United States Patent

Dunn

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[54] RECOVERY OF PRODUCTS FROM ELECTROCHEMICAL FLUORINATION

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[58] **Field of Search** 204/59

[56] **References Cited**

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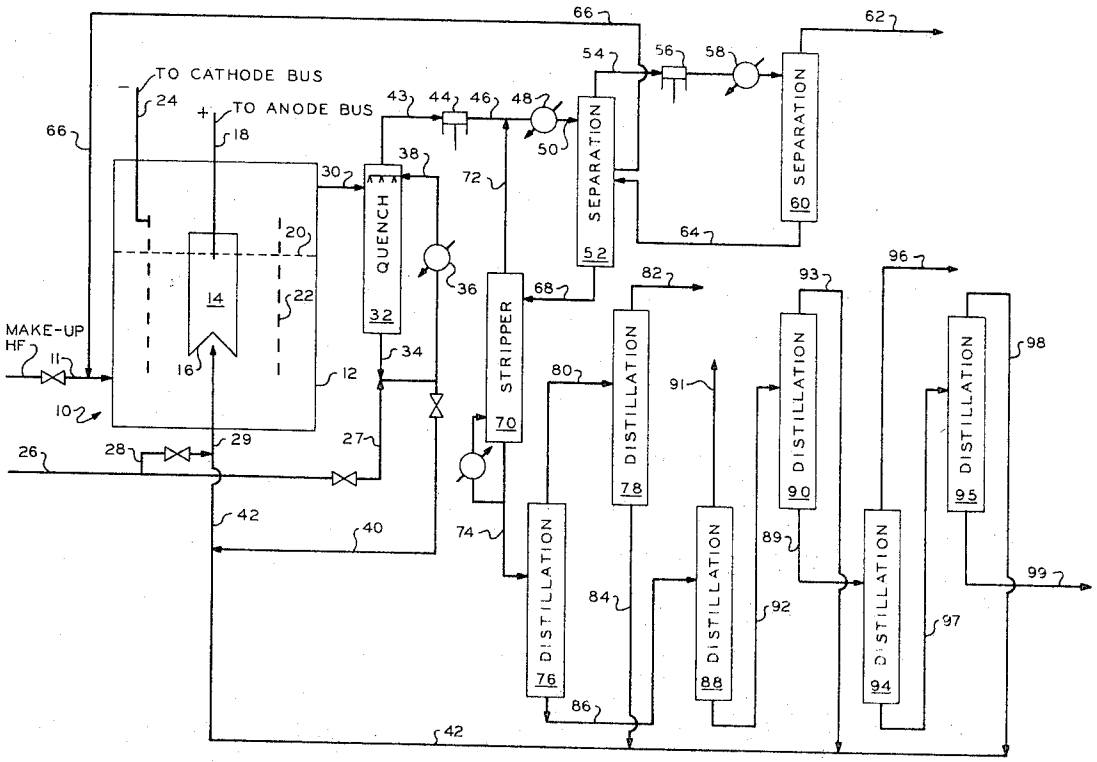
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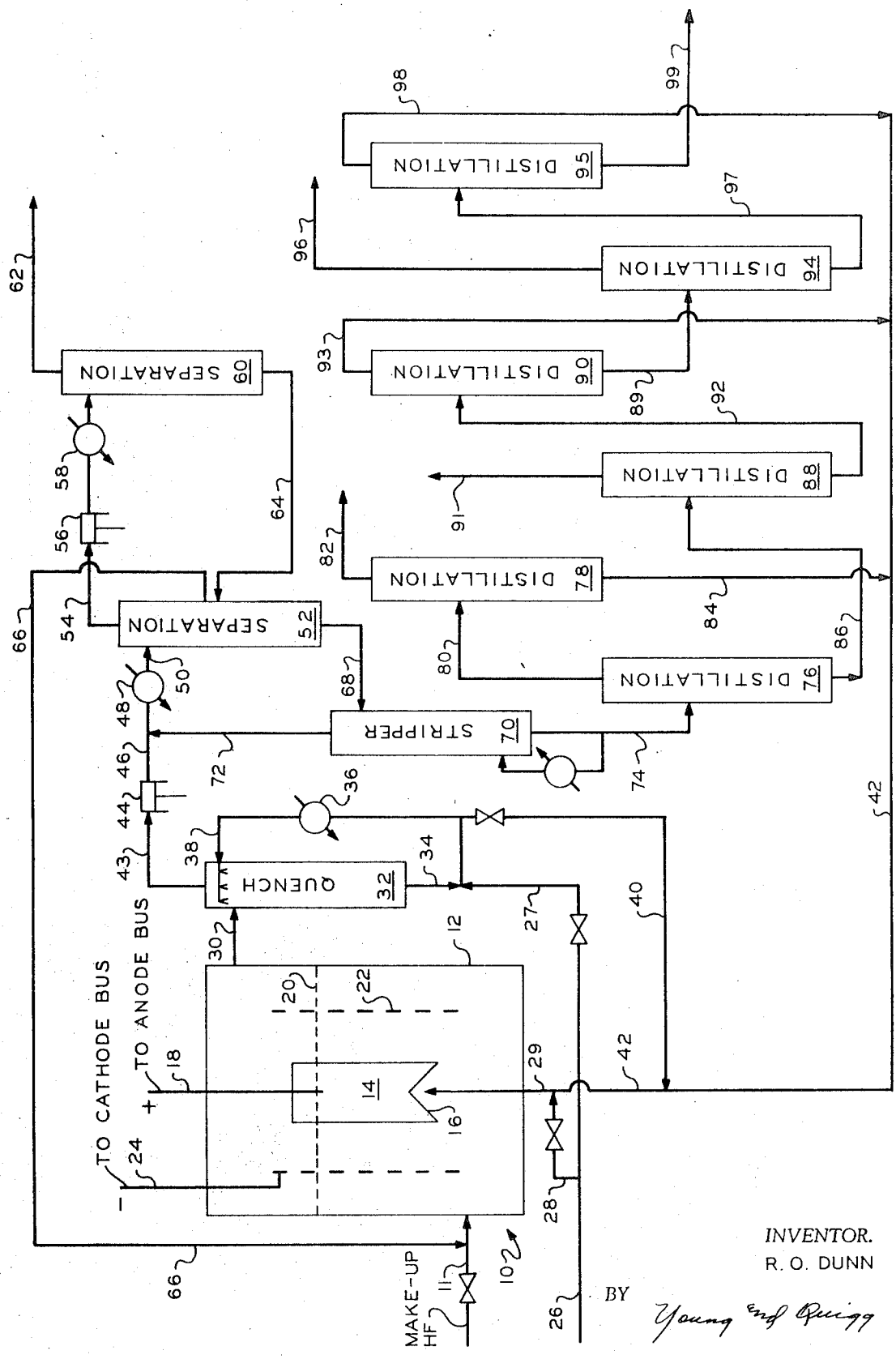
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[57] **ABSTRACT**

Fluorinated products are recovered from an effluent stream from an electrolytic cell in an electrochemical fluorination process by employing a combination of steps comprising cooling said effluent stream to a temperature near its dew point but insufficient to cause any significant condensation thereof, compressing said cooled effluent stream, chilling said compressed effluent stream to a temperature sufficient to condense at least the major portion of the components thereof other than hydrogen, and recovering fluorinated products from said chilled stream. In a preferred embodiment the initial cooling of said effluent stream is effected by directly contacting same with a liquid stream comprising the feedstock to the electrolytic cell.

14 Claims, 1 Drawing Figure





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RECOVERY OF PRODUCTS FROM ELECTROCHEMICAL FLUORINATION

This invention relates to the recovery of products from a cell effluent stream in an electrochemical fluorination process.

Electrochemical fluorination processes for preparing or converting a wide variety of feedstocks into desirable fluorinated products are well known in the art. Generally speaking, these processes usually involve immersing an anode element in an electrolyte and passing an electric current through said electrolyte between said anode and an oppositely charged element, e.g., either another electrode immersed in said electrolyte or the cell body which can serve as said other element or electrode. In one such process a material to be fluorinated is dissolved in the electrolyte and at least a portion of said material is converted into conversion products at the anode. In a variation of this process, the feedstock to be converted is bubbled into the electrolyte through a porous anode, such as a porous carbon anode, and said material is fluorinated in the electrolyte.

Recently it has been discovered that the reaction in an electrochemical fluorination process can be carried out within the confines, e.g., within the pores, of the porous anode itself. This type of operation is of particular utility with many feedstocks because it provides or makes possible a simple one-step route, at relatively high conversions, to produce partially fluorinated products which had previously been difficult to obtain. This process also allows operation at high conversions without substantial formation of cleavage products which are generally produced by the older methods when operating at high conversions. The feedstock to be fluorinated can be introduced into the pores of the porous anode at a point near its bottom and the fluorinated mixture removed from said pores at the top of the anode, generally above the electrolyte level. Passage of the feedstock into the bulk of the electrolyte is thus avoided.

One problem which is common to all of the above-described electrochemical fluorination processes is the recovery of the fluorinated products from the cell effluent stream. In many instances when an electrolyte comprising a current-conducting hydrogen fluoride is employed, said cell effluent will contain hydrogen which is produced as a cathode product, some hydrogen fluoride which vaporizes from the electrolyte, various fluorinated products produced from the feedstock, and unreacted feedstock. Generally speaking, in most instances said fluorinated products will include low boiling compounds comprising fluorinated products which are lower boiling than the feedstock, the desired fluorinated product or products, and a small amount of a fluorinated high boiling by-product. Efficient separation of the hydrogen and hydrogen fluoride from the cell effluent without loss of the low boiling fluorinated products is particularly troublesome.

The present invention provides a solution for the above-described problems. Broadly speaking, the present invention provides a combination of steps wherein a cell effluent stream is cooled to a temperature near its dew point but insufficient to cause any significant condensation of the components thereof. Said cooled effluent stream is then compressed, and then chilled by refrigeration to a temperature sufficient to condense at least the major portion of the components thereof other than hydrogen. Any hydrogen and hydrogen fluoride contained in the chilled stream are then separated therefrom. The fluorinated products are then recovered from the chilled stream.

An object of this invention is to provide an improved electrochemical fluorination process. Another object of this invention is to provide a method for recovering fluorinated products from a cell effluent stream in an electrochemical fluorination process with minimum loss of fluorinated product and high purity of the recovered products. Other aspects, objects, and advantages of the invention will be apparent to those skilled in the art in view of this disclosure.

Thus, according to the invention, there is provided in a process for the fluorination of a fluorinatable organic compound feedstock wherein, an electric current is passed through a current-conducting, essentially anhydrous, liquid hydrogen fluoride electrolyte contained in an electrolysis cell provided with a cathode and an anode, said feedstock is passed into said cell and into contact with said anode and at least partially fluorinated, and fluorinated product is recovered from an effluent stream withdrawn from said cell, the improvement comprising the steps of: (a) passing said effluent stream to a cooling zone and therein cooling said stream to a temperature near its dew point but insufficient to cause any significant condensation thereof; (b) compressing said cooled effluent stream; (c) chilling said compressed effluent stream to a temperature sufficient to condense at least the major portion of the components thereof other than hydrogen; and (d) recovering fluorinated products from said chilled stream.

A number of advantages are realized or obtained in the practice of the invention. The invention provides a method for maximum recovery of fluorinated products of high purity from a cell effluent stream. The invention also provides for maximum recovery of hydrogen fluoride electrolyte in those processes wherein the cell effluent stream contains hydrogen fluoride electrolyte. The cooling effected in the above-described step (a) can be carried out by indirect heat exchange or by direct heat exchange. In a preferred embodiment of the invention, said cooling is effected by directly contacting the cell effluent stream with a quenching liquid which comprises the feedstock to the electrochemical fluorination cell. The quench liquid is thus completely compatible with the cell effluent stream and can be returned to the cell as a portion of the feedstock thereto. Another major advantage of using direct heat exchange in said step (a) cooling is that the use of an expensive large surface area heat exchanger fabricated from expensive stainless steel is avoided. Still another advantage is that any liquid, e.g., such as liquid HF, which may have been entrained in the cell effluent stream will be washed out and removed by the quench liquid prior to compression step (b).

Very few organic compounds are resistant to fluorination. Consequently, a wide variety of feed materials, both normally liquid and normally gaseous compounds, can be used as feedstocks in said process. Organic compounds which are normally gaseous or which can be introduced in gaseous state into the pores of a porous anode under the conditions employed in the electrolysis cell, and which are capable of reacting to form fluorinated products, are presently preferred as starting materials. Generally speaking, desirable organic starting materials which can be used are those containing from two to eight, preferably two to six, carbon atoms per molecule. However, reactants which contain less than two or more than six or eight carbon atoms can also be used. Some general types of organic starting materials which can be used include, among others, the following: alkanes, alkenes, alkynes, amines, ethers, esters, mercaptans, nitriles, alcohols, aromatic compounds, and partially halogenated compounds of both the aliphatic and aromatic series. It will be understood that the above-named types of compounds can be either straight chain, branched chain, or cyclic compounds.

One group of presently preferred starting materials comprises the normally gaseous organic compounds, and particularly the saturated and unsaturated hydrocarbons, containing from two to four carbon atoms per molecule. Normally liquid feedstocks which can be vaporized under cell operating conditions are also preferred starting materials. Some examples of these are methane, ethane, propane, butane, isobutane, ethylene, propylene, butene-2, acetylene, propyne, butyne-1, butadiene, and the like, and mixtures thereof.

One presently more preferred class of starting materials for use in the practice of the invention includes the fluorinatable, partially halogenated compounds having a boiling point higher than at least the major portion of the fluorinated products obtained therefrom. In said halogen-containing feedstocks, the halogen can be any of the halogens, chlorine, bromine, iodine,

or fluorine. Preferably, the halogen is one other than fluorine. Partially chlorinated hydrocarbons have been found particularly useful. Examples of said compounds include, among others, the following: mono-, di-, tri-, and tetrachloroethanes; monofluoro-, mono-, di-, tri-, and tetrachloroethanes; difluoro-, mono-, di-, and trichloroethanes; trifluoro-, mono-, and dichloroethanes; mono-, di-, tri-, and tetrabromoethanes; mono-, di-, tri-, and tetraiodoethanes; etc. Thus, applicable compounds include: methyl chloride; methyl fluoride; chloroform; methylene diiodide; bromoform; chlorofluoromethane; bromochloromethane; 1,2-dichloroethane; 1,1-diiodoethane; 1-bromo-2-fluoroethane; 1,1,2-trichloroethane; 1,1-dichloro-2,2-difluoroethane; 1,2-dichloropropane; 1-bromo-3-iodopropane; 1-chloro-3-fluoropropene; 1,1-dichloro-2,3-difluoropropane; 1,1,1,2-tetrafluoropropane; 1,1-dichlorobutane; 2,3-dibromobutane; 1,1,1-trichloro-3-iodobutane; 1,4-difluorobutene-2; 1,2,3-trichlorobutane; and the like, and mixtures thereof.

The invention is applicable to any electrochemical fluorination process employing an electrolyte comprising essentially anhydrous hydrogen fluoride. The invention is particularly applicable to electrochemical fluorination processes in which porous anodes are employed. In one presently preferred process, a current-conducting, essentially anhydrous, liquid hydrogen fluoride is electrolyzed in an electrolysis cell provided with a cathode and a porous anode (preferably porous carbon), a fluorinatable feedstock is introduced into the pores of said anode and at least a portion of said feedstock is at least partially fluorinated within the pores of said anode, and fluorinated products are recovered from a cell effluent stream.

Briefly, said preferred electrochemical fluorination process comprises passing the feedstock to be fluorinated into the pores of a nonwetting porous anode, e.g., porous carbon, disposed in a current-conducting, essentially anhydrous, hydrogen fluoride electrolyte such as KF·2HF. Said feedstock contacts the fluorinating species within the pores of the anode and is therein at least partially fluorinated. Generally speaking, said fluorination can be carried out at temperatures within the range of from −80° to 500° C. (−112° to 932° F.) at which the vapor pressure of the electrolyte is not excessive. A preferred temperature range is from about 60° C. to about 120° C. (140° to 248° F.). Pressures substantially above or below atmospheric can be employed if desired. Generally speaking, the process is conveniently carried out at substantially atmospheric pressures. The feedstock to be fluorinated is preferably introduced into the pores of the anode at a rate such that there is established a pressure balance within the pores of the anode between the feedstock entering the pores and electrolyte attempting to enter said pores from another and opposing direction. Said feedstock flow rate can be within the range of from 3 to 600 milliliters per minute per square centimeter of anode cross-sectional area, taken perpendicular to the direction of flow and expressed in terms of gaseous volume calculated at standard conditions. Current densities employed can be within the range of 30 to 1,000, preferably 50 to 500, milliamps per square centimeter of anode geometric surface area. Typical cell voltages employed can range from 4 to 12 volts. Converted and unconverted products are withdrawn from the pores of the anode and the products recovered from a cell effluent stream.

Further details of said preferred electrochemical fluorination process can be found in U. S. Pat. No. 3,511,760, issued to H. M. Fox and F. N. Ruehlen.

The drawing is a diagrammatic flow sheet illustrating one presently preferred embodiment of the invention wherein a feedstock, e.g., 1,2-dichloroethane, is fluorinated and the products obtained are separated in accordance with the invention.

Referring now to the drawing, the invention will be more fully explained. By way of example, and not by way of limitation, the invention will be described with particular reference to using 1,2-dichloroethane as the fresh feedstock to the electrolytic cell. Thus, the typical operating conditions given herein in connection with using said feedstock and separating the products therefrom are not to be construed as limiting on the invention. In said drawing there is illustrated an electrolytic cell, denoted generally by the reference numeral **10**, comprising a cell body **12** having an anode **14** disposed therein. As here illustrated diagrammatically, said anode in its simplest form comprises a cylinder of porous carbon having a cavity **16** formed in the bottom thereof. Any suitable anode can be employed in said cell. Examples of other suitable anodes can be found in U. S. Pat. No. 3,511,762, issued to W. V. Childs. A current collector **18**, usually comprising a rod or hollow conduit of a metal such as copper, is provided in intimate contact with the upper portion of said anode **14** and is connected to the anode bus of the current supply. Preferably, the upper end of anode **14** extends above the electrolyte level **20**. However, it is within the scope of the invention for the top of said anode to be below said electrolyte level. A circular cathode **22**, which can be a screen formed of a suitable metal, such as carbon steel or stainless steel, surrounds said anode **14** and is connected to the cathode bus of the current supply by a suitable lead wire **24**. Any suitable source of current and connections thereto can be employed.

In the operation of the system illustrated, a feedstock such as 1,2-dichloroethane is introduced into the cavity portion **16** of said anode via conduits **26**, **28**, and **29**, travels upward through the pores of said anode, and exits from the upper end of the anode above electrolyte level **20**. During passage through said anode, at least a portion of the feedstock is electrochemically fluorinated. Fluorinated products together with remaining unconverted feedstock, hydrogen, and possibly some electrolyte vapors, are withdrawn from the space above the electrolyte within cell **12** via conduit **30**. During the introduction of said feedstock an electric current in an amount sufficient to supply the desired operating current density at the anode is passed between the anode and the cathode.

Preferably, the cell effluent stream in conduit **30** is cooled in quench zone **32** with a stream of liquid comprising the cell feedstock from conduit **26**. Said quench zone **32** can comprise any suitable means for obtaining efficient contact between a liquid stream and a vapor stream. For example, said quench zone **32** can comprise a tower filled with Raschig rings or other suitable contacting material, a bubble cap tower, etc. Initially, at least, quenching liquid is introduced from conduit **26** into conduit **34**, passed through the cooler **36**, and then introduced into quench zone **32** by means of conduit **38**. In quench zone **32**, said cell effluent stream is preferably cooled to a temperature which is near its dew point but which is insufficient to cause any significant condensation of the components thereof. As will be understood by those skilled in the art, said dew point will vary with the composition of said effluent stream and the pressure in said quench zone. Thus, the temperature to which said effluent stream is cooled can vary widely. When 1,2-dichloroethane is the charge stock to the electrochemical fluorination cell, said dew point will usually be in the order of about 115° F. to about 125° F. at essentially atmospheric pressure. Generally speaking, it is desirable to cool said effluent stream as nearly as practical to its dew point without causing said condensation of the components thereof. In most instances, said effluent stream will be cooled to a temperature which is within 3° to 10° F. above said dew point. Condensation is preferably avoided so as to avoid passing liquid into the compressor in the subsequent compression step. It is, however, an advantage of my invention that any material which may be condensed within quench zone **32** will be absorbed by the quench liquid and thus only vapor will pass to compressor **44**. Said cell effluent stream will preferably be cooled to a temperature which is not greater than about 165° F. At temperatures above about 165° F. gaseous streams containing vaporous HF become excessively corrosive to carbon steel equipment. Said cooling or quenching can be carried out by continuously circulating the quenching liquid through quenching zone **32** via conduits **34**, cooler **36**, and conduit **38**, and supplying only sufficient make-up quench liquid from

conduit 26 as is necessary. Any buildup of quenching liquid can be withdrawn from the circulating stream via conduit 40 and returned to the cell as a portion of the feedstock thereto via conduit 42. It is generally desirable to "bleed" a small portion of the quench liquid through conduit 40 and replace same with fresh feedstock via conduit 27, thus preventing buildup of any components which may be absorbed in the circulating quench liquid. However, it is within the scope of the invention to continuously circulate all or a portion of the fresh feed to the cell through said quench zone 32 by means of the manifolding arrangement shown in the drawing.

Quench zone 32 can be conveniently operated at a temperature of about 120° F. and a pressure of about 15 psia. A vaporous stream is withdrawn from said quench zone via conduit 43, compressed in compressor 44 to a pressure of about 30 psia at a temperature of about 210° F. Said compressed effluent stream is passed via conduit 46 into chiller 48 wherein its temperature is decreased to about −20° F. at a pressure of about 27 psia, and is then passed via conduit 50 into first phase separation zone 52 wherein it separates into a gaseous phase and two liquid phases. A gaseous stream comprising hydrogen is withdrawn from first separation zone 52 via conduit 54, compressed in compressor 56 to a pressure of about 85 psia at a temperature of about 155° F., then introduced into chiller 58 where it is chilled by means of refrigeration to a temperature of about −100° F. at a pressure of about 80 psia, and then introduced into second phase separation zone 60. A stream comprising hydrogen is vented from the system via conduit 62. A stream comprising liquid HF is withdrawn from second separation zone 60 via conduit 64 and passed to said first separation zone 52. Liquid HF is withdrawn from first separation zone 52 via conduit 66 and recycled to the electrochemical fluorination cell. Make-up HF can be introduced to the system via conduit 11.

A second liquid stream comprising fluorinated products is withdrawn from first phase separation zone 52 via conduit 68 and introduced into stripping zone 70 wherein any remaining HF contained in the liquid stream can be stripped therefrom and returned via conduit 72 to conduit 46. Vapors for stripping said second liquid stream can be generated by means of the reboiler arrangement shown on the lower portion of stripper tower 70. Said stripping tower 70 can conveniently be operated at a pressure of about 45 psia, a top tower temperature of about 100° F., and a bottom tower temperature of about 166° F.

Said now stripped second liquid stream comprising fluorinated products and unreacted feedstock is withdrawn from stripper zone 70 via conduit 74 and passed to a first distillation zone comprising a first fractionation column 76 and a second fractionation column 78. Said first fractionation column can conveniently be operated at a pressure of about 130 psia, a top tower temperature of about 145° F., and a bottom tower temperature of about 256° F. An overhead stream comprising chloropentafluoroethane is withdrawn from first fractionation column 76 via conduit 80 and introduced into said second fractionation column 78. Said second fractionation column 78 can conveniently be operated at a pressure of about 235 psia, a top tower temperature of about 123° F., and a bottom tower temperature of about 199° F. An overhead stream comprising said chloropentafluoroethane is removed overhead from column 78 via conduit 82 as one of the products of the process. A bottoms stream comprising 1-chloro-1,1,2,2-tetrafluoroethane is removed from said second fractionation column 78 via conduit 84 and passed into conduit 42 for recycle to cell 12 as a portion of the feedstock thereto.

A bottoms stream comprising other fluorinated materials and unreacted 1,2-dichloroethane feedstock is withdrawn from first fractionation column 76 via conduit 86 and passed to a second distillation zone comprising a third fractionation column 88 and a fourth fractionation column 90. Said third fractionation column 88 can conveniently be operated at a pressure of about 70 psia, a top tower temperature of about 126° F., and a bottom tower temperature of about 235° F. An overhead stream comprising 1,2-dichlorotetrafluoroethane is recovered from said third fractionation column 88 via conduit 91 as another product of the process. A bottoms stream comprising said other fluorinated materials and said unreacted 1,2-dichloroethane feedstock is passed from third fractionation column 88 via conduit 92 into said fourth fractionation column 90. Said fourth fractionation column can conveniently be operated at a pressure of about 30 psia, a top tower temperature of about 136° F., and a bottom tower temperature of about 205° F. An overhead stream comprising 1,2-dichloro-1,2,2-trifluoroethane is recovered from said fourth fractionation column 90 via conduit 93 and introduced into said conduit 42 for recycle to said cell 12 as a portion of the feedstock thereto.

A bottoms stream comprising said other fluorinated materials and said unreacted 1,2-dichloroethane feedstock is passed from said fourth fractionation column 90 to a third distillation zone comprising a fifth fractionation column 94 and a sixth fractionation column 95. Said fifth fractionation column can conveniently be operated at a pressure of about 60 psia, a top tower temperature of about 134° F., and a bottom tower temperature of about 180° F. An overhead stream comprising 1,1,2-trichloro-1,2,2-trifluoroethane is recovered from said fifth fractionation column via conduit 96 as another product of the process. A bottoms stream comprising said other fluorinated materials and said unreacted 1,2-dichloroethane is passed from said fifth fractionation column 94 via conduit 97 into said sixth fractionation column 95. Said sixth fractionation column can conveniently be operated at a pressure of about 20 psia, a top tower temperature of about 192° F., and a bottom tower temperature of about 260° F. An overhead stream comprising 1,2-dichloro-1-monofluoroethane and said unreacted 1,2-dichloroethane feedstock is recovered from said sixth fractionation column via conduit 98 and introduced into said conduit 42 for recycle to said cell 12 as a portion of the feedstock thereto. A bottoms stream comprising a small amount of higher boiling heavy materials is withdrawn from sixth fractionation column 95 via conduit 99 and removed from the system.

The following calculated example will serve to further illustrate the invention. The conditions set forth for the operation of the electrochemical fluorination cell are based on numerous laboratory and pilot plant runs carried out for the electrochemical fluorination of 1,2-dichloroethane, and on laboratory distillations of the cell effluent.

EXAMPLE

In this illustrative embodiment a run is carried out for the electrochemical fluorination of 1,2-dichloroethane in a system embodying the essential features of the system illustrated in the drawing and using an electrolyte in cell 10 which has an approximate composition of KF·2HF. Porous carbon cylinders embodying the essential features of anode 14 illustrated diagrammatically in the drawing are employed as anodes. Fresh 1,2-dichloroethane feedstock is introduced via conduits 26 and 28 into the pores of anode 14. Recycle feedstock is supplied by conduit 42. The conversion in electrolytic cell 10 is carried out at an electrolyte temperature of about 210° F., employing a current density of about 250 amperes per square foot of anode geometric surface area, and a voltage of about 9.5 volts, D.C. The pressure in cell 10, conduit 30, and quench zone 32 is substantially atmospheric. A cell effluent stream is withdrawn via conduit 30 and processed essentially as described above in connection with the drawing for the recovery of products therefrom and the return of the recycle stream to the cell via conduit 42. Table I below sets forth the principal components in said cell effluent stream and said recycle streams, and a calculated material balance for the system.

TABLE I.—STREAM NUMBER, MOLS PER HOUR

Component	28	42	29	30	54	62	66	68	74	72	80	82	84
$CClF_2$—$CClF_2$		1.63	1.63	16.43	1.44	0.09		17.32	16.34	0.98	0.83		0.8
$CHClF$—$CClF_2$		14.65	14.65	14.71	0.46	0.01		15.08	14.7	0.38			
$CClF_2$—CH_2Cl		4.9	4.9	5.4	0.08			5.48	5.4	0.08			
$CHClF$—$CHClF$		9.8	9.8	9.8	0.08			9.89	9.8	0.09			
$CHClF$—CH_2Cl		14.7	14.7	14.7	0.06			14.78	14.7	0.08			
CH_2Cl—CH_2Cl*	18.0	24.3	42.3	24.3	0.05			24.39	24.3	0.09			
$CClF_2$—CF_3		0.07	0.07	0.72	0.32	0.15		0.73	0.57	0.16	0.57	0.50	0.07
CHF_2—$CClF_2$		0.60	0.60	0.68	0.11	0.02		0.72	0.66	0.06	0.61	0.01	0.60
$CHClF$—CHF_2		0.35	0.35	0.66	0.03			0.68	0.66	0.02			
CHF_2—CH_2Cl		0.06	0.06	0.66	0.02			0.68	0.66	0.02			
CCl_2F—$CClF_2$		9.00	9.00	10.00	0.11			10.14	10.00	0.14			
$CHCl_2$—$CHClF$		1.96	1.96	1.96				1.96	1.96				
$CHCl_2$—CH_2Cl		0.98	0.98	0.98				0.98	0.98				
HF				15.8	5.6	0.13	15.67	3.56		3.56			
H_2				66.63	66.64	66.63		0.25		0.25			
Total	18.0	83.00	101.00	183.43	75.00	67.03	15.67	106.64	100.73	5.91	2.01	0.51	1.50

Component	86	91	92	93	89	96	97	99	98	11	38	27	40
$CClF_2$—$CClF_2$	15.51	14.71	0.80	0.80									
$CHClF$—$CClF_2$	14.7	0.05	14.65	14.65									
$CClF_2$—CH_2Cl	5.4		5.4	2.7	2.7	0.5	2.2		2.2				
$CHClF$—$CHClF$	9.8		9.8	0.02	9.78		9.78		9.78				
$CHClF$—CH_2Cl	14.7		14.7		14.7		14.7		14.7				
CH_2Cl—CH_2Cl*	24.3		24.3		24.3		24.3		24.3		1,070.0	1.0	1.0
$CClF_2$—CF_3													
CHF_2—$CClF_2$	0.05	0.05											
$CHClF$—CHF_2	0.66	0.30	0.36	0.36									
CHF_2—CH_2Cl	0.66	0.60	0.06	0.06									
CCl_2F—$CClF_2$	10.00		10.00	5.00	5.00	1.00	4.00		4.00				
$CHCl_2$—$CHClF$	1.96		1.96		1.96		1.96		1.96				
$CHCl_2$—CH_2Cl	0.98		0.98		0.98		0.98	Tr	0.98				
HF										66.76			
H_2													
Total	98.72	15.71	83.01	23.59	59.42	1.50	57.92	Tr	57.92	66.76	1,070.0	1.0	1.0

*Fresh feedstock.

From the data set forth above, it will be seen that the invention provides an efficient method for the separation and recovery of products from a cell effluent stream in the electrochemical fluorination of organic compounds.

While certain embodiments of the invention have been described for illustrative purposes, the invention is not limited thereto. Various other modifications or embodiments of the invention will be apparent to those skilled in the art in view of this disclosure. Such modifications or embodiments are within the spirit and scope of the disclosure.

I claim:

1. In a process for the fluorination of a fluorinatable organic compound feedstock wherein, an electric current is passed through a current-conducting, essentially anhydrous, liquid hydrogen fluoride electrolyte contained in an electrolysis cell provided with a cathode and an anode, said feedstock is passed into said cell and into contact with said anode and at least partially fluorinated, and fluorinated product is recovered from an effluent stream withdrawn from said cell, the improvement comprising the steps of:

a. passing said effluent stream to a cooling zone and therein cooling said stream to a temperature near its dew point but insufficient to cause any significant condensation thereof;

b. compressing said cooled effluent stream;

c. chilling said compressed effluent stream to a temperature sufficient to condense at least the major portion of the components thereof other than hydrogen; and

d. recovering fluorinated products from said chilled stream.

2. A process according to claim 1 wherein said effluent stream is cooled in step (a) by directly contacting same with a liquid stream comprising said feedstock.

3. A process according to claim 2 wherein said effluent stream is cooled to a temperature not greater than about 165° F.

4. A process according to claim 2 wherein said compressed stream is chilled in step (c) to a temperature sufficient to condense hydrogen fluoride and higher boiling materials contained therein.

5. A process according to claim 4 wherein:

e. said chilled effluent stream from step (c) is passed to a first phase separation zone;

f. a liquid stream comprising hydrogen fluoride is withdrawn from said first phase separation zone for recycle to said cell;

g. a gaseous stream comprising hydrogen is vented from said first phase separation zone; and

h. said fluorinated products recovered in step (d) are recovered from a second liquid stream comprising same which is withdrawn from said first phase separation zone.

6. A process according to claim 5 wherein:

i. said stream comprising hydrogen in step (g) is compressed, then chilled, and then passed to a second phase separation zone;

j. a stream comprising hydrogen is vented from said second phase separation zone; and

k. a stream comprising liquid hydrogen fluoride is returned from said second phase separation zone to said first phase separation zone.

7. A process according to claim 5 wherein: said second liquid stream of step (h) is passed to a stripper zone; hydrogen fluoride vapors are stripped from said second liquid stream; and said fluorinated products are recovered from said stripped second liquid stream.

8. A process according to claim 6 wherein:

l. said second liquid stream from step (h) is passed to a stripper zone;

m. hydrogen fluoride vapors are stripped from said second liquid stream and returned to said compressed cooled effluent stream of step (b); and

n. said fluorinated products are recovered from said stripped second liquid stream.

9. A process according to claim 2 wherein:

said feedstock to said cell comprises 1,2-dichloroethane; and

said fluorinated products recovered in step (d) include at least one of 1,1,2-trichloro-1,2,2-trifluoroethane; 1,2-dichlorotetrafluoroethane; and monochloropentafluoroethane.

10. A process according to claim 7 wherein:

said feedstock to said cell comprises 1,2-dichloroethane; and

said fluorinated products recovered from said stripped second liquid include at least one of 1,1,2-trichloro-1,2,2-trifluoroethane; 1,2-dichlorotetrafluoroethane; and monochloropentafluoroethane.

11. A process according to claim 8 wherein:
said feedstock to said cell comprises 1,2-dichloroethane; and said fluorinated products recovered in step (n) include at least one of 1,1,2-trichloro-1,2,2-trifluoroethane; 1,2-dichlorotetrafluoroethane; and monochloropentafluoroethane.

12. A process according to claim 7 wherein:
said feedstock to said cell comprises 1,2-dichloroethane;
said stripped second liquid stream is passed to a first distillation zone comprising a first fractionation column and a second fractionation column;
an overhead stream comprising monochloropentafluoroethane is passed from said first fractionation column to said second fractionation column;
an overhead stream comprising said monochloropentafluoroethane is removed overhead from said second fractionation column as a product of the process; and
a bottoms stream comprising 1-chloro-1,1,2,2-tetrafluoroethane is recovered from said second fractionation column and recycled to said cell as a portion of said feedstock.

13. A process according to claim 12 wherein:
a bottoms stream comprising other fluorinated materials and unreacted 1,2-dichloroethane is passed from said first fractionation column to a second distillation zone comprising a third fractionation column and a fourth fractionation column;
an overhead stream comprising 1,2-dichlorotetrafluoroethane is recovered from said third fractionation column as another product of the process;
a bottoms stream comprising said other fluorinated materials and said unreacted 1,2-dichloroethane is passed from said third fractionation column to said fourth fractionation column; and
an overhead stream comprising 1,2-dichloro-1,2,2-trifluoroethane is recovered from said fourth fractionation column and recycled to said cell as a portion of said feedstock.

14. A process according to claim 13 wherein:
a bottoms stream comprising said other fluorinated materials and said unreacted 1,2-dichloroethane is passed from said fourth fractionation column to a third distillation zone comprising a fifth fractionation column and a sixth fractionation column;
an overhead stream comprising 1,1,2-trichloro-1,2,2-trifluoroethane is recovered from said fifth fractionation column as another product of the process;
a bottoms stream comprising said other fluorinated materials and said unreacted 1,2-dichloroethane is passed from said fifth fractionation column to said sixth fractionation column; and
an overhead stream comprising 1,2-dichloro-1-monofluoroethane and said unreacted 1,2-dichloroethane is recovered from said sixth fractionation column and recycled to said cell as a portion of said feedstock.

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