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(54) Titre : PROCÉDE POUR LA PRÉPARATION DES ALPHA-OLEFINES  
(54) Title: PROCESS FOR THE PREPARATION OF ALPHA OLEFINS

(57) **Abrégé/Abstract:**

This invention relates to a process for the preparation of an alpha olefin product which comprises metathesizing an internal olefin under non-equilibrium conditions and thereby producing a lower boiling internal olefin product which is removed as formed, and a higher boiling mid-chain internal olefin product, thereafter contacting and reacting the higher boiling mid-chain internal olefin product with ethylene to produce an alpha olefin product having a narrow molecular weight range.





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<b>(21) International Application Number:</b> PCT/EP97/01400 <b>(22) International Filing Date:</b> 18 March 1997 (18.03.97) <b>(30) Priority Data:</b> 08/618,179                      19 March 1996 (19.03.96)                      US <b>(71) Applicant (for all designated States except CA):</b> SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V. [NL/NL]; Carel van Bylandtlaan 30, NL-2596 HR The Hague (NL). <b>(71) Applicant (for CA only):</b> SHELL CANADA LIMITED [CA/CA]; 400 - 4th Avenue S.W., Calgary, Alberta T2P 2H5 (CA). <b>(72) Inventor:</b> LUTZ, Eugene, Frederick; 11135 Tupperlake, Hous- ton, TX 77042 (US).		<b>(81) Designated States:</b> BR, CA, CN, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> PROCESS FOR THE PREPARATION OF ALPHA OLEFINS		
<b>(57) Abstract</b>  This invention relates to a process for the preparation of an alpha olefin product which comprises metathesizing an internal olefin under non-equilibrium conditions and thereby producing a lower boiling internal olefin product which is removed as formed, and a higher boiling mid-chain internal olefin product, thereafter contacting and reacting the higher boiling mid-chain internal olefin product with ethylene to produce an alpha olefin product having a narrow molecular weight range.		

## PROCESS FOR THE PREPARATION OF ALPHA OLEFINS

This invention relates to a process for the preparation of an alpha olefin product having a narrow molecular weight range which comprises metathesizing an internal olefin under non-equilibrium conditions and thereby producing a lower boiling internal olefin product which is removed during formation, and a higher boiling mid-chain internal olefin product, thereafter contacting and reacting the mid-chain internal olefin product with ethylene and thereby producing an alpha olefin product having a narrow molecular weight range.

Reactions of olefinic molecules in the presence of metal-containing catalysts to produce other olefinic molecules are known in the art as "metathesis" or "disproportionation" reactions. The olefin disproportionation reaction can be visualized as the breaking of two existing double bonds between the second and third carbon atoms in one molecule, and between the fourth and fifth carbon atoms in a second molecule, respectively, and the formation of two new olefins, where one new double bond is between the second and fourth carbon atoms noted and the second new double bond is between the third and fifth carbon atoms noted, respectively. A typical olefin disproportionation process is illustrated by U.S. Patent No. 3,261,879, issued July 19, 1966, to Banks, wherein two similar non-symmetrical molecules of an olefin react in the presence of certain catalysts to produce one olefin of a higher carbon number and one olefin of a lower carbon number. For example, propylene disproportionates by the process of U.S. Patent No. 3,261,879 to produce ethylene and butylenes.



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It is recognized in the industry that some molecular weight ranges of alpha olefins are more valuable than others. While there are many processes for the production of alpha olefins, most of these processes result in a wide range of olefin products. It would therefore be advantageous to have a process which would result in the production of an alpha olefin product having a selective narrow molecular weight range.

The present invention is therefore directed to a process in which internal olefins can be subjected to a metathesis step followed by an ethenolysis step in order to selectively produce a narrow molecular weight range of alpha olefins.

The present invention therefore relates to a process for the preparation of an alpha olefin product which comprises: a) metathesizing an internal olefin having from 6 to 30 carbon atoms under non-equilibrium conditions and thereby producing a lower boiling internal olefin product which is removed during formation, and a higher boiling mid-chain internal olefin product, and b) contacting and reacting the higher boiling mid-chain internal olefin product with ethylene, thereby producing an alpha olefin product having a narrow molecular weight range.

As used herein, the term "lower boiling" internal olefin product is used to refer to an internal olefin product having a boiling point lower than that of the starting internal olefin feed. The term "higher boiling mid-chain" internal olefin product is used to refer to an internal olefin product in which the double bond is at or near the middle of the chain, as can be determined by nuclear magnetic resonance spectrometry (NMR) analysis, and which has a boiling point greater than that of the starting internal olefin feed. The term "ethenolysis", as used herein, refers to the reaction of an olefin with





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non-equilibrium conditions, i.e., one of the products is removed as formed.

In general, any disproportionation or metathesis catalysts previously employed in such reactions can be utilized in the first step of the present process. A variety of catalysts have been employed for conducting disproportionation reactions, such as those disclosed in U.S. Patent No. 3,340,322, issued September 5, 1967; U.S. Patent No. 3,637,892, issued January 25, 1972; U.S. Patent No. 3,760,026, issued September 18, 1973; U.S. Patent No. 3,792,108, issued February 12, 1974; and U.S. Patent No. 3,872,180, issued March 18, 1975. Among the catalysts that have been developed for disproportionation are inorganic refractory materials containing molybdenum and/or tungsten oxide. These catalysts may also contain a promoter to enhance the disproportionation catalyst activity. Elemental metal promoters selected from the group consisting of barium, magnesium, tungsten, silicon, antimony, zinc, manganese and tin are disclosed in U.S. Patent No. 4,568,788, issued February 4, 1986, U.S. Patent No. 4,522,936, issued June 11, 1985, U.S. Patent No. 4,524,235, issued June 18, 1985 and U.S. Patent No. 4,629,719, issued December 16, 1986. In addition, organometallic compounds, such as aluminium and tin alkyls to promote solid catalysts including molybdenum and rhenium oxide for the disproportionation are disclosed in U.S. Patent No. 4,454,368, issued June 12, 1984 and U.S. Patent No. 3,829,523, issued August 13, 1974.

While any of the aforementioned metathesis or disproportionation catalysts may be utilized, the catalyst generally utilized in the metathesis step of the present invention is one in which molybdenum or rhenium oxide are deposited on an inorganic oxide support, preferably on a support of silica, alumina or aluminium phosphate. Prior to its use, the catalyst is

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typically activated by calcination carried out in a conventional manner. A particularly suitable catalyst is molybdenum oxide supported on alumina.

Suitable metathesis step reaction conditions, e.g.,  
5 temperature, pressure, flow rates, etc., vary somewhat depending upon the specific catalyst composition, the particular feed olefin, etc. The metathesis step is typically carried out at temperatures ranging from -10 °C to 250 °C and at pressures in the range of 0.01333 to  
10 266.6 kPa (0.1 mm Hg to 2000 mm Hg). The metathesis step is usually effected in a liquid phase and if desired, liquid reaction diluents are utilized. Examples of suitable diluents are saturated hydrocarbons. If the diluent is present, it is present in amounts up to 20  
15 moles of diluent per mole of internal olefinic reactant.

In the first step of the process, the starting internal olefin feed is subjected to metathesis with the resulting products being a lower boiling internal olefin product which is removed as it is formed by reactive  
20 distillation, and a higher boiling mid-chain internal olefin product. It is essential that the lower boiling internal olefin product be removed during formation in order to obtain the greatest amount possible of the desired higher boiling mid-chain olefin product which can  
25 then be converted by ethenolysis into the desired alpha olefin product having a narrow molecular weight range. If the lower boiling internal olefin product is not removed as it is formed, an equilibrium distribution of products will be formed and the higher boiling mid-chain  
30 olefin product will be formed in lesser amounts.

Following the metathesis step, the higher boiling mid-chain olefin product is separated by means of distillation from any unconverted starting internal olefin feed. Any unconverted starting olefin feed



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material is then recycled in order to maximize use of the internal olefin feed.

The higher boiling mid-chain internal olefin product from the metathesis step is then subjected to a second step, an ethenolysis step in which the higher boiling mid-chain internal olefin product is contacted and reacted with ethylene in the presence of an ethenolysis catalyst to form an alpha olefin product having a narrow molecular weight range.

Suitable ethenolysis catalysts, which are generally any ethenolysis catalysts known to be utilized in the reaction of ethylene with an olefin can be utilized in the second step of the present process. These catalysts are suitably supported on an inorganic oxide support. However, the ethenolysis catalyst suitable for use in the present invention should have little, if any, double bond isomerization activity, as the presence of such would result in side reactions leading to isomerization of alpha olefins to internal olefins thus resulting in the production of lower amounts of alpha olefins. Particularly suitable for use in the ethenolysis step of the present invention is a rhenium-on-alumina catalyst such as that disclosed in U.S. Patent No. 3,647,906, issued March 7, 1972. Also suitable would be a heterogeneous molybdenum oxide catalyst such as that disclosed in U.S. Patent No. 3,658,927, issued April 25, 1972. Any of the disproportionation catalysts mentioned with respect to the first step of the present process may also be utilized in this second step provided they have little or no double bond isomerization activity.

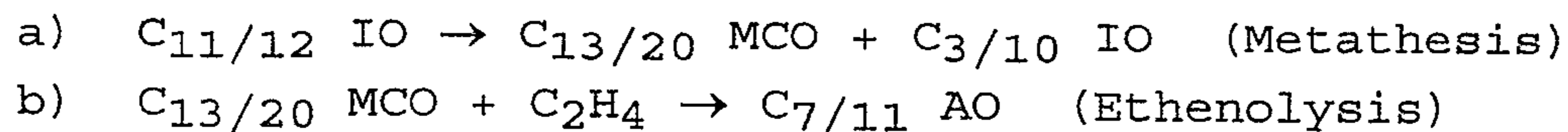
Suitable ethenolysis step reaction conditions, e.g., temperature, pressure, flow rates, etc., vary somewhat depending upon the specific catalyst composition, the particular feed olefin, etc. The ethenolysis step is typically carried out at temperatures ranging from about -10 °C to about 250 °C and at pressures in the range of



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272 to 10446 kPa (25 psig to 1500 psig). The ethenolysis step is usually effected in a liquid phase and if desired, liquid reaction diluents are utilized. Examples of suitable diluents are hydrocarbons which are free from aliphatic unsaturation, such as cyclic or alicyclic alkanes which can be readily separated from alpha olefin product and mid-chain olefin feed by conventional means, such as, for example, distillation. If the diluent is added, it is present in amounts up to 20 moles of diluent per mole of mid-chain internal olefinic reactant.

By way of illustration, the process of the present invention can be used as follows to metathesize a C<sub>11/12</sub> substantially linear internal olefin (IO) to produce a C<sub>13/20</sub> higher boiling mid-chain internal olefin (MCO) and a C<sub>3/10</sub> internal olefin which is removed as formed and thereafter ethenolyze the C<sub>13/20</sub> higher boiling mid-chain internal olefin (MCO) to produce a linear alpha olefin product having a narrow molecular weight range, i.e., C<sub>7</sub> to C<sub>11</sub> (AO):



The two-step process of the invention can be carried out either batchwise or continuously, using a fixed catalyst bed, or a stirrer equipped reactor or other mobile catalyst contacting process as well as any other well known contacting technique. With a fixed bed reactor, for example, continuous flow operation for the metathesis step is at pressures in the range of 0.01333 to 266.6 kPa (0.1 mm Hg to 2000 mm Hg), preferably 0.5333 to 101.3 kPa (4 mm Hg to 760 mm Hg), with catalysts having densities ranging from 0.3 to 2.0 gram per ml and surface areas greater than 100 m<sup>2</sup>/g, and at temperatures in the range of -10 °C to 250 °C, preferably at 30 °C to 150 °C, at volume hourly space velocities in the

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range of 0.1 to 10.0 parts by volume of mid-chain internal olefin feed per part by volume of catalyst per hour are suitable.

5 The narrow range alpha olefin products prepared according to the present invention can be utilized in a wide variety of applications. For example, the olefins can be used as comonomers for the manufacture of linear low density polyethylene, or the olefins can be oligomerized to synthetic lubricants. Higher molecular weight olefins are useful as intermediates in the preparation of surfactants.

10 The process of the instant invention will be further illustrated below by the following examples.

Examples

15 Catalyst A

Catalyst A, utilized in the examples below, was prepared by comulling 125.3 g of alumina with 6.0 g of molybdenum trioxide in the presence of an aqueous acetic solution made up by dissolving 5.6 g of glacial acetic acid in 101.5 g of deionized water. After mulling for 20 30-45 minutes, the resulting paste was extruded to produce 1.2 mm trilobes. Following drying at 121 °C for several hours, the extrudate was calcined at 510-593 °C for 2 hours.

25 Using a conventional dry pore volume impregnation technique, 100 g of trilobe extrudate was impregnated with 70 ml of an aqueous solution containing 8.0 g of ammonium dimolybdate. After aging for 2 hours, the catalyst was dried at 121 °C for 2-3 hours, and then 30 calcined at 427-538 °C for 2 hours.

\* Catalyst B

Catalyst B utilized in the examples below was prepared using a conventional dry pore volume impregnation technique. A solution suitable for 35 impregnating 150.0 grams of calcined alumina support with



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a pore volume of 0.8-1.0 ml/g was prepared as follows. An impregnation solution was made by combining 28.13 grams of ammonium perrhenate and 220.10 grams of deionized water. After adding the entire solution to the alumina support in several small portions with continuous rotary agitation, the impregnated support was dried in a Rotovac at 25 to 121 °C with vacuum decreasing for 47.996 kPa (360 mm Hg) to 0.533 kPa (4 mm Hg) and then calcined in air by heating from 37° to 500 °C at 2°/min and holding at 500 °C for 2 hours.

Example 1

a) Metathesis Step

In a nitrogen glove-box, 30 ml of Catalyst A was added to glass reactor having a glass wool plug to hold the catalyst in place. After the catalyst was loaded, a round bottomed flask was attached to the bottom of the tube while the top was sealed with a stopper to prevent air intrusion when the tube was removed from the glove-box. Outside of the glove-box, a three-necked flask was attached to the bottom and nitrogen was passed into the column via an oil bubbler such that the pressure could not exceed atmospheric pressure. With a nitrogen flow, the stopper at the top was removed and replaced with a thermocouple that extended half of the way into the catalyst bed. The thermocouple passed through a metal 24/40 joint that was fitted into a metal pipe such that a feed inlet and a vacuum outlet were available to add olefin to the top of the catalyst bed and to pull a vacuum about the feed inlet.

When the catalyst bed assembly was complete, a C<sub>8/9/10</sub> internal olefin was pumped at a rate of 30 ml/hour from an Isco pump into the top of the reactor. Pressure at the top and bottom of the reactor was about atmospheric (760 mm Hg) and warm oil at 119 °C was circulated through the reactor jacket. Approximately

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two hours after liquid break-through was achieved, a spot sample was collected in an ice bath for gas chromatography analysis. At the end of the run period, all product at the bottom of the reactor and all low boiling olefin in the cold trap was collected, weighed, and analyzed by gas chromatography. After the mid-chain olefin was recovered by distillation, NMR was used to determine that the double bond was at or near the center of the olefin chain.

Results obtained from the metathesis step are presented in Table I.

b) Ethenolysis Step

In a nitrogen glove-box, 15 ml of Catalyst B was charged to a metal, fixed bed, jacketed reactor. The catalyst was held in place with an inverted metal screen basket at the bottom of the tube. A thermocouple was extended from the top of the reactor to the center of the catalyst bed. The top of the fixed bed reactor was fitted with a feed inlet line, pressure relief valve, thermocouple, and pressure transducer. Both the top and the bottom had block valves so that the reactor could be sealed in the glove-box after catalyst loading. After the reactor was removed from the glove-box, it was equipped with liquid and gas feed lines for the inlet at the top and a Grove back-pressure regulator at the bottom for pressure control. Circulating hot oil was used to heat the reactor. When the reactor assembly was complete, it was tested for leaks at 3961 kPa (560 psig) nitrogen.

The fixed bed reactor was started by pumping the liquid C<sub>11/15</sub> higher boiling mid-chain internal olefin product from the metathesis step from an Isco piston pump downflow. When liquid break-through was achieved, ethylene was introduced and pressure was raised to 2168 kPa (300 psig) by means of the back-pressure



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regulator. Oil was circulated through the reactor jacket at 32 °C. As the reaction exothermed, a plastic bag of ice was inserted into the oil bath so that the temperature in the middle of the catalyst was maintained at 32±2 °C. Product was collected in an ice cooled round bottomed flask that vented to a dry ice cooled cold trap. The first spot sample to check conversion of the higher boiling mid-chain internal olefin was taken about two hours after ethylene was added to the reactor. Ethylene flow through the reactor was monitored regularly and maintained at about 30-40 ml/minute. At the end of a day's run, conversion and selectivity were determined by gas chromatography analysis. Linear alpha olefin content of product was determined by gas chromatography analysis before and after hydrogenation.

The results obtained from the ethenolysis step are presented in Table II.

#### Example 2

##### a) Metathesis Step

This was performed as in Example 1, except that catalyst B was used in an amount of 10 ml, the internal olefin feed was C<sub>11/12</sub>, pumped at the rate of 10 ml/hour, the pressure during the 2 hour metathesis step was 0.5333-0.6666 kPa (4-5 mm Hg) and the warm oil circulated through the reactor jacket had a temperature of 45 °C.

Results obtained from the metathesis step are presented in Table I.

##### b) Ethenolysis Step

This was performed as in Example 1, except that catalyst B was used in an amount of 20 ml and the high boiling mid-chain internal olefin product from the metathesis step was C<sub>13/20</sub>.

The results obtained from the ethenolysis step are presented in Table II.

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Comparative Example A

The ethenolysis step was carried out in a manner similar to the ethenolysis step in Example 2 above except that a C<sub>18</sub> internal olefin having a random double bond distribution was used instead of the C<sub>13/20</sub> higher boiling mid-chain internal olefin product.

The results obtained from the ethenolysis step are presented in Table II.

Example 310 a) Metathesis Step

This was performed as in Example 1, except that the amount of catalyst A used was 10 ml, the internal olefin feed was C<sub>11/12</sub>, pumped at the rate of 10 ml/hour, and the pressure during 2 hour metathesis was 17.33 kPa (130 mm Hg).

Results obtained from the metathesis step are presented in Table I.

15 b) Ethenolysis Step

This was performed as in Example 1, except that the amount of catalyst A used was 20 ml and the high-boiling mid-chain internal olefin product from the metathesis step was C<sub>13/20</sub>.

Product was collected in an ice cooled round bottomed flask that vented to a dry ice cooled cold trap. A spot sample was collected to determine higher boiling mid-chain internal olefin conversion and linear alpha olefin selectivity about three hours after the temperature reached 80 °C. Ethylene flow through the reactor was monitored regularly and maintained at about 30-40 ml/minute. Linear alpha olefin content of product was determined by gas chromatography analysis before and after hydrogenation.

The results obtained from the ethenolysis step are presented in Table II.



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Example 4a) Metathesis Step

5 This was performed as in Example 2, except that the amount of catalyst B used was 30 ml, the internal olefin feed was C<sub>14</sub>/17, pumped at the rate of 30 ml/hour, and the pressure during the 2.5 hour metathesis step was 0.133-0.266 kPa (1-2 mm Hg).

Results obtained from the metathesis step are presented in Table I.

10 b) Ethenolysis Step

This was performed as in Example 2, except that the amount of catalyst B used was 15 ml, the high-boiling mid-chain internal olefin product from the metathesis step was C<sub>18</sub>/25.

15 The results obtained from the ethenolysis step are presented in Table II.

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TABLE I  
Mid-Chain Olefins (MCO) via Fixed Bed Reaction LHSV, 1.0

Ex. No.	Internal Olefin Feed	Catalyst	Temp. °C	Vac., kPa	Vac., (mm Hg)	Conv. %wt	MCO Product	Double Bond at Mid-chain, %wt	Sel. %wt
1	C <sub>8/9/10</sub>	A	119	100	(~760)	62.0	C <sub>11/15</sub>	86	77
2	C <sub>11/12</sub>	B	45	0.53 <sup>-</sup> 0.67	(4-5)	88.6	C <sub>13/20</sub>	99	75
3	C <sub>11/12</sub>	A	120	17.33	(130)	85.6	C <sub>13/20</sub>	97	76
4	C <sub>14/17</sub>	B	45	0.133 <sup>-</sup> 0.267	(1-2)	55.5	C <sub>18/25</sub>	99	60



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TABLE II  
 Linear Alpha Olefins by Ethenolysis of MCO  
 Ethylene Pressure 2168 kPa (300 psig)

Ex. No.	MCO	Cat.	LHSV	Temp. °C	Conv., %wt	LAO Selectivity, %wt							LAO Content %wt <sup>b)</sup>		
						≤C6	C7/8	C9	C10/11	C12	C13/14	C15/16			
1	C11/15	B	1.0	32	65	24.1	46.2	20.9	8.7						93-97 <sup>c)</sup>
2	C13/20	B	1.0	32	45	5.0	19.0	16.9	57.2	1.9					97-98 <sup>d)</sup>
Comp. Ex. A	C18 IOa)	B	1.0	50	50	1.8	4.8	3.3	8.3	4.6	12.2	17.7			
3	C13/20	A	0.5	80	40	4.6	20.8	18.5	49.0	7.0					90 <sup>e)</sup>
4	C18/25	B	1.0	32	30	3.7	9.1	8.5	25.0	13.9	25.6	14.2			96-97 <sup>f)</sup>

- a) Internal olefin having a random double bond distribution.  
 b) Purity of principal products produced.  
 c) Principal product : C7/8  
 d) Principal product : C9/10/11  
 e) Principal product : C9/10/11  
 f) Principal product : C10/11/12

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Discussion of the Results

The key step in the present invention is the conversion of random internal olefins, which are non-symmetrical, i.e., olefins having different size alkyl groups attached to the double bond, to mid-chain olefins, which are symmetrical, i.e., olefins having about the same size alkyl groups attached to the double bond. As can be seen in Table II, Example 2 shows that ethenolysis of a C<sub>13/20</sub> mid-chain olefin produced a C<sub>7/12</sub> alpha olefin selectivity of 95.0% by weight. By contrast, in Comparative Example A, the ethenolysis of a random C<sub>18</sub> internal olefin results in the production of olefins in the range of from C<sub>3</sub> to C<sub>33</sub>, and, as can also be seen in Table II, gives a C<sub>7/12</sub> alpha olefin selectivity of 21.0% by weight. The production of the mid-chain olefins, as summarized in Table I, was accomplished by metathesizing random internal olefins under non-equilibrium, reactive distillation conditions where olefins having a lower boiling point than the starting internal olefin feed were flashed as formed, thus leaving higher boiling, more symmetrical mid-chain olefins. It is also important to note that the catalysts used, particularly those utilized in the ethenolysis step, should not have significant double bond isomerization activity as catalysts containing such activity will result in much poorer product selectivity and purity.



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CLAIMS:

1. A process for the preparation of a alpha olefin product which comprises: a) metathesizing an internal olefin having in the range of from 6 to 30 carbon atoms under non-equilibrium conditions and thereby producing a lower boiling internal olefin product which is removed as formed, and a higher boiling mid-chain internal olefin product, and b) contacting and reacting the higher boiling mid-chain internal olefin product with ethylene, thereby producing an alpha olefin product having a narrow molecular weight range.
2. The process of claim 1, wherein said internal olefin has in the range of from 6 to 24 carbon atoms.
3. The process of claim 1 or 2, wherein step a) is carried out in the presence of a metathesis catalyst comprising at least one of molybdenum or rhenium supported on an inorganic oxide support.
4. The process of any one of claims 1 to 3, wherein said step a) is carried out at a temperature in the range of from -10°C to 250°C and a pressure in the range of from 0.01333 kPa (0.1 mm Hg) to 266.6 kPa (2000 mm Hg).
5. The process of any one of claims 1 to 4, wherein step b) is carried out in the presence of a ethenolysis catalyst comprising at least one of molybdenum or rhenium supported on an inorganic oxide support.
6. The process of any one of claims 1 to 5, wherein step b) is carried out at a temperature in the range of from -10°C to 250°C and a pressure in the range of from 272 kPa (25 psig) to 10446 kPa (1500 psig).

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