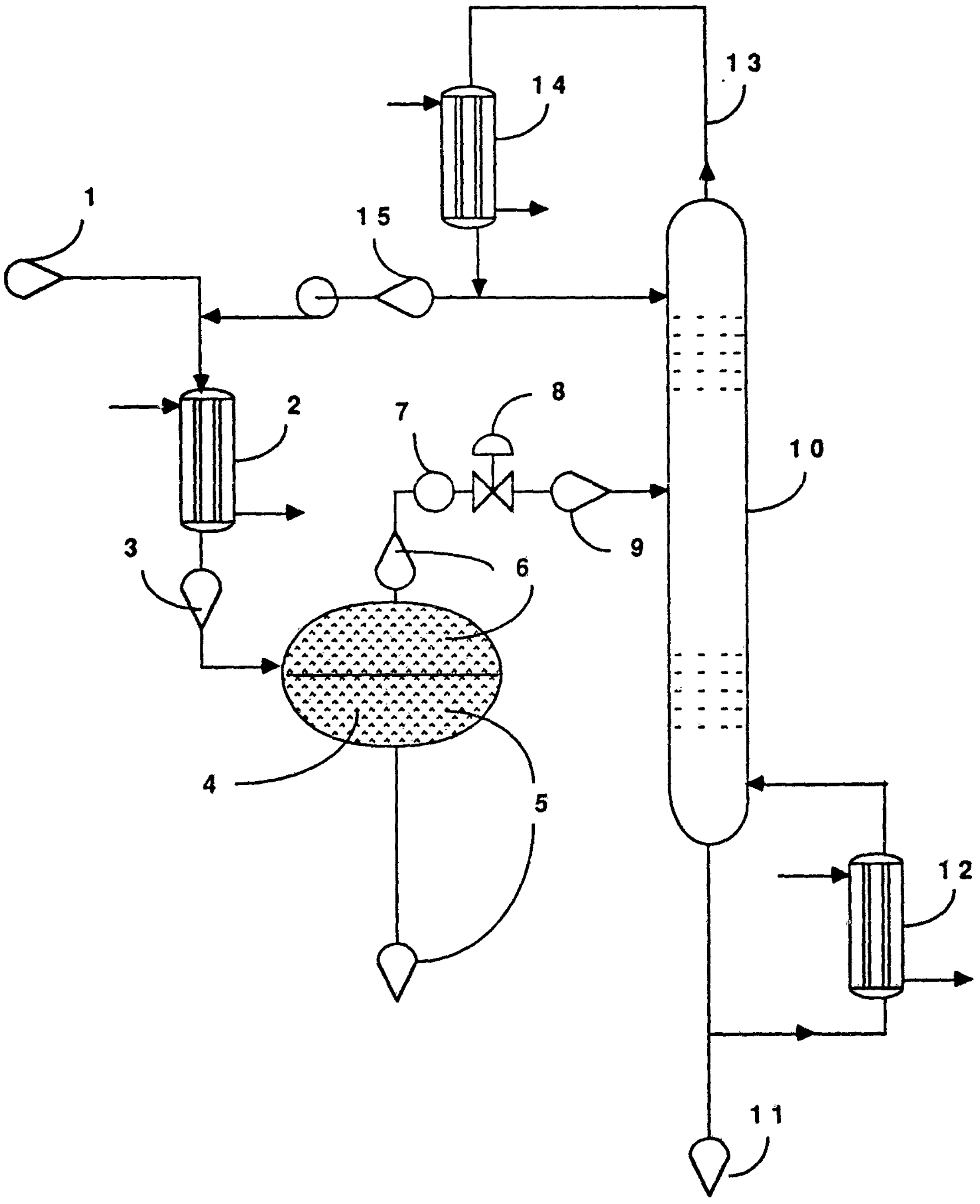
39113/89

F I G. 1



F I G. 2