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# JOURNAL OF MOLECULAR CATALYSIS A: CHEMICAL

*PatentsALERT*

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## CATALYST PREPARATION

**5707918**

### **Hydrocarbon isomerization catalyst blend**

Wu An-hsiang; Drake Charles A; Melton Ralph J. Bartlesville, OK, USA; assigned to Phillips Petroleum Company

A Group VIII metal- and chloride-containing composition is prepared by a method which comprises mixing (A) a solid material containing at least one Group VIII metal (Pt and/or Pd and/or Ni) and alumina as the support and (B) an alumina-supported aluminum- and chlorine-containing compound which has been prepared by mixing an alumina material with  $\text{AlCl}_3$  and/or at least one organoaluminum chloride (such as ethylaluminum dichloride), heating the mixture at a temperature of about  $600^\circ\text{--}750^\circ\text{C}$  and then with an HCl-containing gas at a temperature of about  $100^\circ\text{--}750^\circ\text{C}$ . The thus-obtained composition comprising components (A) and (B) is employed as a catalyst in the isomerization of C4–C8 alkanes and/or cycloalkanes (e.g., *n*-butane).

**5707921**

### **Method of preparing isomerization catalyst composition**

Wu An-hsian; Drake Charles A; Melton Ralph J. Bartlesville, OK, USA; assigned to Phillips Petroleum Company

A Group VIII metal- and chloride-containing composition (effective as an alkane/cycloalkane isomerization catalyst) is prepared by a method which comprises mixing at least one aluminum- and chlorine-containing compound (preferably  $\text{AlCl}_3$ ) with a solid material containing at least one Group VIII metal (Pt and/or Pd and/or Ni) and alumina, heating the obtained solid mixture in an inert gas, and then treating the solid mixture with a gas mixture comprising hydrogen gas and HCl and/or chloroalkane (such as  $\text{CCl}_4$ ).

**5711869**

### **Synthetic crystalline aluminosilicate for the catalytic conversion of hydrocarbons in petrochemical processes**

Tissler Arno. Tegernheim, GERMANY; assigned to Ecolith-Zeolith GmbH

The subject invention describes a synthetic crystalline aluminosilicate of the pentasil type and method for

using the same as a catalyst or a catalyst component in petrochemical processes for the catalytic conversion of hydrocarbons and their derivatives into useful organic compounds and intermediates.

**5711919**

### **Device for catalytic dehydrogenation a C2 + paraffinic charge comprising a self-cooling system**

Minkkinen Ar; Burzynski Jean-Pierre. Saint Nom La Breteche, FRANCE; assigned to Institut Francais du Petrole

A device for catalytic dehydrogenation of a C2 + paraffinic cut with an improved system for cooling the effluent is applicable, for example, to the synthesis of methyl *tert*-butyl ether. Liquid charge is evaporated in the calandria of a heat exchanger in the optional presence of at least one part recycled hydrogen, then optionally compressed in a compressor before being preheated in an exchanger by effluent 1 and introduced into a dehydrogenation reactor. The effluent cooled in the tubes of exchanger can be mixed with a cryogenic phase resulting from the isentropic expansion of a hydrogen-rich phase separated in a separator, the hydrogen optionally being recycled to the calandria of the heat exchanger. The olefins recovered with the unconverted paraffins are stabilized in a column.

**5712213**

### **Rejuvenation process for a solid alkylation catalyst**

Joly Jean-Francois; Benazzi Eric; Marcilly Christian; Euzen Jean-Paul; Forestiere Alain. Lyons, FRANCE; assigned to Institut Francaise Du Petrole

The invention concerns a rejuvenation process for a solid catalyst for the alkylation of at least one isoparaffin by at least one olefin. The process is characterised in that the catalyst is rejuvenated in the reaction zone by circulating an isoparaffin-rich liquid fraction over the bed, said fraction originating from a zone for separating the reaction effluent extracted from the reaction zone.

**5712214**

### **Regeneration of aromatization catalysts**

Huang Yao-Jyh Robert; Fung Shun Chong. Taipei, TAIWAN; assigned to Exxon Research & Engineering Company

A low-pressure, high-temperature, wet posttreatment after oxyhalogenation during regeneration to improve activity and selectivity recovery of a regenerated catalyst involves exposing a halogenated catalyst to a gaseous stream including water having a partial pressure of up to about 1.5 psia, oxygen having a partial pressure of less than about 4.5 psia, and an inert gas at a temperature within the range of about 450°C to about 530°C at a pressure within the range of about 14.7 psia to about 65 psia for a time sufficient to effect a hydrogen halide partial pressure in offgas from the halogenated catalyst of less than about 0.004 psia to effectively remove excess halide from the halogenated catalyst.

**5714659****Catalyst composition and processes therefor and therewith**

Wu An-hsiang; Drake Charles A. Bartlesville, OK, USA; assigned to Phillips Petroleum Company

A catalyst composition and a process for hydrodealkylating a C9 + aromatic compound, such as, for example, 1,2,4-trimethylbenzene to a C6 to C8 aromatic hydrocarbon, such as a xylene are disclosed. The composition comprises an alumina, a metal oxide, and an acid site modifier selected from the group consisting of silicon oxides, boron oxides magnesium oxides, tin oxides, titanium oxides, zirconium oxides, molybdenum oxides, germanium oxides, indium oxides, lanthanum oxides, cesium oxides, and combinations of any two or more thereof. The process comprises contacting a fluid which comprises a C9 + aromatic compound with the catalyst composition under a condition sufficient to effect the conversion of a C9 + aromatic compound to a C6 to C8 aromatic hydrocarbon.

**5714660****Catalyst composition and processes therefor and therewith**

Wu An-hsian; Drake Charles A; Melton Ralph J. Bartlesville, OK, USA assigned to Phillips Petroleum Company

A catalyst composition and a process for hydrodealkylating a C9 + aromatic compound, such as, for example, 1,2,4-trimethylbenzene to a C6 to C8 aromatic

hydrocarbon, such as a xylene are disclosed. The composition comprises an alumina, a metal oxide, and an acid site modifier selected from the group consisting of silicon oxides, phosphorus oxides, boron oxides, magnesium oxides, tin oxides, titanium oxides, zirconium oxides, molybdenum oxides, germanium oxides, indium oxides, lanthanum oxides, cesium oxides, and combinations of any two or more thereof. The process comprises contacting a fluid which comprises a C9 + aromatic compound with the catalyst composition under a condition sufficient to effect the conversion of a C9 + aromatic compound to a C6 to C8 aromatic hydrocarbon.

**5716896****One-step synthesis of methyl *t*-butyl ether from *t*-butanol using beta-zeolite catalysts modified with lithium plus rare earths**

Knifton John Frederick; Dai Pei-Shing Eugene. Austin, TX, USA

Disclosed is a method for producing alkyl tertiary alkyl ethers which comprises reacting *t*-butanol with an alkanol in the presence of a catalyst which exhibits extended life comprising beta-zeolite modified with one or more metals selected from the group consisting of Group IA and Group IIIB of the Periodic Table and continuously contacting said alkanol and *t*-butanol in a molar amount from about 10:1 to 1:10 over said zeolite catalyst at a temperature of about 20°C to about 250°C and a pressure of about atmospheric to about 1000 psig.

**5716897****Sulfur tolerant hydrocarbon conversion catalyst**

Galperin Leonid B; Bedard Robert. Wilmette, IL, USA; assigned to UOP

Applicants have developed a catalyst for various hydrocarbon conversion processes which is tolerant to the presence of large amounts of sulfur (1000 ppm) in the feedstream. The catalyst consists of a MeAPSO support that has dispersed thereon palladium and a modifier, such as lanthanum or dysprosium.

**5720929****Device for catalytic dehydrogenation of a C2 + paraffinic charge comprising means for inhibiting the freezing of water in the effluent**

Minkinen Ar; Burzynski Jean-Pierre; Larue Joseph. Saint Nom La Breteche, FRANCE; assigned to Institut Francais Du Petrole

A device for the catalytic dehydrogenation of a C2 + paraffinic hydrocarbon charge is applicable to the synthesis of methyl *tert*-butyl ether. Effluent coming from the dehydrogenation reactor and containing olefins and water is cooled in at least one heat exchanger, saturated with water in a column and sent to a stripping column where it is at least partly put in contact with a recycled aqueous liquid phase containing a solvent, preferably methanol. The compressed gaseous effluent in which the water is thereby inhibited from freezing by the methanol is cooled in a heat exchanger then separated in separator into olefins and into hydrogen. An aqueous liquid phase with methanol is decanted at and recycled in column.

**NATURAL GAS AND SYNGAS CONVERSION****5705688****Process for the purification of acrylic acid obtained by catalytic oxidation of propylene**

Fauconet Michel; Esch Marc; Samuel Yves; Laurent Denis. Valmont, FRANCE; assigned to Elf Atochem SA

Acrylic acid obtained by catalytic oxidation of propylene, extracted by countercurrent washing of the reaction gases which at least one hydrophobic heavy solvent in an extraction column is purified. To this end, a distillation of the stream obtained at the bottom of the column is conducted in a column so as to obtain a very pure acrylic acid stream at the top of the column, allowing acrylic acid to pass at the bottom; the bottom stream is conveyed as feed into the lower part of a distillation column from which a stream rich in maleic anhydride and impurities of boiling temperatures situated between that of acrylic acid and that of the heavy solvent is drawn off sideways on a tray situated between the feed and the top of the column; a stream rich in acrylic acid, which is returned as feed to the column is distilled at the top of the column; and a stream

containing heavy solvent and heavy impurities, which is recycled to the top of the column, is recovered at the bottom of the column.

**5707917****Catalyst for the selective oxidation of hydrocarbons**

Geus John Wilhelm; Overbeek Rudolf Alfred. Bilthoven, NETHERLANDS; assigned to Engelhard De Meern BV

The invention relates to a heterogeneous vanadium–phosphorus oxide catalyst system for the selective oxidation of hydrocarbons, which may or may not be saturated, comprising a support based on one or more metal oxides, and vanadium–phosphorus oxide in an amount of from 0.01 to 45 wt.%, based on the weight of the catalyst and calculated as (VO)2P2O7, to a process for the selective oxidation of an organic compound in the presence of a vanadium–phosphorus oxide catalyst, which process comprises an oxidation and a reduction phase, wherein a hydrocarbon is contacted with said catalyst in the reduction phase and in oxidized or nonoxidized form is adsorbed to the catalyst, whereafter the thus loaded catalyst is brought into the oxidation phase, the desired product is formed in the presence of gaseous oxygen and subsequently separated.

**5707920****Isobutanol synthesis catalyst**

Vanderspurt Thomas Henry; Greaney Mark Ala; Leta Daniel Paul; Koveal Russell John; Disko Mark Michael; Klaus Angela V; Behal Sutinder K; Harris Robert B. Delaware Township, NJ, USA; assigned to Exxon Research and Engineering Company

The invention relates to a catalyst for conversion of methanol, ethanol alone or in combination with *n*-propanol to isobutanol. The catalyst is a noble metal supported on at least a first phase having poorly crystalline manganese- and zinc-doped zirconium oxide phase containing about 71 to about 91 at.% zirconium, about 10 to about 16 at.% manganese and about 4 to about 8 at.% zinc and a second phase of irregularly shaped hetaerolitelike crystals containing about 65 to about 69 at.% manganese, about 31 to about 35 at.% zinc and zero to about 5 at.% zirconium embedded in the first phase. The catalyst is useful in making isobutanol.

**5714644****Process and catalyst for the selective hydrogenation of butynediol to butenediol**

Irgang Matthias; Menger Volkmar; Miesen Ernest; Stops Peter; Graf Fritz. Heidelberg, GERMANY; assigned to BASF Aktiengesellschaft

PCT No. PCT/EP95/02592 Sec. 371 Date Mar. 20, 1997 Sec. 102(e) Date Mar. 20, 1997 PCT Filed Jul. 4, 1995 PCT Pub. No. WO96/01242 PCT Pub. Date Jan. 18, 1996. A process is disclosed for the selective hydrogenation of butynediol to 2-butene-1,4-diol using a palladium catalyst to which either copper and zinc, or silver and zinc, or copper, silver and zinc are added as doping agents. In the preferred embodiment, it has been possible, by optimizing the composition of the catalyst, to improve the activity and selectivity of the catalysts in question while also eliminating the need to handle toxic substances during the catalyst manufacturing process.

**5714651****Use of polymerization inhibitor to prolong the life of a Lewis acid catalyst**

Elsheikh Maher Yousef; Bolmer Michael Sheppard. Wayne, PA, USA; assigned to Elf Atochem North America Inc

A method for extending the life of catalyst used to fluorinate olefins, e.g., a supported Lewis acid catalyst in the catalytic hydrofluorination of vinylidene chloride by conducting the hydrofluorination in the presence of a polymerization inhibitor for the vinylidene chloride.

**5719299****Process for the catalytic vapor phase oxidation of ethylene**

Te Raa Arend Jan. Klundert, NETHERLANDS; assigned to Shell Oil Company

A process for the catalytic vapor-phase oxidation of ethylene with a molecular oxygen-containing gas, in a reactor comprising a multitude of reaction tubes containing a supported silver catalyst and surrounded by a heat-exchange fluid which enters the reactor in liquid form and leaves the reactor in vapor form, characterized in that between 5 and 100 wt.% of the liquid heat-exchange fluid is introduced to the reactor at its downstream end, at a temperature which is at least 20°C below the temperature of the heat-exchange fluid on leaving the reactor.

**5720901****Process for the catalytic partial oxidation of hydrocarbons**

De Jong Krijn Pieter; Schoonebeek Ronald Jan; Vonkeman Koert Alexander. Amsterdam, NETHERLANDS; assigned to Shell Oil Company

This invention concerns a process for the catalytic partial oxidation of a hydrocarbon feedstock. The process comprises contacting a feed mixture comprising the hydrocarbon feedstock and an oxygen-containing gas, which feed mixture also comprises nitrogen, with a catalyst capable of catalyzing the partial oxidation of the hydrocarbon feedstock, wherein the feed mixture also comprises a sulfur-containing compound. This process allows sulfur-containing hydrocarbon feedstocks to be processed without first undergoing a sulfur removal treatment. Accordingly, a process for the preparation of carbon monoxide and/or hydrogen from a hydrocarbon feedstock comprises subjecting the hydrocarbon feedstock in a first stage to a catalytic partial oxidation process as described above and subjecting at least a portion of the product of the first stage in a second stage to a desulfurization process.

**5705679****Honeycomb catalyst for vinyl acetate synthesis**

Nicolau Ioan; Colling Philip M; Johnson Leland R. Corpus Christi, TX, USA

A catalyst particularly useful in the preparation of unsaturated esters, such as the reaction of ethylene, oxygen and acetic acid in a vapor phase to form vinylacetate. The catalyst comprises a honeycomb carrier coated with silica and containing palladium and gold throughout the silica coat. Such catalysts show reduced pressure drop and a high space time yield for vinyl acetate.

**5705688****Process for the purification of acrylic acid obtained by catalytic oxidation of propylene**

Fauconet Michel; Esch Marc; Samuel Yves; Laurent Denis. Valmont, FRANCE; assigned to Elf Atochem SA

Acrylic acid obtained by catalytic oxidation of propylene, extracted by countercurrent washing of the reaction gases which at least one hydrophobic heavy solvent in an extraction column is purified. To this end, a

distillation of the stream obtained at the bottom of the column is conducted in a column so as to obtain a very pure acrylic acid stream at the top of the column, allowing acrylic acid to pass at the bottom; the bottom stream is conveyed as feed into the lower part of a distillation column from which a stream rich in maleic anhydride and impurities of boiling temperatures situated between that of acrylic acid and that of the heavy solvent is drawn off sideways on a tray situated between the feed and the top of the column; a stream rich in acrylic acid, which is returned as feed to the column is distilled at the top of the column; and a stream containing heavy solvent and heavy impurities, which is recycled to the top of the column, is recovered at the bottom of the column.

#### 5712413

##### **Process for the preparation of *n*-hydrocarbyl-substituted amides, such as *tert*-butylacrylamide via the Ritter reaction using solid heteropolyacid catalysts**

Burrington James D; Rhubright Douglas C; Ramey Chester. Mayfield Village, OH, USA; assigned to The Lubrizol Corporation

Hydrocarbyl-substituted amides are prepared by a process comprising contacting a nitrile with a hydrocarbylating agent, such as an alkylating agent, in the presence of a catalyst comprising a heteropolyacid or salt thereof.

#### 5714610

##### **Process for the preparation of 3-methylpiperidine and 3-methylpyridine by catalytic cyclization of 2-methyl-1, 5-diaminopentane**

Heveling Josef; Armbruster Eric; Siegrist Walter. Naters, SWITZERLAND; assigned to Lonza Ltd

PCT No. PCT/EP94/01005 Sec. 371 Date Nov. 2, 1995 Sec. 102(e) Date Nov. 2, 1995 PCT Filed Mar. 30, 1994 PCT Pub. No. WO94/22824 PCT Pub. Date Oct. 13, 1994. In a process for preparing 3-methylpiperidine or 3-methylpyridine from 2-methyl-1,5-diaminopentane in the gaseous phase, the initial product is made to flow over catalysts. In the first step, 3-methylpiperidine is produced, and if required 3-methylpyridine is produced in a second step.

#### 5714624

##### **Oxotitanium complex, asymmetric hydrogenation catalyst comprising the complex, and process for producing beta-hydroxy ketone or alpha-hydroxy carboxylic acid ester using the complex**

Nakai Takeshi; Kitamoto Dai; Sayo Noboru. Kanagawa, JAPAN; assigned to Takasago International Corporation

A novel oxotitanium complex represented by general formula (I) is disclosed: (\*See Patent for Chemical Structure\*) (I) wherein R1 and R2 may be the same or different and each represents a hydrogen atom, a lower alkyl group, a lower alkoxy group, a halogen atom, a phenyl group, a substituted phenyl group, a trialkylsilyl group, a monoalkyldiphenylsilyl group, a dialkylmonophenylsilyl group, a triphenylsilyl group, a substituted triphenylsilyl group, or a lower alkoxy carbonyl group, provided that R1 and R2 may be bonded to each other to form a hydrocarbon ring or a substituted hydrocarbon ring in cooperation with the carbon atoms to which R1 and R2 are bonded; R3 and R4 may be the same or different and each represents a hydrogen atom, a lower alkyl group, a lower alkoxy group, a benzoyl group, a benzenesulfonyl group, or a halogen atom, provided that R3 and R4 may be bonded to each other to form a hydrocarbon ring or a substituted hydrocarbon ring in cooperation with the carbon atoms to which R3 and R4 are bonded; and *n* is 1 or 2. The novel oxotitanium complex is useful as an asymmetric reaction catalyst. A process for producing a beta-hydroxy ketone or an alpha-hydroxy carboxylic acid ester in the presence of the novel oxotitanium complex is also disclosed.

#### ORGANOMETALLIC CATALYSIS

#### 5703030

##### **Bleach compositions comprising cobalt catalysts**

Perkins Christopher Mark; Scheper William Michael. Cincinnati, OH, USA; assigned to The Procter & Gamble Company

Bleach compositions comprising certain carboxylate-containing cobalt catalysts are provided. More specifically, the invention relates to automatic dishwashing detergents and laundry compositions which provide en-

hanced cleaning/bleaching benefits (especially tea stain removal) through the selection of cobalt catalysts having the formula: (\*See Patent for Tabular Presentation\*) PS wherein cobalt is in the +3 oxidation state; and M is a substituted and unsubstituted C1–C30 carboxylate-containing ligand having the formula RC(O)O – .

#### 5705464

##### **Automatic dishwashing compositions comprising cobalt catalysts**

Scheper William Michael; Perkins Christopher Mark. Lawrenceburg, IN, USA; assigned to The Procter & Gamble Company

Automatic dishwashing detergent compositions comprising certain cobalt catalysts are provided. More specifically, the invention relates to automatic dishwashing detergents which provide enhanced cleaning/bleaching benefits (especially tea stain removal) through the selection of cobalt bleach catalyst having the formula: (\*See Patent for Tabular Presentation\*) PS wherein cobalt is in the +3 oxidation state;  $n$  is 4 or 5 (preferably 5); M is one or more ligands coordinated to the cobalt by one site;  $m$  is 0, 1 or 2 (preferably 1); B is a ligand coordinated to the cobalt by two sites;  $b$  is 0 or 1 (preferably 0), and when  $b = 0$ , then  $m + n = 6$ , and when  $b = 1$ , then  $m = 0$  and  $n = 4$ ; and T is one or more appropriately selected counteranions present in a number  $y$ , where  $y$  is an integer to obtain a charge-balanced salt (preferably  $y$  is 1 to 3; most preferably 2 when T is a  $-1$  charged anion); and wherein further said catalyst has a base hydrolysis rate constant of less than  $0.23 \text{ M}^{-1} \text{ s}^{-1}$  ( $25^\circ\text{C}$ ). Preferred automatic dishwashing compositions comprise amylase and/or protease enzymes. Included are methods for washing tableware in domestic automatic dishwashing appliances using the cobalt catalysts.

#### 5710339

##### **Bisphosphines as catalysts for asymmetric reactions**

Laue Christia; Schruml oder Georg; Arlt Dieter. Monheim, GERMANY; assigned to Bayer Aktiengesellschaft

The invention relates to enantiomerically pure bisphosphines of the general formula (I) (\*See Patent for Chemical Structure\*) (I) where R is as defined in the

description, a process for their preparation and their use in metal complexes as catalysts for asymmetric reactions, in particular asymmetric hydrogenations.

#### 5710342

##### **Process for preparing carbonyl compounds by catalysed oxidation of olefins and catalysts present microemulsions**

Imre Laszlo; Schomuml acker Reinhard; Daun Judi. Leverkusen, GERMANY; assigned to Bayer Aktiengesellschaft

Carbonyl compounds can be prepared by catalytic oxidation of olefins with oxygen or oxygen-containing gases in a microemulsion (ME) as reaction medium and catalyst support. The catalysts present in ME and used for this purpose are characterized by a content of 0.0001–10% by weight of a Pd compound and by a content of 0.0005–20% by weight of a subgroup metal compound or a quinone, based on the total weight of the catalysts present in the ME.

## POLYMERISATION CATALYSIS

#### 5710222

##### **Method for controlling the melting points and molecular weights of syndiotactic polyolefins using metallocene catalyst systems**

Ewen John A; Reddy B R; Elder Michael. Houston, TX, USA; assigned to Fina Technology Inc

This invention is for a method for varying the melting points and molecular weights of syndiotactic polyolefins by using metallocene catalysts with substituents on the aryl rings of a fluorene ligand in certain positions. Electron donating substituents, for example, dialkylamino, halogen, and alkoxy groups, when present on fluorene at C1, C2, C3 or C2 and C7 render the catalyst less active and decrease the polymer molecular weight. Electron donating substituents, such as alkyl, dialkylamino, halogen or alkoxy, at C4 not only increased the catalyst efficiency but also dramatically increased the polymer molecular weight. Bulky hydrocarbyl substituents, such as alkyl, cyaloalkyl and aryl groups, at C2 and C7 increase the melting point. Bulky substituents, such as hydrocarbyl groups like alkyl, cycloalkyl, aryl, alkoxy and dialkylamino groups etc., at C4/C5 decrease the melting point. When multiple



substituents are present on fluorene, their effects are additive.

#### 5710223

##### **Process for polymerizing propylene by using a novel metallocene catalyst**

Fukuoka Daisuk; Tashiro Takash; Kawaai Koji; Saito Junji; Ueda Takashi; Kiso Yoshihisa; Imuta Junichi; Fujita Terunor; Nitabaru Masatoshi; Yoshida Masayasu. Kuga gun, JAPAN; assigned to Mitsui Petrochemical Industries Ltd

A process for polymerizing propylene utilizes a metallocene catalyst component of the formula (I) (\*See Patent for Chemical Structure\*) (I) wherein M is a transition metal, R1 is a C2–C6 hydrocarbon, R2 is a C6–16 aryl group, X1 and X2 are each a halogen atom and Y is a divalent hydrocarbon or a divalent silicon-containing group. Propylene polymers produced thereby have such properties that the triad tacticity of propylene units is high, the proportion of inversely inserted units based on the 2,1-insertion of propylene monomer is in a specific range and the intrinsic viscosity ( $\eta$ ), as measured in decahydronaphthalene at 135°C, is in a specific range.

#### 5710225

##### **Heteropolyacid catalyzed polymerization of olefins**

Johnson John; Burrington James D. Euclid, OH, USA; assigned to The Lubrizol Corporation

Phosphotungstic acid salts are used to polymerize C2–C30 alpha-olefins and derivatives thereof to form highly reactive products having a high content of terminal vinylidene groups and beta-isomers in resonance therewith. Isobutylene has been polymerized to form a Mn 300–20 000 polymer.

#### 5710297

##### **Metallocenes, and their use as catalysts**

Weller Thomas; Aulbach Michael; Kuml uber Frank; Erker Gerhard; Psiorz Christian; Bachmann Bernd; Osan Frank. Mainz, GERMANY; assigned to Hoechst Aktiengesellschaft

The present invention relates to a stereorigid metallocene compound containing, as ligands, at least two

substituted or unsubstituted cyclopentadienyl groups which are bonded to one another via a monocyclic or polycyclic ring system, where at least one cyclopentadienyl group is fused to the monocyclic or polycyclic ring system, and to a process for the preparation of a cycloolefin copolymer. The cycloolefin copolymers obtained in this way have high tear strengths and are suitable for the production of extrusion parts and injection moldings.

#### 5712404

##### **Cyclopentadienyl-type ligands, metallocene compounds catalyst systems, preparation and use**

Peifer Bernd; Alt Helmut G; Welch M Bruce. Bayreuth, GERMANY; assigned to Phillips Petroleum Company

In accordance with the present invention there is provided a process for preparing a cyclopentadienyl-type ligand comprising reacting an alkali metal compound and a fulvene-type compound to form a dianion, and then reacting the dianion with water. In accordance with another embodiment of the present invention a process for preparing a metallocene compound is provided comprising reacting an alkali metal compound, a fulvene-type compound, and a transition metal-containing compound. In another embodiment, the metallocene compound and a cocatalyst are combined to form a catalyst system. Other aspects of the invention include the cyclopentadienyl-type ligands, metallocene compounds, and catalyst systems thus produced and polymerization processes employing the catalyst systems.

#### 5714424

##### **Multicomponent polyolefin catalysts**

Warthen John L; Glemza Rimantas. Baltimore, MD, USA; assigned to WR Grace & Co-Conn

The present invention encompasses a monolithic multicomponent composite polyolefin catalyst particle comprising at least two distinct supported catalyst components wherein each catalyst component is capable of producing a resin having a melt index different from at least one other catalyst component under constant reaction conditions. Methods of producing and using the multicomponent catalyst are disclosed.

**5714425****Catalyst compositions and process for preparing polyolefins**

Chabrand Christine Jacqueline; Little Ian Raymond; McNally John Paul. Martigues, FRANCE; assigned to BP Chemicals Limited

Catalyst compositions comprising metallocene complexes having polymerisable olefinic groups substituent on an organic group containing a cyclopentadienyl nucleus may be used for the preparation of polyolefins. The catalyst compositions may be in the form of polymers comprising the metallocene complex and may be suitably supported on inorganic supports. Polymers having a broad range of density and melt indices as well as low hexane extractables and excellent powder morphology and flowability may be obtained by use of the catalyst compositions. Preferred metallocene complexes are zirconium complexes in which the polymerisable olefinic group is vinyl.

**5714426****Olefin polymerization catalyst**

Tsutsui Toshiyuki; Yoshitsugu Ken; Toyota Akinori. Kuga gun, JAPAN; assigned to Mitsui Petrochemical Industries Ltd

In accordance with the present invention, there are provided ethylene copolymers composed of structural units (a) derived from ethylene and structural units (b) derived from alpha-olefin of 3–20 carbon atoms, said ethylene copolymers having (A) a density of 0.85–0.92 g/cm<sup>3</sup>, (B) an intrinsic viscosity ( $\eta$ ) as measured in decalin at 135°C of 0.1–10 dl/g, (C) a ratio (Mw/Mn) of a weight average molecular weight (Mw) to a number average molecular weight (Mn) as measured by GPC of 1.2–4, and (D) a ratio (MFR10/MFR2) of MFR10 under a load of 10 kg to MFR2 under a load of 2.16 kg at 190°C of 8–50, and being narrow in molecular weight distribution and excellent in flowability.

**5714427****Catalyst system comprising two zirconocenes and aluminoxane**

Winter Andreas; Dolle Volker; Spaleck Walter. Glashutten, GERMANY; assigned to Hoechst Aktiengesellschaft

A catalyst system comprising an aluminoxane and a transition-metal component (metallocene) is used, the

transition-metal component comprising at least one zirconocene of the formula I (\* See Patent for Chemical Structure\*) (I) R1R2ZrR14(CR8R9)mR7R15(CR8R9)n (Ia) and at least one zirconocene of the formula Ia or alternatively at least 2 zirconocenes of the formula I.

**5714428****Double metal cyanide catalysts containing functionalized polymers**

Le-Khac Bi. West Chester, PA, USA; assigned to Arco Chemical Technology LP

Double metal cyanide (DMC) catalysts useful for epoxide polymerizations are disclosed. The catalysts comprise a DMC compound, an organic complexing agent, and from about 2 to about 80 wt.% of a functionalized polymer or a water-soluble salt derived from the polymer. The catalysts are easy to prepare and isolate, are substantially noncrystalline, and have high activity for polymerizing epoxides. Polyols made from the catalysts have exceptionally low unsaturations, low viscosities, and reduced levels of high molecular weight polyol tail.

**5714555****Catalyst compositions and process for preparing polyolefins**

Chabrand Christine Jacqueline; Little Ian Raymond; McNally John Paul. Martigues, FRANCE; assigned to BP Chemicals Limited

Catalyst compositions comprising metallocene complexes having polymerisable may be used for the preparation of polyolefins. The catalyst compositions may be in the form of polymers comprising the metallocene complex and may be suitably supported on inorganic supports. Polymers having a broad range of density and melt indices as well as low hexane extractables and excellent powder morphology and flowability may be obtained by use of the catalyst compositions. Preferred metallocene complexes are zirconium complexes in which the polymerisable group is vinyl.

**5714568****Methods of preparing polyesters from cyclic organic carbonates in the presence alkali metal-containing catalysts**

Nava Hildeberto. Cary, NC, USA; assigned to Reichhold Chemicals Inc

A process for preparing an esterification product is disclosed. The process comprises providing a polyfunctional organic acid or anhydride, a cyclic organic carbonate compound, and a catalyst selected from the group consisting of an alkali metal, an alcohol-derived salt of the alkali metal, and mixtures thereof, and reacting the polyfunctional organic acid or anhydride and the cyclic organic carbonate compound in the presence of the catalyst to form the esterification product.

**5714570****Method for the preparation of polyester by use of composite catalyst**

Kim Chung Yup; Cho Hyun Nam; Yoo Hoi Wan; Kim Hyung Joon. Seoul, South Korea; assigned to Korea Institute of Science and Technology

A method for preparing polyester by use of a catalyst. The method comprises the steps of: esterifying either a terephthalic acid, a dicarboxylic acid containing terephthalic acid or derivatives thereof with either ethylene glycol or a glycol containing ethylene glycol, or derivatives thereof to produce an esterification product containing either bis(beta-hydroxyethyl) terephthalate, its low molecular weight polymer, or a combination of beta-hydroxyethyl terephthalate and its low molecular weight polymer; and continuously polycondensing the obtained esterification product to prepare a polyester, wherein in the said polycondensing step, a composite polymerization catalyst dissolved in an ethylene glycol or an ethylene glycol-containing solution, consisting of a compound of antimony, a compound of titanium, and a compound of tin is used. The method can considerably reduce both the esterification time and the polycondensation time and provides a good color, reduced content of diethylene glycol and reduced concentration of terminal carboxyl groups in the prepared polyesters.

**5716898****Process for the production of a microspheroidal powder of low dispersity, microspheroidal powder of low dispersity thus obtained, catalysts for the polymerization of alpha-olefins and process for the polymerization of alpha-olefins**

Derleth Helmut; Grosjean Abel. Nienburg, GERMANY; assigned to Solvay (Societe Anonyme)

Process for the production of a microspheroidal powder of low dispersity, according to which a hydrogel is prepared, an aqueous suspension of the ground hydrogel is subjected to atomisation in a stream of gas, gelled particles containing moisture are collected from the atomisation, the particles are brought into contact with an organic liquid in order to remove at least some of the moisture and the particles are then subjected to drying. The process applies to the production of silica powders intended to serve as support for chromium oxide catalysts which may contain titanium oxide and are suitable for the polymerisation of ethylene.

**5714639****Double metal cyanide catalysts and methods for making them**

Bowman Paul T; Hinney Harry R; Meeker Robert L. Hurricane, WV, USA; assigned to Arco Chemical Technology LP

Improved double metal cyanide (DMC) catalysts useful for making epoxide polymers are disclosed. One catalyst is a paste of a double metal cyanide (DMC) compound, an organic complexing agent, and water. The paste comprises at least about 90 wt.% of particles having a particle size within the range of about 0.1 to about 10  $\mu\text{m}$  as measured by light scattering in polyether polyol dispersions of the catalyst particles. Preferred catalysts have a bimodal particle size distribution. The paste catalyst is more active and simpler to prepare than powder DMC catalysts. Polyols made from the paste catalyst have low unsaturations, low viscosities, and narrow molecular weight distributions, and are useful in making a variety of polyurethane products.

**5719235****Catalyst system, use of this catalyst system for the (co)polymerization of olefins, process for preparing this catalyst system and olefin (co)polymerization process**

Zandona Nicola. Waterloo, BELGIUM; assigned to Solvay (Societe Anonyme)

Catalyst system comprising an ionic metallocene derived from a transition metal, a catalytic solid containing an element of group IVB, magnesium and a halo-

gen, and an aluminium derivative. Process for preparing this catalyst system, according to which a mixture of a neutral metallocene and of the catalytic solid is prepared, the mixture thus obtained is placed in contact with an aluminium derivative and an ionizing agent is added to it. Process for (co)polymerization of an olefin, according to which a mixture of the neutral metallocene and of the catalytic solid is prepared, the mixture thus obtained is placed in contact with an aluminium derivative and the olefin, and the ionizing agent is added to it.

#### 5719241

##### **Process for producing polyolefins and polyolefin catalyst**

Razavi Abbas; Debras Guy L. Mons, BELGIUM; assigned to Fina Research SA

The present invention provides a process for preparing polyolefins having a multimodal or at least bimodal molecular weight distribution by contacting in a reaction mixture under polymerization conditions at least one olefin and a catalyst system comprising a supported catalyst component comprising an alumoxane and at least two metallocenes containing the same transition metal and selected from mono-, di-, and tri-cyclopentadienyls and substituted cyclopentadienyls of a transition metal and wherein at least one of the metallocenes is bridged and at least one of the metallocenes is unbridged.

#### 5721185

##### **Homogeneous olefin polymerization catalyst by abstraction with Lewis acids**

LaPointe Robert E; Stevens James; Nickias Peter; McAdon Mark H. Midland, MI, USA; assigned to The Dow Chemical Company

A process for preparing addition polymerization catalysts having a limiting charge separated structure corresponding to the formula: (\*See Patent for Tabular Presentation\*) PS wherein: L is a derivative of a substituted delocalized pi-bonded group imparting a constrained geometry to the metal active site and containing up to 50 nonhydrogen atoms; M is a metal of Group 4 or the Lanthanide series of the Periodic Table of the Elements; X independently each occurrence is hydride or a hydrocarbyl, silyl or germyl group having

up to 20 carbon, silicon or germanium atoms; and A – is an anion of a Lewis acid, A, having relative Lewis acidity greater than or equal to that of phenylbis(perfluorophenyl)borane, said anion being compatible with the metal cation, the steps of the process comprising contacting a derivative of a Group 4 or Lanthanide metal corresponding to the formula: (\*See Patent for Tabular Presentation\*) PS wherein L, M, and X are as previously defined, with the Lewis acid, A, under conditions to cause abstraction of X and formation of the anion  $XA -$ .

## ENVIRONMENTAL CATALYSIS

#### 5712213

##### **Rejuvenation process for a solid alkylation catalyst**

Joly Jean-Francois; Benazzi Eric; Marcilly Christian; Euzen Jean-Paul; Forestiere Alain. Lyons, FRANCE; assigned to Institut Francaise Du Petrole

The invention concerns a rejuvenation process for a solid catalyst for the alkylation of at least one isoparaffin by at least one olefin. The process is characterised in that the catalyst is rejuvenated in the reaction zone by circulating an isoparaffin-rich liquid fraction over the bed, said fraction originating from a zone for separating the reaction effluent extracted from the reaction zone.

#### 5712214

##### **Regeneration of aromatization catalysts**

Huang Yao-Jyh Robert; Fung Shun Chong. Taipei, TAIWAN; assigned to Exxon Research & Engineering Company

A low-pressure, high-temperature, wet posttreatment after oxyhalogenation during regeneration to improve activity and selectivity recovery of a regenerated catalyst involves exposing a halogenated catalyst to a gaseous stream including water having a partial pressure of up to about 1.5 psia, oxygen having a partial pressure of less than about 4.5 psia, and an inert gas at a temperature within the range of about 450°C to about 530°C at a pressure within the range of about 14.7 psia to about 65 psia for a time sufficient to effect a hydrogen halide partial pressure in offgas from the halogenated catalyst of less than about 0.004 psia to

effectively remove excess halide from the halogenated catalyst.

**5716590****Catalytic hydrodehalogenation of halogen-containing compounds of group iv elements**

Roewer Gerhard; Paetzold Uwe; Walter Holger. Freiberg, GERMANY; assigned to Wacker-Chemie GmbH

Catalytic dehalogenation (or hydrodehalogenation) of halogen-containing compounds of elements of group IV of the periodic table in the presence of hydrogen is carried out using a finely-dispersed catalytically active material which comprises silicon and at least one transition metal, and which is characterized by high catalytic activity and stability. This process can be used, for example, for synthesizing compounds or alternatively for decomposing halogen-containing compounds, for instance in waste-water or waste-gas purification processes. It is also suitable for dehalogenation (hydrodehalogenation) of halogen-containing silane compounds, for instance of silicon tetrachloride or alkyl trichlorosi-

lane compounds, and the original purity of the silanes used as starting materials is retained by the products. Tetrachlorosilane, for example, can be converted into trichlorosilane.

**5719097****Catalyst comprising a modified solid oxide**

Chang Clarence D; Santiesteban Jose G; Stern David L. Princeton, NJ, USA

There is provided a catalyst comprising a hydrogenation/dehydrogenation component, such as a noble metal, and an acidic solid component comprising a Group IVB metal oxide modified with an oxyanion of a Group VIB metal. An example of this catalyst is zirconia, modified with tungstate and platinum. There is also provided a method for preparing this catalyst. This catalyst may be used, for example, to isomerize C4 to C8 paraffins. The feed to this paraffin isomerization reaction may, optionally, include cyclic hydrocarbons, such as benzene or cyclohexane, which may undergo ring opening reactions during the course of the isomerization reaction