

NEW PATENTS

ELECTROCHEMISTRY

Electrolytic Purification of NO_3^- -Containing Water

IONEX LTD. *British Appl.* 2,348,209
The electrolytic removal of NO_3^- ions (1) from H_2O involves electrolysis with current passed between a Rh coated cathode and an anode. H_2O can also be passed through an ion exchange column containing nitrate-selective anion resin to exchange with HCO_3^- (2) and/or Cl^- ions. In the eluted solution, (1) are converted by electrolysis to N_2 gas and the solution is replenished with (2) and/or Cl^- .

Cermet Composite for Inert Electrodes

ALCOA INC. *U.S. Patent* 6,126,799
A cermet composite (1) for inert electrodes is prepared by treating a mixture comprising a compound of Fe and a compound of Ni, Sn, Zn, Y, Cr and/or Ta; and an alloy or mixture containing 70–99.8 wt.% Cu and 0.2–30 wt.% Pt, Pd, Rh, Ir, Ag or Au at elevated temperature in an atmosphere containing O_2 . (1) is used as anodes in molten salt baths for production of metals by electrolytic reduction.

ELECTRODEPOSITION AND SURFACE COATINGS

Platinum Electroforming or Electroplating Bath

HONG KONG PRODUCT. COUNC. *British Appl.* 2,351,089
A Pt electroforming or electroplating bath comprises: 15–100 g l^{-1} Pt (Pt(II) and Pt(IV)) as haloplatinics or haloplatinics of alkali metals; and 50–400 g l^{-1} of an acid of sufficient strength to maintain $\text{pH} \leq 1$ in the bath. The bath is used to produce hollow Pt jewellery and for medical, electronic and aviation uses.

Anodes Used in Steel Strip Electroplating

I.U. FILTER CORP. *World Appl.* 00/60,141
An anode (1) comprises a valve metal substrate coated with three subsequent layers containing a Pt group metal or its oxide. (1) has an improved service life at low pH and/or high temperature and/or high current density. (1) is used in electrolyte processes for the preparation of Cl_2 , Br_2 and H_2O_2 ; the electro-deposition of Cr, Cu and Zn; and for high speed electroplating such as electroplating of steel strip.

Rhodium Electroplating

LUCENT TECHNOLOGIES INC. *World Appl.* 00/68,149
Rh is reacted with H_2SO_4 to produce a first Rh sulfate solution which is cooled to $< 20^\circ\text{C}$. This solution is neutralised by adding a base to precipitate Rh hydroxide while the Rh solution is at $< 25^\circ\text{C}$. Rh hydroxide is further combined with H_2SO_4 to produce a second Rh sulfate solution. The electrolyte has $\text{pH} < 1$, the Rh concentration is 1–10 g l^{-1} and the Rh sulfate complex does not contain any Rh-Rh bonds. Electroplated layers have low stress and full brightness over a thickness of 0.1–60 microinches.

APPARATUS AND TECHNIQUE

pH Electrode

UNIV. SOUTH AUSTRALIA *World Appl.* 00/67,010
A pH electrode (1) with a pH-sensitive region on an electrically conductive support comprises a resistive polymer substrate and particles of a Group VA or Group VIII metal, with resistivity of 10–100 'Kohms/square'. (1) may be used to measure O_2 concentration and to determine the pH of extracellular fluid, intramyocardial pH, etc. (1) is mechanically robust and simple, and accurate at constant O_2 pressure.

Nitrogen Oxide Sensor

KOREA ADV. INST. SCI. & TECHNOL. *U.S. Patent* 6,113,859
A bar-type NO_x gas sensor for measuring the density of NO_x exhaust gas from vehicles comprises a heater formed on an Al_2O_3 bar. A sheet of 1 mm thickness, made of Al_2O_3 , mullite, etc., covers the heater and a Pt thin film electrode is formed on the sheet. A thin WO_3 sensing film (1) is formed on the electrode. The sensor is used to detect the density of NO_x gas accurately, based on variations in electrical conductivity. The sensor has improved structure and prevents temperature variations in (1).

Detecting Label-Bearing Targets

UNIV. NORTH CAROLINA *U.S. Patent* 6,127,127
The presence of label-bearing targets, such as biomolecules in samples, is detected using an electrode containing a non-conducting self-assembled monolayer of phosphonate molecules having a group covalently bound to a binding member. The monolayer and the target complex are contacted with, for example, $\text{Ru}(\text{bpy})_3^{2+}$ to oxidise the label-bearing target. Label-bearing targets such as nucleic acids, proteins, antigens, antibodies, etc., can be detected.

Fabrication of a UV Lamp for Cleaning Air

CHUNG SHAN INST. SCI. & TECHNOL. *U.S. Patent* 6,135,838
Fabrication of a UV lamp for treating waste gases involves formulation of a photocatalyst coating sol; dip coating a glass fibre cloth with the photocatalyst sol. The cloth, impregnated with oxidation catalyst of Pd, Pt, Au or Ag, is wrapped on a UV lamp. The waste gases are treated by UV irradiation to generate free electron and electron hole pairs which can decompose pollutants into harmless gases.

Cermet Electrodes for Sensors

ROBERT BOSCH G.m.b.H. *German Appls.* 1/99/06,306–307
Cermet electrodes for a sensor are produced by applying a Pt-Zr dioxide paste to a substrate to form electrodes. Zr is added to the paste. The electrodes are sintered and then treated to form 3-phase boundaries, using glass coal as a pore former, to produce open porosity. The sensor determines the O_2 concentration in I.C.E. exhaust gases. The current load capacity is increased.

HETEROGENEOUS CATALYSIS

Hydrogen Refinement Apparatus

MATSUSHITA ELECTRIC IND. CO. LTD.

European Appl. 1,046,612

A H₂ refinement apparatus comprises a reaction chamber equipped with a CO-shifting catalyst body (1) which contains Pt supported on a metal oxide carrier of BET specific surface area of $\geq 10 \text{ m}^2 \text{ g}^{-1}$. Operation of the apparatus is controlled by keeping the temperature of (1) at 150–450°C. The apparatus operates stably when activated and stopped repeatedly. It provides improved heat resistance and reduced deterioration of (1) and decreases CO concentration.

Production of Pyrroles

BASF A.G.

European Appl. 1,046,639

Pyrrole derivatives are prepared by the catalytic dehydrogenation of pyrrolidines at 150–300°C and 0.01–50 bar on a catalyst comprising: Pd on a rare earth or Group IVB metal oxide; or Pt and Pd on Al₂O₃ or a rare earth or Group IVB metal oxide; and alkali(ne earth) metal oxide. The catalyst has high activity and stability.

Exhaust Gas Emissions Control

JOHNSON MATTHEY PLC

World Appl. 00/53,903

A catalyst system for the aftertreatment of exhaust gases from a gasoline-fuelled I.C.E. (1) designed to operate under stoichiometric conditions, comprises a first catalyst of Pt and/or Rh, to catalyse a shift reaction between CO and H₂O or the conversion of a reactant to generate a H₂-enriched exhaust gas; a source of gaseous O₂; a trap for hydrocarbons; and a three-way catalyst to oxidise catalytically hydrocarbons and CO and to reduce NO_x. Pollutants are reduced from (1), particularly during start-up, under rich conditions.

Hemi-Hydrogenation of Dinitriles

RHODIA FIBER & RESIN INTERM. *World Appl.* 00/59,870

Hemi-hydrogenation of dinitriles into the corresponding aminonitriles uses H₂ in the presence of a catalyst system comprising Ru supported on an acetylene black (1) (obtained by pyrolysis of paraffinic oils). Selectivities of $\geq 80\%$ in aminonitriles for a percentage transformation of dinitrile of $\geq 60\%$ may be obtained. (1) gives higher selectivities in aminonitriles than obtained with Raney Ni catalysts.

Hydrocarbon Hydrogenation Catalyst

PHILLIPS PETROLEUM CO.

World Appl. 00/64,846

A hydrocarbon hydrogenation catalyst, useful for converting highly unsaturated hydrocarbons containing S impurities to less saturated hydrocarbons, comprises Pd/TiO₂ (1), prepared by combining TiO₂ and Pd, and drying and calcining the combination. (1) is used for preparing monoolefins from hydrocarbons with a higher degree of unsaturation, for example, the feed stream from a depropaniser. In the presence of S, (1) provides increased selectivity to a less unsaturated hydrocarbon.

Dual Functional Catalyst for Acid Catalysis

CHINA PETRO-CHEM. CORP.

U.S. Patent 6,117,812

A 0.01–1 or 0.02–0.6% Pd/Al₂O₃ (1) or superacid-type catalyst, for acid catalysis, comprises multiple window lattice shaped pellets each having external teeth with notch peripheral surface area to increase the efficiency of fractionation. (1) is produced by moulding, for example Al(OH)₃, to a required shape, calcining at 800–1500 or 900–1300°C, adding Pd salt solution and drying. (1) can be loaded directly into catalytic distillation columns. Removing the deactivated catalyst is easy.

Selective Dehydrocyclisation of Paraffins

UOP LLC

U.S. Patent 6,132,595

A catalyst system for the selective dehydrocyclisation of paraffins has a non-acidic large core molecular sieve and a surface layer of one or more of Group IVA metals, In, Group VIIB metals, Fe, Zn, Au, Bi and uniformly distributed Pt group metal. A bound L-zeolite catalyst with a surface layer of Sn or In and uniformly distributed Pt results in substantial yield improvements in the catalytic reformation process.

Oxidation of Volatile Organic Compounds

NASA U.S. NAT. AERO. & SPACE ADMIN.

U.S. Patent 6,132,694

Oxidation of volatile organic compounds (VOCs) to CO₂ and H₂O is initiated at low temperatures. A gaseous mixture comprising a VOC and an oxidising agent (such as ambient air containing the VOC) is exposed to a catalyst (1) containing 1–50 wt.% Pt, Pd, Rh, Au and/or Ag, and 50–99 wt.% of a metal oxide, such as Sn, which possesses one or more stable oxidation states. (1) requires minimum heat and all the oxidised components are non-toxic so air pollution is prevented.

Dehydrocyclisation of Hydrocarbons

PHILLIPS PETROLEUM CO.

U.S. Patent 6,140,546

Dehydrocyclisation of hydrocarbons, such as paraffins, is performed by contacting dehydrocyclisable hydrocarbon in the presence of a catalyst containing Pt group metal. AlCl₃ (0.01–10 ppb) is provided to the dehydrocyclising feed stream. The catalyst has an extended run life and longer operating runs between regeneration. An increased and stable research octane number (RON) and stabilised BTX (benzene, toluene and xylene) production are obtained.

Isomerisation of Xylenes

UOP LLC

U.S. Patent 6,143,941

A selective process for isomerising a non-equilibrium feed mixture of xylenes and ethylbenzene is described. It comprises contacting the feed mixture in the presence of H₂ with an oil-dropped spherical catalyst containing a zeolitic aluminosilicate (1), a Pt group metal and an amorphous Al phosphate binder. Isomerisation occurs at 300–600°C and 100 kPa–5 MPa. (1) has a pore diameter of 5–8 Å and a 4.6 torr H₂O capacity of 3–5 wt.%. The catalyst has superior activity, selectivity and stability.

Exhaust Purification for Motorcycles

YAMAHA MOTOR CO. LTD. *Japanese Appl.* 2000/234,514

An exhaust gas purification catalyst for motorcycles has Pt, Rh and Pd as the catalyst elements carried on both surfaces of a plate bent to form a honeycomb, which is then arranged in the enlarged-diameter portion of an exhaust tube (1). Pressure loss in (1) is reduced and leads to improvements in engine performance and fuel consumption. Loss by melting, damage or rupture of the catalyst-carrying plate, caused by high temperature, is prevented and the efficiency of hydrocarbon purification during low speed running of the engine is improved.

Combustion of Methane Fuel

DENRYOKU CHUO KENKUSHO

Japanese Appl. 2000/254,505

A catalyst for combustion of CH₄ fuel comprises using Pd acetate to form Pd or PdO layers on a support containing SnO₂. The oxidising property of the catalyst is improved irrespective of temperature. CH₄ conversion ratio is also improved. The generation of N₂O by pre-burning is reduced. The catalyst does not deteriorate even after a prolonged period in use.

Direct Synthesis of Hydrogen Peroxide

DEGUSSA-HUELS A.G.

German Appl. 1/99/12,733

A heterogeneous catalyst used in the direct synthesis of H₂O₂ from H₂ and O₂, is based on Pd or ≥ two metals of the Pt group metals and sub-Group I, optionally using a solvent. The catalyst is produced by spray or flame pyrolysis of an aerosol of a solution or suspension of a metal compound, followed by separation of the resultant metal or alloy particles from the gas stream. The catalyst gives significantly higher H₂ selectivity and/or attainable H₂O₂ concentrations than existing catalysts.

HOMOGENEOUS CATALYSIS

(2-(Arylsulfonyl)-ethenyl)-benzene Derivatives

SUMITOMO CHEM. CO. LTD.

British Appl. 2,348,201

A safer and more efficient process (1) for producing (2-(arylsulfonyl)-ethenyl)-benzene derivatives, useful as intermediates for pharmaceutical and agricultural products, is described. (1) comprises reacting a 2-(arylsulfonyl)-ethanol with an acid anhydride in the presence of a base and reacting the product with an aromatic halide in the presence of a Pd catalyst, such as Pd chloride, acetate, oxide, etc., and a base. (1) avoids the handling of solid aryl vinyl sulfones.

Promoted Hydrosilation Reactions

CK WITCO CORP.

European Appl. 1,035,126

Compounds containing Si-C bonds are prepared in high purity by the hydrosilation of hydridosilane with an olefinic reactant, in the presence of a Pt catalyst and a carboxylic acid promoter having 14C atoms, a molecular weight > 60 and a boiling point > 118°C at atmospheric pressure. The yields and rates of hydrosilation are improved under mild conditions.

δ-Lactone Production

FORSCHUNGSZENTRUM KARLSRUHE G.m.b.H.

European Appl. 1,036,791

Production of δ-lactone comprises reacting butadiene and CO₂ under pressure and at constant reactant mole ratio in the presence of Pd₂(dba)₃, Pd(PPh₃)₄ or Pd(OAc)₂/PPh₃ (dba = dibenzylideneacetone) catalyst. High yields are obtained at lower operating temperature. The method is based on the observation that high partial pressures of CO₂ inactivate the catalyst and this effect far outweighs the expected advantage of the increased CO₂ pressure.

Production of Diol Mononitrate Esters

DINAMITE DIPHARMA S.p.A. *European Appl.* 1,038,862

Mononitrate esters of dihydroxyalkyl, dihydroxycycloalkyl or dihydroxypolycycloalkyl compounds are produced by hydrogenating the corresponding dinitrate esters using a Pt(0) catalyst. The process avoids using carcinogenic hydrazine hydrate and avoids waste disposal problems associated with Zn/acid reduction. Esters, such as isosorbide mononitrate, are useful for treating cardiovascular diseases.

Carbonylation of Conjugated Dienes

SHELL INT. RES. MIJ. B.V.

World Appl. 00/56,695

A catalyst (1) for carbonylation of conjugated dienes, such as for the production of dimethyl adipate and methyl pentenoate, comprises Pd cations and a bridged cyclic P-containing ligand and anions. The ligand comprises two 5- or more-membered cyclic groups each containing P, linked by a 1-4 atom organic group. (1) has an unexpectedly high activity (allowing molar ratios of conjugated diene to Pd of > 300:1) while still achieving high selectivity.

Optically Active β-Hydroxyester Derivatives

UBE IND. LTD.

U.S. Patent 6,121,475

An optically active β-hydroxyester derivative (1) is prepared by an enantiomeric aldol addition reaction of an aldehyde and ketene silylacetal in the presence of an organic base and a Pt-containing catalyst prepared from a bisphosphine-aryloxyacyl Pt complex and strong acid in an O₂-containing gas. (1) are intermediates for pharmaceuticals and agrochemicals.

FUEL CELLS

Solid Electrolyte Fuel Cell

SHINKO ELECTRIC IND. CO. LTD. *European Appl.* 1,058,329

A solid electrolyte fuel cell for generating power includes a solid electrolyte device with electrodes formed on both sides of an ion-conductive solid electrolyte substrate. O₂ is supplied to the cathode side while CH₄ gas is supplied to the anode side. The electrode on the anode side acts as oxidation catalyst for the CH₄ fuel. Either CoNiO₂ or CoO particles are blended in a porous Pt layer, or PdCoO₂ particles are formed on the surface of the porous Pt layer, as the oxidation catalyst. The fuel cell has superior and more efficient power generation performance.

Water Gas Shift Reaction

MATSUSHITA ELECTRIC WORKS LTD.

World Appl. 00/54,879

A catalyst (1) for the H₂O gas shift reaction comprises at least Pt supported on a metal oxide carrier. (1) has good efficiency in removing the CO in H₂, such as the reformed gas in the electric power-generating system of a fuel cell at 200–400°C. (1) is adaptable for use in a start-stop repeat-operation small portable fuel cell power-generating system.

Ternary Catalyst for Fuel Cell Electrode

SYMYX TECHNOLOGIES INC. *World Appl.* 00/55,928

A Pt-Ru-Ni catalyst for electrochemical reactor devices, fuel cell electrodes and other catalytic structures, is dispersed on the surface of an electrically conductive support. The electrochemical conversion of a hydrocarbon-based fuel and O₂ to H₂O, CO₂ and electricity in the fuel cell involves oxidising the fuel by contacting with the catalyst. The latter has high catalytic activity and high resistance to poisoning by CO. The efficiency of the fuel cell is improved and its size can be decreased.

Self-Support Fuel Battery System

TOYO ENG. CORP. *Japanese Appl.* 2000/233,903

A self-support type fuel battery system for motor vehicles comprises a methanolysis device (1) and a shift reaction device (2). H₂O vapour is supplied to the H₂ permeation sides of the Pd films of (1) then (2). A non-transparent gas is formed comprising damp H₂ and CO₂. Moist H₂ is supplied to a fuel cell anode and air to the cathode. Unreacted air, H₂ and non-transparent gas are then supplied to the heating unit of (1). Waste gas from the heating unit is used as the heat source for gasification of MeOH and H₂O.

Electrode for Fuel Batteries

TOYOTA JIDOSHA K.K. *Japanese Appl.* 2000/243,406

An electrode for fuel batteries contains a cathode formed of C particles supporting Pt particles and TiO₂ particles, an electrolyte film and an anode. When generating electricity using H₂ and O₂ as fuel, UV rays of the wavelength \leq 410 nm are irradiated on the cathode. The cathode reaction is accelerated by the UV irradiation giving high output from the battery.

ELECTRICAL AND ELECTRONIC ENGINEERING

Highly Temperature Stable Conductive Barrier

SHARP K.K. *European Appls.* 1,054,440–441

A highly temperature stable conductive barrier (1) comprises a substrate, a first barrier layer and an Ir-refractory metal-oxygen (Ir-M-O) composite film. (1) suppresses diffusion of Ir into the substrate so the Ir-M-O composite film remains conductive, and resists peeling and hillock formation during high temperature annealing, even in O₂. (1) is used for an electrode of a ferroelectric capacitor, in nonvolatile metal/ferro/metal/insulator/Si (MFMSI), DRAM, etc., devices.

Ruthenium and Platinum Organometallics

TANAKA KIKINZOKU KOGYO K.K. *World Appl.* 00/58,245

Ru and Pt organometallic compounds (1) are used for forming Ru, Ru oxide or Pt thin film on a substrate by organometallic vapour phase epitaxy. Thin film of excellent thermal stability can be deposited at constant rates. (1) are used as electrode materials in semiconductor devices. Downsizing current in electrical apparatus is achieved.

Giant Magnetoresistive Stack

SEAGATE TECHNOLOGY LLC *World Appl.* 00/63,714

A giant magnetoresistive (GMR) stack for use as the read sensor (1) in a magnetic read head includes a Ni-Fe-Cr seed layer; a free layer of ferromagnetic material having a rotatable magnetic moment; a ferromagnetic pinned layer of fixed magnetic moment; a Cr-Mn-Pt pinning layer adjacent to the latter; and a nonmagnetic spacer layer between the free and pinned layers. (1) exhibits a GMR ratio of \geq 12%, the highest ever reported in simple spin valves. The Cr-Mn-Pt pinning layer has a high blocking temperature to stop exchange coupling disappearing.

Palladium-Containing Product

SUPERIOR MICROPOWDERS LLC *U.S. Patent* 6,159,267

A particulate product (1) used as catalysts in the preparation of thick film paste, comprises high quality particles (0.1–4 μ m) in which a polycrystalline metallic phase comprises $>$ 10 wt.% Pd. Impaired film performance at high electrical conductivity is avoided. (1) has good dispersion of particles in the paste, and a low degree of particle agglomeration. (1) can be used in the manufacture of multilayer capacitors, multichips, flat panel displays, etc.

Multilayer Ceramic Capacitor

KYOCERA CORP. *Japanese Appl.* 2000/223,352

A multilayer ceramic capacitor consists of a dielectric ceramic layer (1) using BaTiO₃ as its major constituent, and an internal Ag-Pd electrode layer. When (1) is (100-*a*)BaTiO₃.*a*Bi₂Ti₂O₇, it contains (in mol. parts) a major constituent having $a = 2.5$ –6.0, and in equivalents: 0.7–1.9 Nb in Nb₂O₅; 0.8–1.9 Zn in ZnO; a rare earth element (RE) \leq 0.4 (0 is not included) in RE₂O₃; and 0.4–8.3 Si in SiO₂. The multilayer ceramic capacitor has high dielectric constant, \geq 2000, and improved thermal properties.

Multilayer Printed Wiring Board

IBIDEN CO. LTD. *Japanese Appl.* 2000/252,622

A multilayer printed wiring board includes roughening surface on each conductive Cu pattern formed on corresponding resin insulating layers, with 0.5 mg m⁻² Pd atoms being distributed between the patterns. The exfoliation of the conductor circuit (1) is eliminated or reduced and the migration of Cu between (1) at high temperature and high humidity is suppressed.

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