

METHOD OF PRODUCING HYDROFLUOROCARBONS

FIELD OF INVENTION

The present invention relates generally to the preparation of hydrofluorocarbons and fluorocarbons. More specifically, the invention relates to a catalytic fluorination process that improves the yield of the desired hydrofluorocarbon/fluorocarbon and prolongs the activity of the fluorination catalyst used.

BACKGROUND OF THE INVENTION

The production of hydrofluorocarbons (HFCs) and fluorocarbons (FCs) is well known in the art. Generally, production methods involve fluorinating chlorinated organic compounds to produce the desired HFC or FC compounds, and then recovering the desired compounds through distillation. Among the preferred fluorination methods is vapor-phase, catalytic fluorination. For example, in difluoromethane (HFC-32) production, a chlorinated organic compound, such as, for example, methylene chloride (CH_2Cl_2), and a fluorinating agent, such as, for example, hydrogen fluoride (HF), are preheated and reacted with each other in the presence of a fluorination catalyst to generate a product stream.

The desired HFC or FC compound is recovered by the use of distillation from the product stream which also contains other materials such as by-products of the reaction. Distillation is well known in the art and typically involves the use of distillation means, such as a recycle column, which is operated at particular pressures and temperatures to separate the desired compound from the product stream. Distillation pressure and temperature are interrelated such that higher operating pressures generally correspond to higher distillation temperatures. Distillation temperatures dictate a column's cooling requirements.

For purposes of discussion herein, there are basically two type of cooling--high temperature and low temperature. High-temperature cooling refers to cooling to a temperature no less than about 0°C . Such high-temperature cooling can be achieved relatively easily and inexpensively using common equipment and

refrigerants. On the other hand, low-temperature cooling relates to cooling to a temperature no greater than about -15°C . The equipment and refrigerants required for low-temperature cooling tend to be substantially more expensive than those needed for high-temperature cooling. Thus, in industry, it is highly preferable to operate the recycle column within a pressure range which permits the use of high-temperature cooling, hereafter referred to as "high-temperature distillation."

To achieve proper flow of the product stream, the recycle column is operated at a pressure lower than that of the reactor. Therefore, taking into account the pressure drop between the reactor and the recycle column, the customary practice is to conduct the reaction at a pressure which permits the use of high-temperature distillation. A reaction pressure sufficient for high-temperature distillation is determined readily by someone skilled in the art of distillation. It depends upon a number of factors, including, for example, the pressure differential between the reactor and the recycle column, the phase characteristics of the desired product, and the other product stream constituents. For example, a satisfactory pressure for the high-temperature distillation of HFC-32 is above about 100 psig.

Although used widely, the prior art methods of producing HFCs and FCs through fluorination and distillation suffer from several shortcomings. Among the more significant shortcomings is catalyst deactivation during fluorination. This leads to lower yields. In an attempt to maintain a catalyst's activity, a regenerating agent, such as chlorine, is typically co-fed with the reactants into the reactor in continuous fashion. The continuous addition of chlorine, however, adds to the formation of generally undesirable by-products.

By-products complicate distillation and can significantly reduce product yield and decrease product quality. For example, in the production of HFC-32, the formation of chlorofluoromethane (CFC-12) is increased substantially with the use of increased amounts of chlorine. Unfortunately, CFC-12 and HFC-32 form a low-boiling azeotropic mixture from which it is difficult to separate the desired HFC-32 product. Distillation claims about 10 parts HFC-32 for every part CFC-12 removed from the product. Even if the commercial production volume is high

enough to accommodate such losses, the unwanted by-products present disposal problems.

Therefore, a need exists for preparing HFCs and FCs by a process that does not suffer from the aforementioned and other shortcomings. The present invention fulfills this need among others.

DESCRIPTION OF THE INVENTION AND PREFERRED EMBODIMENTS

The present invention identifies a reaction pressure range for producing fluorinated organic compounds, such as hydrofluorocarbons and fluorocarbons, in a fluorination/distillation process that minimizes catalyst deactivation and the formation of unwanted by-products and that increases yields. The identified reaction pressure range is lower than customarily practiced, and preferably, lower than the pressure conventionally used for high-temperature distillation.

Although relatively high reaction pressures are preferred in the prior art production of HFCs and FCs due to the advantages of high-temperature distillation, a significant and surprising reduction in catalyst deactivation has been realized by using relatively low reaction pressures in accordance with the present invention. Consequently, the amount of oxidizing agent (for example, chlorine) needed to regenerate the catalyst is reduced. The use of smaller amounts of oxidizing agent in turn reduces the formation of undesirable by-products which are responsible for low product yields, which complicate distillation, and which present disposal problems. Therefore, the use of the present invention's "low pressure" process results in the formation of a product stream that is more readily distillable than that produced by the use of a "high pressure" process of the prior art. This provides for higher product yields, simpler distillation, and lower by-product formation.

In a preferred embodiment, the present invention provides a process for producing a fluorinated organic compound comprising: (a) reacting reaction medium, having at least a fluorination agent and an organic compound, in the presence of a fluorination catalyst, while maintaining a relatively low reaction pressure, to produce a product stream; and (b) recovering the desired fluorinated organic compound from the product stream by distillation. Each of these steps is described hereafter in greater detail. For illustrative purposes, the preparation of

difluoromethane is considered specifically, although it should be understood that the process of the present invention may be applied to the preparation of a variety of HFCs and FCs, such as, for example, pentafluoroethane (HFC-125), 1,1,1,2 tetrafluoroethane (HFC-134a), and hexafluoroethane (FC-116).

5 The reaction medium used in the process of the present invention comprises a fluorination agent and an organic compound.

 A suitable fluorination agent includes any material capable of providing fluorine in the reaction. A preferred fluorination agent is substantially anhydrous hydrogen fluoride (HF). The presence of water in the reaction tends to deactivate
10 the fluorination catalyst. The term "substantially anhydrous", as used herein, means that the HF contains less than about 0.05 weight % water and preferably contains less than about 0.02 weight % water. It should be understood, however, that the presence of water in the catalyst can be compensated for by increasing the amount of catalyst used.

15 The organic compound may be any compound that contains a carbon-bonded chlorine or other atom replaceable by fluorine and/or that contains a carbon-carbon unsaturated bond that is saturatable with fluorine. Suitable organic compounds include, for example, hydrochlorofluorocarbons (compounds containing carbon, chlorine, fluorine and hydrogen), hydrochlorocarbons
20 (compounds containing carbon, chlorine and hydrogen) and chlorofluorocarbons (compounds containing carbon, chlorine and fluorine), chlorocarbons (compounds containing carbon and chlorine) or mixtures of two or more thereof. In a preferred embodiment, the chlorinated organic is methylene chloride (HCC-30).

 The reaction medium includes preferably recycled material which is
25 recovered from the product stream. If a continuous recycle stream of a high-boiling fraction obtained in the distillation is added to the reaction medium, a large excess of fluorination agent to organic compound should be used. Using higher mole ratios of fluorination agent to organic compound generally results in higher yields and selectivity. Additionally, the use of a large excess of fluorination agent
30 will decrease catalyst deactivation rates and result in less decomposition in preheaters and vaporizers, especially when the reaction is conducted at pressures in excess of 3 atmospheres. In the production of HFC-32, a large excess of HF

will result also in the reduction of hydrochlorofluoromethane (HCFC-31) produced as well as the concentration of unreacted HCC-30. Generally, a ratio of HF to HCFC-31, as measured after separation of HFC-32 from the product stream, of at least about 25:1 to at least about 300: 1, preferably at least about 50:1 to at least about 200:1 and more preferably at least about 75:1 to at least about 150:1 is used.

Although the reaction medium preferably includes recycled material, fresh starting materials may be used. When the process is run without continuous recycle, a sufficient amount of fluorination agent should be supplied to the reaction to provide at least a stoichiometric amount of fluorine relative to the chlorine of the chlorinated organic compound. In the preferred embodiment, wherein HF and HCC-30 are used, the HF to HCC-30 mole ratio is preferably from about 1:1 to about 10: 1, and more preferably from about 1:1 to about 4:1. Optionally, fresh HCFC-31 may be added to the reaction medium.

As desired, one or more of the reactants comprising the fluorination agent and the chlorinated organic compound may be preheated in at least one vaporizer before feeding to the reactor. The term "preheating" refers to vaporizing and optionally superheating the reactants. Suitable temperatures for preheating range from about 125° to about 400°C, preferably from about 150° to about 350°C, more preferably from about 175° to about 275°C, and still more preferably from about 200° to about 250°C. The vaporizer, as well as other vessels used in this process, may be made of any suitable corrosion resistant material.

The reactor is charged preferably with a fluorination catalyst before feeding the reactants to the reactor. The term "fluorination catalyst", as used herein refers to an inorganic metal catalyst which promotes a reaction involving the substitution of fluorine for chlorine in a chlorinated organic molecule. Such fluorination catalysts are known to those skilled in the art. Exemplary catalysts include, without limitation, chromium, copper, aluminum, cobalt, magnesium, manganese, zinc, nickel and iron oxides, hydroxides, halides, oxyhalides and inorganic salts thereof, $\text{Cr}_2\text{O}_3/\text{Al}_2\text{O}_3$, $\text{Cr}_2\text{O}_3/\text{AlF}_3$, $\text{Cr}_2\text{O}_3/\text{carbon}$, $\text{CoCl}_2\text{CrO}_3/\text{Al}_2\text{O}_3$, $\text{NiCl}_2/\text{Cr}_2\text{O}_3/\text{Al}_2\text{O}_3$, $\text{CoCl}_2\text{AlF}_3$ and $\text{NiCl}_2/\text{AlF}_3$. Additionally, supported metal catalysts such as nickel, cobalt, zinc, iron, and copper supported on chromia,

magnesia, or alumina may be used. Such chromium oxide/aluminum oxide catalysts are known and are described, for example, in U.S. Patent No. 5,155,082. Preferably, chromium oxide, a commercially available catalyst, is used. The chromium oxide may be crystalline or amorphous. Preferably, amorphous chromium oxide is used.

Before adding the reactants to the reactor, it may be preferable to pretreat the catalyst chemically and/or physically to create active sites which facilitate a fluorination reaction. For example, the catalyst can be pretreated by calcining it under a flow of inert gas such as nitrogen at a temperature comparable to or higher than that of the fluorination reaction. Next, the calcined catalyst is exposed to a fluorinating agent alone or in combination with up to about 5 to about 99 weight percent of an inert gas at a temperature from about 200°C to about 450°C for at least about an hour. Preferably, the catalyst is subjected to a third step in which an oxidation agent, such as chlorine, is contacted with the catalyst to improve its reactive properties further. Preferably, the chlorine is diluted with from about 60 to about 75% HF and/or from about 20 to about 30% of an inert gas. The chlorine may be passed over the catalyst at a total volume chlorine to total volume catalyst ratio of about 1: 3,000, preferably about 10:1,000, more preferably about 50:500. Exposure time may be from about 1 to about 200 hours, preferably about 5 to about 70 hours, more preferably about 10 to about 30 hours. The chlorine exposure may be conducted at any temperature and pressure convenient to the fluorination reaction.

The reactants can be fed individually or as a mixture to the reactor to form a reaction medium. Once the reaction is under way, the reactants may be continuously added under pressure to supply the additional amounts of reactants needed to continue the process.

The temperature at which the fluorination reaction is conducted and the period of reaction will depend on the starting materials, amounts used, and catalyst used. One of ordinary skill in the art can optimize readily the conditions of the reaction to obtain the desired results. Temperatures are generally between about 125° and about 425°C, preferably between about 150° and about 300°C, and still more preferably between about 200° and about 250°C.

Contact times are dependent on several factors including, for example, catalyst concentration, the type of catalyst, and the temperature. The time required for the reactants to pass through the catalyst bed (assuming a 100% void catalyst bed) is typically from about 1 to about 120 seconds, preferably from about 2 to 60 seconds, more preferably from about 4 to about 50 seconds, and still more preferably from about 5 to about 30 seconds.

As mentioned above, the process of the present invention is conducted at relatively low pressure compared to customary practice which typically involves pressures high enough to ensure high-temperature distillation. The minimum pressure needed for high-temperature distillation depends upon the product stream, more specifically, the low-boiling point fraction of the product stream. The distillation pressure should be high enough to partially condense the low-boiling point fraction at a given temperature. In the case of high-temperature distillation, the temperature is greater than about 0°C. Preferably, the reaction pressure used in the process of the present invention is lower than that which is needed to effect high-temperature distillation. For example, the minimum pressure needed for the high-temperature distillation of HFC-32 is above about 100 psig.

In accordance with the process of the present invention, the reaction pressure typically should be no greater than about 135 psig, more preferably between about atmospheric pressure and about 100 psig, still more preferably between about 20 psig and about 80 psig, and most preferably between about 40 psig and about 75 psig.

The process of the present invention may comprise an optional step in which an oxidizing agent is added to the reaction to regenerate the catalyst. Suitable oxidizing agents are well known in the art. They include, for example, elemental chlorine or oxygen. The oxidizing agent can be added in any suitable way, for example, the oxidizing agent can be added continuously or intermittently, for example, by mixing it with the reactants and feeding it as needed to maintain catalyst activity. In the preferred form, the oxidizing agent is added periodically which reduces the need for monitoring the reaction and the oxidizing feed on a continuous basis. Alternatively, in accordance with prior art techniques, catalyst activity may be maintained by regenerating the catalyst during periodic shut downs

of the reaction. Since the catalyst does not deactivate as quickly under the operating pressures of the present invention, the need to shut down the reactor is less frequent than under the operating conditions of the prior art.

5 As mentioned above, by conducting the reaction at a pressure prescribed herein, the catalyst tends to maintain its activity for a longer period of time than if the reaction is carried out at higher pressures. Accordingly, reduced amounts of oxidizing agent can be used. This translates to a reduction in the formation of undesirable by-products. For example, under optimal conditions in the high pressure production of HFC-32, the amount of CFC-12 found in low-boiling
10 fraction is usually higher than about 500 ppm. The corresponding amount of CFC-12 is significantly lower when using the process of the present invention. The concentration of CFC-12, at preferred pressures, is less than about 250 ppm, at more preferred pressures less than about 100 ppm, and at still more preferred pressures less than about 50 ppm.

15 The desired HFC or FC compound is recovered from the reaction mixture using conventional apparatus and techniques. In the preferred embodiment, the product stream is separated into low- and high-boiling fractions, and then the desired compound is recovered from its respective fraction. For example, in the production of difluoromethane, the product stream is likely to include also
20 HCFC-31 and HCl, as well as unreacted feed stock such as HF and HCC-30. This product stream is fed into the recycle column for separation. The high-boiling fraction, or bottom stream, from the stripper comprises unreacted HF and HCC-30 and intermediate reactant HCFC-31. Preferably, this mixture is recycled to the reactor as mentioned above. The low-boiling fraction, or top stream, which
25 contains difluoromethane, HCl, HF, and reaction by-products, is recovered.

Alternatively, separation of the fractions may be performed in two steps. In the first step, the product stream is quenched, that is, the temperature of the product stream is reduced to below its dew point. Quenching may be conducted in a packed column containing any suitable corrosion-resistant packing material and a
30 suitable refluxing liquid such as HF, HCC-30, and/or HCFC-31. The quenched product is fed subsequently into a recycle column.

Substantially pure product is recovered from the low-boiling fraction by any suitable method. Preferably, the recovery is performed by a series of substeps including, treating the gaseous mixture in an HCl distillation column or aqueous HCl absorption tower under conditions suitable to remove HCl and trace HF. The crude product, such as difluoromethane, is then treated with a first caustic scrubber under conditions which effect neutralization of residual acidity and which form a neutralized product. Typically, the caustic scrubber contains water, sodium hydroxide, or potassium hydroxide. Next, the neutralized product is treated in a second caustic scrubber, preferably comprising sodium hydroxide together with a sulfite, such as sodium sulfite under conditions which are effective in removing residual chlorine and in forming a substantially chlorine-free product. The substantially chlorine-free product is then treated with a sulfuric acid scrubber followed by a solid desiccant, such as any suitable, commercially available, molecular sieve that absorbs residual moisture from the product-containing gas stream to form a substantially moisture-free product. Finally, the substantially moisture-free product is conducted through a plurality of distillation columns under conditions sufficient to remove the residual impurities and produce substantially pure product, for example, greater than 99.97 weight percent difluoromethane. Any residual HCFC-31 removed in this last step may be recycled as mentioned above. In the case of difluoromethane production, the reduction of CFC-12 and, consequently, the CFC-12/HFC-32 azeotrope minimizing the distillation steps above.

EXAMPLES

The examples described below are illustrative of the practice of the invention. More specifically, the examples describe a low pressure process for producing difluoromethane in which catalyst deactivation and the formation of undesirable by-products are reduced relative to the results obtained in the use of a high pressure process.

Example 1

This example illustrates that maintaining the reactor at relatively low pressures in the production of difluoromethane prolongs the activity of the fluorination catalyst. A 4 in diameter Monel 400 reactor was charged with 4 l of chromium oxide catalyst which was subjected to the following pretreatment process. The catalyst was dried under 20 standard liters per minute (slpm) nitrogen flow at a temperature of 30°C for 8 h. The catalyst was conditioned by adding HF at a rate of 0.2 lb/h to the nitrogen flow at a reactor temperature of 250°C. The HF flow was increased to 1 lb/h at a rate low enough to prevent an excessive catalyst exotherm, which could lead to the well known "glow" phenomenon. The temperature was then gradually increased to 350°C and held for 4 h. The catalyst bed temperature was then decreased to 250°C and chlorine introduced to the HF/N₂ mixture at a rate of 500 standard cubic centimeters per minute (sccm) for a period of 24 h.

After this pretreatment procedure, the chlorine and nitrogen flows were discontinued and HCC-30 was mixed with HF and passed through a preheater at 185°C. The vaporized HCC-30 and HF mixture was fed to the reactor which was maintained at a predetermined pressure, as set forth in Table 1 below. The effluent from the reactor was quenched using a heat exchanger and fed into a recycle column maintained at a pressure 5 - 10 psig below the reactor pressure. The low-boiling distillation components, HCFC-31, HF and HCC-30, were recycled to mix with fresh HF and HCC-30 feed stream and were fed to the preheater and reactor at a flow rate of 4 lb/in. The fresh HF and HCC-30 feed rates were maintained at 0.6 and 1.2 lb/in respectively.

The experimental procedure involved conducting reactions at different pressures, as set forth in Table 1 below. For the experiments at 45 and 75 psig, chlorine was added to the feed mixture at a flow rate of 200 sccm for a period of 6 - 8 h as needed to maintain catalyst activity. At 135 psig operation, chlorine was added in an amount necessary to bring catalyst activity up to previous levels, typically 300 - 400 g over a 6 h period. The resulting HCC-30 conversion was 85 - 95%. A sudden drop in conversion to 60 % signaled the need for chlorine addition in order to bring the conversion back up to previous levels. The time

between required chlorine additions to maintain catalyst activity was surprisingly found to increase at the lower pressures of the process of the present invention. This is shown in Table 1 below.

Table 1 - Chlorine Addition

<u>Reactor Pressure (psig)</u>	<u>Time Between Cl Additions (hrs.)</u>
45	100
75	60
135	20

Example 2

This example illustrates that maintaining the reactor at relatively low pressures in the production of difluoromethane reduces the formation of low-boiling by-products. The different pressures used in the comparative tests are identified in Table 2 below. The other reaction conditions were as described in Example 1. The low-boiling components separated in the distillation column, HCl and HFC-32, were passed through a caustic scrubber containing 10 % KOH where HCl was removed. The crude HFC-32 product was dried and collected.

Additional low-boiling by-products that may be formed during the reaction are CFC-12, HCFC-22 and HFC-23. These by-products represent significant yield losses, especially CFC-12 which forms an azeotrope with HFC-32. The rate of formation of these by-products was surprisingly found to increase with increasing reactor pressure, as shown in Table 2 below. The by-product concentration is shown as parts per million (ppm) of the difluoromethane (HFC-32) in the crude product. The time on stream is expressed as the number of hours of operation at the specified pressure.

Table 2
Low-Boiling By-Products

<u>Pressure (psig)</u>	<u>Time on Stream (h)</u>	<u>CFC-12 (ppm)</u>	<u>HCFC-22 (ppm)</u>	<u>HFC-23 (ppm)</u>
45	260	n.d.	n.d.	n.d.
75	240	n.d.	n.d.	n.d.
75	977	19	102	1497
135	577	654	2815	2816

n.d. = not detected, detection limit 1 ppm.

As shown in Table 2 above, the low-boiling by-products are only observed at low pressure (45 and 75 psig) after a significant length of time on stream, whereas at high pressure (135 psig), they are immediately observed at high levels.

5 Example 3

This example illustrates that maintaining the reactor at relatively low pressures in the production of difluoromethane reduces the formation of high-boiling by-products. The different pressures used in the comparative tests are identified in Table 3 below. The other reaction conditions were as described in

10 Example 1. High-boiling byproducts may be formed in the process. Examples of high-boiling by-products are methyl chloride (HCC40) and HCFC-2 1. Although these materials are recycled to the reactor, along with the unreacted HF, HCC-30 and HCFC-3 1, they represent, nevertheless, significant yield losses.

Furthermore, HCC-40 reacts very slowly to HFC-41 and thus accumulates in the

15 recycle stream, requiring periodic shut downs to purge the system. The rate formation of these high-boiling by-products was surprisingly found to increase with increasing reactor pressure, as shown in Table 3 below. The concentrations are given as a weight percent of the total organic concentration of the recycle stream.

20 Table 3
High-Boiling By-Products

<u>Pressure</u> <u>(psig)</u>	<u>Time on</u> <u>Stream (h)</u>	<u>HCC-40</u> <u>(wt. %)</u>	<u>HCFC-21</u> <u>(wt %)</u>
45	260	n.d.	n.d.
25 75	914	n.d.	n.d.
135	180	0.23	n.d.
135	582	0.64	n.d.
135	703	0.97	1.34

30 As shown in Table 3 above, the high-boiling by-products are not detectable at low pressure (45 and 75 psig) after a significant length of time on stream, whereas at high pressure (135 psig), HCC-40 is observed after a relatively short time and HCFC-21 after a longer time.

CLAIMS

WHAT IS CLAIMED IS

1. A process for producing a fluorinated organic compound comprising:
reacting an organic compound with a fluorination agent in the presence of a
fluorination catalyst, while maintaining a pressure less than
sufficient for high-temperature distillation, to produce a product
stream; and
recovering said fluorinated organic compound from said product stream by
low-temperature distillation.
2. The process of claim 1, wherein said reaction pressure is maintained at no
greater than about 100 psig.
3. The process of claim 2, wherein said reaction pressure is maintained at
between about 20 and about 80 psig.
4. The process of claim 3, wherein said reaction pressure is maintained at
between about 40 and about 75 psig, and said low-temperature distillation is
performed at no greater than about -15°C.
5. The process of claim 1, further comprising
periodically contacting said catalyst with oxidizing agent.
6. A process for producing a fluorinated organic compound comprising:
reacting an organic compound with a fluorination agent in the presence of a
fluorination catalyst, while maintaining a pressure not greater than
about 135 psig, to produce a product stream; and
recovering said fluorinated organic compound from said product stream by
distillation.
7. The process of claim 6, wherein said reaction pressure is maintained at
between about atmosphere and about 100 psig.



8. The process of claim 7, wherein said reaction pressure is maintained at between about 20 and about 80 psig.
- 5 9. A process for producing difluoromethane comprising:
reacting a methylene chloride vapor with hydrogen fluoride vapor in the
presence of a fluorination catalyst, while maintaining less than
sufficient pressure for high-temperature distillation, to produce a
product stream; and
recovering said difluoromethane from said product stream by low-
10 temperature distillation.
10. The process of claim 9, wherein said reaction pressure is maintained at no greater than about 100 psig.
- 15 11. The process of claim 10, wherein said reaction pressure is maintained at between about 20 and about 80 psig.
12. The process of claim 11, wherein said reaction pressure is maintained at between about 40 and about 75 psig.
- 20 13. The process of claim 9, further comprising
periodically contacting said catalyst with oxidizing agent.
- 25 14. A process for producing difluoromethane comprising the steps of:
reacting a methylene chloride with hydrogen fluoride in a reactor charged
with a fluorination catalyst to produce a product stream, while
maintaining reaction pressure such that CFC-12 concentration is no
more than about 500 ppm of said low-boiling point fraction;
recovering by distillation from said product stream a high-boiling fraction
30 and a low-boiling fraction, said low-boiling fraction comprising
difluoromethane; and
recovering said difluoromethane from said low-boiling fraction.



15. The process of claim 14, wherein said reactor pressure is maintained such that CFC-12 concentration is no more than about 100 ppm of said low-boiling point fraction.

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16. The process of claim 15, wherein said reactor pressure is maintained such that CFC-12 concentration is no more than about 50 ppm of said low-boiling point fraction.

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17. The process of claim 16, wherein said reactor is maintained at a pressure no greater than about 100 psig and at a temperature no greater than about -15°C.

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18. A process for maintaining the activity of a fluorination catalyst in a reaction in which a fluorinated carbon compound is synthesized by reacting a chlorinated organic compound with a fluorination agent in a reactor charged with said fluorination catalyst to produce a product stream from which the fluorinated carbon compound is recovered by distillation; said process comprising:

maintaining the reactor at a pressure less than sufficient for high-temperature distillation.

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19. The process of claim 14, wherein said reactor pressure is maintained at between about atmospheric and about 100 psig.

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20. The process of claim 15, wherein said reactor pressure is maintained at between about 20 psig and about 80 psig.

