

Patents

CATALYSIS – APPLIED AND PHYSICAL ASPECTS

Carbon Supported Palladium Catalyst

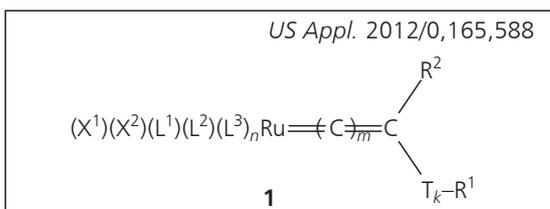
Université Catholique de Louvain, *European Appl.* 2,468,701; 2012

The Suzuki-Miyaura C–C coupling reaction here consists of an aryl halide reacting with an arylboronic acid in an organic solvent in the presence of a Pd/C catalyst (the specific surface area of the C support is $\sim 100 \text{ m}^2 \text{ g}^{-1}$) and an inorganic base selected from K_2CO_3 , NaOH, KOH, Na_2CO_3 or NaHCO_3 . The reaction is carried out at constant pH, preferably pH ~ 10.6 .

Ruthenium-Carbene Complexes

BASF SE, *US Appl.* 2012/0,165,588

A Ru-carbene complex, **1**, supported on SiO_2 is used in olefin metathesis for the preparation of cycloalkadienes from cycloalkene monomers, polycycloalkenylenes or a combination of these in a continuously operated reactor. X^1 and X^2 can either be identical or different and are each an anionic ligand; L^1 , L^2 and L^3 are each an uncharged electron donor ligand; T is O, S, NR or PR; R is H or a C_1 – C_{40} radical; R^1 and R^2 are H, a C_1 – C_{40} radical or two or more ligands or radicals selected from X^1 , X^2 , L^1 , L^2 and L^3 and may be joined to one another to form a cyclic or polycyclic ring system; k is 0 or 1; n is 0 or 1 and m is 0, 1 or 2.



CATALYSIS – INDUSTRIAL PROCESS

Manufacture of Vinyl Chloride

Solvay SA, *World Appl.* 2012/084,643

Vinyl chloride is manufactured by reacting C_2H_2 with HCl, using a catalytic system comprising of an *N*-alkylated imidazole selected from 1-methylimidazole, 1-ethylimidazole, 1-butylimidazole, 1-hexylimidazole, 1-octylimidazole, 1-decylimidazole, 1-methyl-2-octylimidazole, 1-ethyl-2-methylimidazole, 1-butyl-2-methylimidazole, 1-hexyl-2-methylimidazole

and 1-decyl-2-methylimidazole and a compound of a metal chosen from Pt, Pd, Os, Ru, Au and Hg, preferably Pd, for example, PdCl_2 . The reaction is carried out between room temperature and 220°C .

CATALYSIS – REACTIONS

Saccharification of Biomass Materials

Tanaka Noble Metal Industrial Co, Ltd, *Japanese Appl.* 2012-110,873

The saccharification of biomass materials is catalysed by Ru or Pd catalyst supported by cellulose carbonisation products containing 2.4–7.0 wt% SO_3H groups. The cellulose carbonisation products have a degree of crystallinity of 1.8–2.4. The catalyst is manufactured by carbonisation of cellulose powder at 400 – 900°C for 1–10 h, immersion of the resulting supports in a solution containing Ru and/or Pd, then heating in concentrated H_2SO_4 or fuming H_2SO_4 for the binding of the SO_3H groups to the supports. Cellulose was saccharified using a Ru catalyst supported on carbonised cellulose containing SO_3H groups at 230°C for 1 min to give glucose in 40% yield and 68% cellulose conversion.

Hydrogenation of Vegetable Oil

Russian Federation represented by the Ministry of Industry and Trade of the Russian Federation, *Russian Patent* 2,452,563; 2012

A Pd-containing catalyst is used in the hydrogenation of vegetable oils resulting in a reduced content of *trans*-isomers. The catalyst consists of crystallites of catalytically active Pd applied over a C carrier surface with a granule size of 1.0–5.0 nm, specific surface area of 100 – $450 \text{ m}^2 \text{ g}^{-1}$ and a mean pore size of $\geq 4 \text{ nm}$.

EMISSIONS CONTROL

Lean-Burn Engine Oxidation Catalyst

Johnson Matthey Plc, *World Appl.* 2012/085,572

An exhaust system consists of a first oxidation catalyst containing Pt or Pt-Pd supported on a reducible oxide selected from Mn, Fe, Sn, Cu, Co, Ce or optionally a stabilised homologue of one of these disposed on a first honeycomb monolith substrate. The loading of the pgm on the monolith substrate is $>10 \text{ g ft}^{-3}$. The first oxidation catalyst may comprise of a molecular sieve which consists of Cu and/or Fe and at least one precious metal.

FUEL CELLS

Oxygen Evolution Catalyst

Johnson Matthey Plc, *World Appl.* 2012/080,726

A catalyst layer for a fuel cell consists of an electrocatalyst and a crystalline metal oxide oxygen evolution catalyst. The oxygen evolution catalyst comprises of: (a) one or more metals selected from the lanthanides, Y, Mg, Ca, Sr, Ba, Na, K, In, Tl, Sn, Pb, Sb and Bi; (b) one or more metals selected from Ru, Ir, Os and Rh; and (c) O. The atomic ratio of the first metal(s) to the second metal(s) is from 1:1.5 to 1.5:1 and the atomic ratio of (first metal(s) + second metal(s)):oxygen is from 1:1 to 1:2. The formula of the oxygen evolution catalyst is $(AA')_a(BB')_bO_c$ where A and A' are selected from group (a); B is selected from group (b); B' is selected from (b), or from Ca, Mg or rare earth metals; $a = 0.66-4.5$; $b = 1-3$; and $c = 3-11$.

Palladium/Carbon Oxidation Electrode

Hitachi Ltd, *European Appl.* 2,469,632; 2012

The MEA for a fuel cell comprises an anode containing a catalyst selected from Pt, Ru, Ir, Rh, Os, W, Mo, Fe, Co, Ni or Mn, a solid PEM and a cathode. An additional HCOOH oxidation electrode containing Pd/C and a solid polymer electrolyte is formed between the anode GDL and the anode and between the cathode GDL and the cathode.

Platinum Alloy Catalyst with Conductive Polymer Coating

Hyundai Motor Co, *US Appl.* 2012/0,135,137

A Pt catalyst on a C support selected from C black, C nanotubes, C nanofibres, C nanocoils and C nanocages is prepared by coating the surface of the Pt catalyst with either polypyrrole or polyaniline. A transition metal salt selected from nitrates, sulfates, acetates, chlorides or oxides of Pd, Ru, Co, Fe, Ni, Ti, V or Cr is supported on the coated catalyst and this is heat treated at 700–1000°C.

METALLURGY AND MATERIALS

Palladium-Based Metallic Glasses

California Institute of Technology, *US Appl.* 2012/0,168,037

A metallic glass free from Ni and Cu consists of a biocompatible alloy represented by the formula: $Pd_aX_bSi_cP_dGe_e$ where X is Ag, Au or a combination of both; a (in at%) = ~74–86; $b = \sim 2-5$; $c = \sim 8-10$; $d = 4-8$; and $e = \sim 0-3$. This may be used to form a 3D object and be used in biomedical components such as dental or

orthodontic components, implant or fixation devices or in jewellery applications.

APPARATUS AND TECHNIQUE

Analyte Sensor

Abbott Diabetes Care Inc, *US Appl.* 2012/0,138,484

An analyte sensor consists of a counter electrode and a working electrode with a sensing layer disposed on it. This layer comprises an analyte-responsive enzyme, a redox mediator containing an Os-containing complex or a Ru-containing complex having at least one pyridine boronic acid ligand and a thickener comprising urea, a urea derivative, urethane, a urethane derivative, a polyvinyl pyrrolidone polymer or a polyvinyl pyrrolidone polymer derivative. It is claimed that the analyte sensor has $\geq 90\%$ of its initial sensitivity after ≥ 14 days.

ELECTRICAL AND ELECTRONICS

Integrated Circuit Device

R. N. Akolkar and J. S. Clarke, *US Appl.* 2012/0,161,320

An integrated circuit device consists of a substrate with a layer of dielectric material on its surface. The dielectric material has a depression where one side is in contact with a metal layer selected from Ru, Pt, Ir, Pd, Re and Rh, preferably Ru, plus Co. A Cu seed layer is electrodeposited into the depression with a Co dopant. The metal layer is 1–4 nm thick.

Ruthenium Sputtering Target

General Research Institute for Nonferrous Metals, *Chinese Appl.* 102,485,378; 2012

A Ru sputtering target is heated at a rate of $50^\circ\text{C min}^{-1}$ from room temperature to 1200°C , it is then heated at a rate of $25^\circ\text{C min}^{-1}$ to $1400-1700^\circ\text{C}$ and hot pressed at 35 MPa for 10–60 min. The resulting Ru sputtering target has a relative density $>98\%$, the average grain size is $<20 \mu\text{m}$ and the O content is $<200 \text{ ppm}$.

NANOTECHNOLOGY

Novel Platinum Nanoparticles

Tata Chemicals Ltd, *World Appl.* 2012/095,863

A modified Pt NP has a mercapto alkyl acid and optionally an alkyl thiol attached to it and is supported on a substrate selected from rice husk ash, mesoporous silica, microporous silica, nanosilica and alumina. This is prepared by reacting a solution comprising of a Pt

precursor and a mercapto alkyl acid with a reducing agent. This second solution is heated to 50–100°C. The molar ratio of the mercapto alkyl acid and the alkyl thiol varies in the range of 0.25–100. The size of Pt NP varies in the range of 1–1000 nm and the Pt content is 5–98 wt%.

SURFACE COATINGS

Manufacturing Osmium Membrane

Beijing Institute of Satellite Environmental Engineering, *Chinese Appl.* 102,453,932; 2012

The process for manufacturing Os membrane comprises: (a) cleaning the quartz glass substrate, drying, preheating for 5–10 min under vacuum; (b) depositing a Cr layer and a Au layer both having a thickness of 5–20 nm by a PVD method; (c) cleaning to remove the residual reagent; (d) vacuum drying to obtain the Cr/Au coated quartz glass; (e) electroplating the Cr/Au coated quartz glass as cathode and Pt gauze electrode anode for 10 min whilst controlling the current density of 2.5–3.5 A dm⁻³, changing a new electroplating solution and re-electroplating for >10 min whilst controlling the current density of 2.5–3.5 A dm⁻³ to form an Os layer of thickness >1 μm. The Os electroplating solution is made after removing pinholes and reducing electrode surface tension by adding a surfactant to distilled water. This is then boiled, cooled and diluted to obtain 0.01 g l⁻¹ surfactant solution. The complexing agents NH₂SO₃H (25 g l⁻¹) and NH₂SO₃NH₄ (10 g l⁻¹) are added to completely dissolve (NH₄)₂OsCl₆ (10 g l⁻¹). This method can avoid the cracking phenomenon during Os electroplating and increase the thickness of the Os layer. The membranes can be used for preparing space atomic oxygen environment detectors.