

NEW PATENTS

METALS AND ALLOYS

Gold-Colour Tarnish Resistant Alloy

N. J. LAMONTAGNE et al *U.S. Patent 5,409,663*
An In-free gold-coloured alloy comprises (by wt.%): ≤ 10 Au, 28–35 Cu, 19.5–22.5 Ag, 6–11 Pd, 22–32 Zn, 0.1–1 Al and 0.5–3 Pt. Also claimed is a gold-coloured alloy containing: 5–11 Au, 28–35 Cu, 19.5–22.5 Ag, 6–11 Pd, 22–32 Zn, 0.1–1 Al, 0.5–3 Pt, 5–8 Ni, 0.1–1 Si and 0.01–0.1 B. The alloy is tarnish and corrosion resistant.

CHEMICAL COMPOUNDS

Chloropentane Ammine Iridium Dichloride

TANAKA KIKINZOKU KOGYO K.K. *Japanese Appl. 6/298,535*
IrCl₃ is dissolved in HCl, added to an autoclave with NH₃ water at 120°C for 24 h, to form chloropentane ammine Ir dichloride, followed by filtration and purification by recrystallisation. The process is simple and efficient and the compound is used as a catalytic material in vehicles.

ELECTROCHEMISTRY

Electrode Used in Electrolytic Processes

IMPERIAL CHEM. IND. P.L.C. *World Appls. 95/5,498–99A*
An electrode with a valve metal (alloy) substrate is given an outer coating of an electrocatalytically-active material, that contains ≥ 10 mol % of Ru oxide and ≥ 20 mol % of a non-noble metal oxide, by thermal spraying or PVD. Also claimed is an electrode, where the coating has a loading of 5–100 g/m² of nominal electrode surface. The electrode has long life and is used in the electrolytic production of Cl₂, etc.

Electrolytic Production of Palladium Deuteride

OSAKA GAS CO. LTD. *Japanese Appl. 6/293,985*
Production of Pd deuteride by electrolysis comprises occluding D into Pd at 10°C lower than normal electrolytic temperature and raising the temperature to the normal electrolytic temperature. The Pd deuteride can initiate normal temperature nuclear fusion. In an example, a Pd plate cathode, Pt wire anode, and LiOD 0.5 mol/l D₂O solution were used during electrolysis at 10°C and 200 mA/cm². The D:Pd ratio was 0.95.

Iridium Oxide-Coated Electroplating Electrode

NIPPON STEEL CORP. *Japanese Appls. 6/293,998–294,000*
A Ti nitride ceramic layer is on the surface of a Ti metal base material and an Ir oxide ceramic layer on the surface of the Ti nitride ceramic layer. Layers of Ir and Ir oxide may be formed on the surface of the Ti base. The Ir oxide layer protects the electrode base from penetrating pin holes, thus preventing corrosion, and so can be used under severe conditions.

Corrosion Resistant Insoluble Electrode

NIPPON STEEL CORP. *Japanese Appl. 6/299,396*
The insoluble electrode comprises a conductive metal electrode mother material, a conductive IrO₂ top layer and a layered structure of porous non-conductive material and conductive oxide consisting mainly of IrO₂ to fill the voids in the porous non-conductive material between the mother material and the top layer. The electrode has a high corrosion resistance even under a high current density.

Electrode for Surface Treating Stainless Steel

DAISO CO. LTD. *Japanese Appl. 6/346,267*
The electrode comprises a conductive metal base of valve metal or its alloy, covered with an electrode active layer containing Pt group metal oxide prepared by the sol-gel method and a valve metal or oxide of Group IVB (not C). The base body is protected from electrolyte owing to the dense and strongly adhered thin wet gel film. In an example, the electrode was used as the anode in aqueous Na sulphate using a Pt cathode at 200 A/dm². The electrode is used for electroplating of Sn, Zn, Cr or treating stainless steel.

Preparation of Ammonia or Alkali Solution

BASF A.G. *German Appls. 4,326,539–40*
Preparation of NH₃ or aqueous alkali solution (1) and peroxodisulphate (2) from electrolytes containing NH₄ or alkali sulphate involves electrolysis of dilute or concentrated sulphate solution in a cell with cation exchanger membrane and a smooth Pt anode, using additive containing C and S and/or N. H₂S₂O₈ and/or H₂SO₄ are also prepared by electrolysis of H₂SO₄ solution. NH₃ or (1) and (2) are prepared with high current yield and the process uses waste liquors.

ELECTRODEPOSITION AND SURFACE COATINGS

Electroless Plating on Palladium Seed Layer

ASTARIX INC. *World Appl. 95/2,900A*
A structure comprising Al-Pd alloy with an active catalytic surface, provided by etching away Al, acts as a catalytic 'seed' layer. Small isolated regions can be plated at the same rate as larger regions or dense arrays. Good adhesion of the plated film is obtained. It is useful in electroless plating for forming high-resolution conductive wiring patterns in semiconductors, etc.

Selective Seeding for Electroless Plating

INT. BUSINESS MACHINES CORP. *U.S. Patent 5,380,560*
A polyimide surface of a metal or alloy is selectively seeded for electroless deposition of a further metal by treating with an activating solution of PdSO₄, Pd(ClO₄)₂, Pd(CF₃SO₃)₂, Pd(NO₃)₂ or Pd(BF₄)₂. Pd is removed from the polyimide by rinsing with deionised H₂O or aqueous Na citrate and plated further with metal. The seeding of metal interconnections on Pd(II) polyimide dielectrics is highly selective.

Electroless Plating

SHIPLEY CO. INC.

U.S. Patent 5,395,652

Electroless plating for multilayer circuits uses a catalyst of reduced noble metal bromo complex with bromide ions : noble metal (Pd) ions $\geq 100:1$. The substrate to be plated is contacted with the bromo Pd solution and the catalyst is then reduced by, for example, a borohydride or an amine borane, and electrolessly plated. A stable electroless plating is achieved. There is high catalytic activity even with a large Br molar excess and no displacement coatings are formed on metallic surfaces.

Platinum Coating with Uniform Thickness

TANAKA KIKINZOKU KOGYO K.K.

Japanese Appl. 6/336,698

Pt coating comprises holding a workpiece with top and bottom clamps coated with insulation material in a Pt coating jig, applying chemical etching as pretreatment, holding the workpiece with top and bottom clamps uncoated with insulating material in the Pt jig, immersing the workpiece into the Pt coating bath and applying electric current through the uncoated top and bottom clamps. Pt is coated at a uniform thickness around the clamped part.

Palladium-Copper Alloy Plating

TAKAMATSU MEKKI KOGYO K.K.

Japanese Appl. 6/340,983

A Pd-Cu alloy plating coating layer contains 50–95% Pd and 2–50% Cu and is made by electroplating using a solution containing, for example, ethylene diamine Pd complex salt and Cu sulphate. The coating may have a metal plating layer and/or Cu alloy plating layer as a base layer. The Pd-Cu plating coating is used for watch cases or glasses for a white coloured appearance. It has high coating quality and avoids inducing allergies by using Cu instead of Ni.

Electroless Plating Catalyst

ISHIHARA YAKUHIN K.K.

Japanese Appl. 7/11,448

Electroless plating catalyst for selective coating of Ag base material comprises an aqueous solution containing 0.0001–0.5 mol/l Pd compound and 0.1–10 mol/l of alkali metal halide, sulphate of alkali earth metal, NH₃ halide or NH₃ sulphate. The catalyst solution assures selective electroless plating only on the Cu base material, and eliminates skip-phenomenon on printed wiring board.

Palladium Coating Solution

KOJIMA KAGAKU YAKUHIN K.K.

Japanese Appls. 7/11,475–76

Pd coating solution comprises: soluble Pd salt 1.0–40.0 g/l as Pd; pyridine sulphonate or its salt, or pyridine carboxylate or its salt 0.1–20.0 g/l; and urea or its salt 1.0–50.0 g/l. The Pd coating solution may also contain an anionic surfactant. This high purity Pd coating solution forms a Pd deposit, preferably $\geq 5 \mu\text{m}$, suitable for electronic devices or for ornaments. The obtained Pd deposit has high gloss and high ductility, does not crack under bending or under NH₃ exposure testing, and does not discolour under bonding at an elevated temperature.

Electroplated White Palladium Coatings

ENTHONE-OMI INC.

German Appl. 4,428,966

A process for depositing white Pd coatings on a substrate comprises immersing the substrate in a Pd electroplating bath containing, as brighteners, special sulphone and special N compounds, such as for myl benzene sulphonate and sulphopropyl pyridinium betaine. The process provides a stable electroplating solution which enables the production of white Pd coatings, preferably $\leq 5 \mu\text{m}$ thick, without using metallic brighteners such as Ni and Co salts. It is used for producing a white finish on jewellery, etc.

APPARATUS AND TECHNIQUE

Gas Vapour Sensor Element

MOTOROLA INC.

U.S. Patent 5,386,715

A gas vapour sensor element for arsine or phosphine comprises a substrate with a surface and a Au-Pd alloy layer on the surface. The alloy layer has an as-applied by volume Pd concentration of 1–20%. The process of sensing gas vapours comprises exposing an Au-Pd alloy film disposed on the substrate to gas vapour and detecting a change in a work function of the film. The sensing element can detect less than 1 ppb of phosphine and much less than 1 ppb arsine.

Organopolysiloxane Composition

SHINETSU CHEM. IND. CO. LTD.

Japanese Appl. 6/345,970

An organopolysiloxane composition useful as a sealant for optical devices comprises alkenylorganopolysiloxane, silicone compound, Pt catalyst and inorganic filler with a refractive index different from that of the cured product, such as SiO₂-TiO₂ glass. The composition gives cured product, with good moisture and heat resistance, mechanical strength, low thermal expansion coefficient and superior transparency.

HETEROGENEOUS CATALYSIS

Sulphided Platinum Charcoal Catalyst

DEGUSSA A.G.

European Appl. 629,439A

Sulphided catalyst containing 0.1–5.0 wt.% Pt on activated charcoal and 0.1–2 mole S/mole Pt is obtained by mixing an aqueous Pt compound solution with an aqueous suspension of activated charcoal and heating the mixture at 70–100°C. The solution also contains an oxidant and the charcoal has particle diameter of 10–40 μm , specific surface area of $> 500 \text{ m}^2/\text{g}$ and pH of > 5 . The catalyst is used in the selective hydrogenation of *p*-aminodiphenyl amine and after partial poisoning by S has improved catalytic activity.

Exhaust Gas Purification Catalyst

NE CHEMCAT CORP.

European Appl. 633,052A

An exhaust gas purification catalyst comprises crystalline Ir silicate with atomic ratios 50–800 for Si:Ir and ≥ 15 for Si:Al. Also claimed is a catalyst-coated structure comprising a refractory support at least partially coated with catalyst. The catalyst is highly durable to hydrothermal ageing and has high activity for exhaust gas from lean burn I.C.E., boilers or gas turbines.

Catalytic Combustion Device for Purification of Polluted Air

MANNESMANN A.G. *European Appl.* 633,065A
Device for purification of polluted exhaust air by heterogeneous catalysis consists of a non-metallic, gas-permeable, spiral formed carrier element, that has an adsorbing layer of a coated fibrous material impregnated with Pt by sol-gel deposition, with a heatable metallic woven band. The carrier element for the coating substances is a band of knitted glass-fibre yarn, which with the metallic woven band is formed into an annular spiral disk. The device has high specific surface area and purifies exhaust air/gases from diesel engines, food processing, chemical manufacture and waste processing.

Ozone Decomposition

E. DODUCO DUERRWAECHTER G.m.b.H. & CO.
European Appls. 634,205A and 635,685A
An unheated ozone decomposition catalyst consists of a metal or plastic mesh support coated with noble metal, such as Pt, Pd, etc. An air conditioning unit for cooling ambient air has the O₃-splitting Pd catalyst in front of the cooling apparatus. The catalyst can decompose O₃ in an air stream in air conditioning and ventilating equipment, cars, etc. It is effective at ambient temperature and also at 0°C.

Continuous Dialkyl Carbonate Production

BAYER A.G. *European Appl.* 634,388A
Continuous production of dialkyl carbonates of formula CO(OR)₂ (1), where R = 1-4C alkyl, is effected by gas-phase reaction of CO with RONO in the presence of a supported Pt group metal catalyst, an inert gas, ROH and optionally NO, with periodic or continuous addition of a halogen. The reaction is performed at 50-170°C and 0.5-10 bar using RONO:CO volume ratio of 0.1-10:1. The (1) are useful as solvents, carbonylating and acylating agents, intermediates for urethanes, ureas and gasoline octane improvers. Addition of halogen avoids possible corrosion problems associated with HCl gas addition.

Paraffin Dehydrogenation

ENIRICERCHÉ S.p.A. *European Appl.* 637,578A
Light olefins (especially 2-5C) are dehydrogenated in a reactor at 450-800°C, 0.1-3 atm and GHSV 100-10,000/h over a Pt group metal catalyst of 0.1-33.6 wt.% Ga (as Ga₂O₃); 1-99 ppm Pt; 0-5 wt.% (as oxide) alkali or alkaline earth metal; and 0.08-3 wt.% modifying SiO₂ on δ , θ , δ/θ , θ/α or $\delta/\theta/\alpha$ Al₂O₃. Coked catalyst is regenerated at > 4000°C.

Production of Phenols

TOSOH CORP. *European Appl.* 638,536A
Phenol (1) production comprises reacting an aromatic compound with O₂ and H₂ in the liquid phase in the presence of a supported Group VIII metal catalyst containing Ru, Rh, Ir and particularly Pd and Pt, and in the co-presence of a V compound, such as V(III) acetylacetonate, etc. Halogens in the catalyst are \leq 0.15 wt.%. (1) are produced with high selectivity. High catalytic activity is achieved over a long time. Phenols are used for resins and alkyl phenols.

Partial Oxidation of Natural Gas

SNAMPROGETTI S.p.A. *European Appl.* 640,559A
Synthesis gas and formaldehyde are obtained by catalytic partial oxidation of natural gas, with a Pt group metal catalyst at \geq 300°C. The catalyst is a wire mesh or is deposited on an inorganic carrier. The Pt group metal(s) are 0.1-20 wt.% of the wt. of catalyst plus carrier, Syngas is obtained at UHSV and lower temperatures than at present. Further reaction can produce MeOH and optionally dimethyl ether, or be used in the Fischer-Tropsch synthesis.

Pristine Alkylsiloxanes

DOW CORNING CORP. *European Appl.* 641,799A
A pristine alkylsiloxane (A), containing 1-3C alkyl or phenyl, is prepared by reaction of siloxane polymer with olefinic compound in the presence of a supported Pt catalyst. Yield of (A) is > 99.9% and consists of < 10 ppm SiH, < 100 ppm (CH₂=CH- and < ppm Pt. Also claimed is a preparation of (A) by reacting under anhydrous conditions a SiH containing siloxane polymer and an olefinic compound with \geq 4C, in the presence of the catalyst and O₂. The process produces extremely pure alkylsiloxanes which are clear, colourless and odourless.

Dibenzylamine Preparation

DSM N.V. *European Appl.* 644,177A
Dibenzylamine preparation comprises hydrogenation of benzaldehyde in the presence of NH₃ and a Pd-containing catalyst, using 3-60% of a solvent or dispersion medium with respect to benzaldehyde. Preferably, the solvent or dispersion medium is H₂O in an amount of 5-20 wt.%; the catalyst is Pd/C and the reaction is performed at 80-120°C. High selectivity of > 95% with complete conversion is obtained. High production capacities are achieved and simple recovery procedures can be used.

Catalyst for Thermal Decomposition of Nitrogen Oxides

NAT. SCI. COUNCIL *European Appl.* 645,171A
A catalyst for the thermal decomposition of NO in I.C.E. or industrial exhaust gases comprises an Al₂O₃ or Mg aluminate support and an active phase of Pd crystallites on a Pd alloy dispersed on the support. The Pd alloy is Pd crystallites modified by addition of at least one alloying metal of Cu, Ag or Au. The catalyst has high activity especially at \sim 800°C, is resistant to poisoning by high concentrations of O₂ or SO₂ in the feed gas and retains high activity over time.

Catalyst for Removing Dioxins

NE CHEMCAT CORP. *European Appl.* 645,172A
Catalyst (1), for treating exhaust gases containing dioxins comprises Pt, Pd and/or Ir or their oxides on a support (2) of SiO₂/B₂O₃/Al₂O₃ composite oxide or zeolite, is obtained by spraying or dripping a solution or dispersion of the active components onto a powder of (2). Exhaust gas (3) containing dioxins at concentration below 10 ng/m³ is contacted with (1) at 150-280°C. (1) has long life and reduces dioxin concentration in (3) to below 0.1 ng/m³ and also suppresses additional dioxin formation.

Palladium Three-Way Catalyst

ENGELHARD CORP.

World Appl. 95/235A

A layered catalyst composite consisting of 2 similar layers is used as a three-way catalyst to treat automotive exhaust gas. The first layer comprises a first support, a first Pd component, an oxygen storage component, and optionally a Pt group metal component other than Pd, a Zr component, an alkaline earth metal component, and either La or Nd. The second layer is similar, but has no O₂ storage component. The support is dispersed in an aqueous liquid and the other components are introduced in H₂O-soluble or dispersed form. The mixture is applied to the substrate and the H₂O-soluble components are converted to insoluble forms to form the first layer; the second layer is then applied in a similar way.

Purification of Organic Impurities in Inert Gas

SINCO ENG. S.P.A.

World Appl. 95/2,446A

O₂ or O₂ containing gas (1) is added to an inert gas containing organic compounds as impurities circulated at 250–600°C on a catalyst bed of Pt optionally with Pd on an inert porous support to purify the gas. Amount of (1) added is stoichiometric with respect to impurities, or in excess, so that gas leaving the bed contains < 10 ppm O₂. Inert gas is N₂; impurities are aldehydes, glycols and glycol oligomers. The process avoids a deoxidation stage with H₂ prior to recycling of purified gas.

Catalyst for I.C.E. Exhaust Gases

PERSTORP A.B.

World Appl. 95/7,138A

A catalyst for I.C.E. is manufactured by depositing oxides of Pt and La or Ce via an aqueous salt solution and hydroxide. Preferably, only one solution is used and the molar ratio Pt:Ce (or La) is 3:1 to 1:3. The support body is a porous ceramic and the support is wash-coated to increase its surface area. The catalyst can remove CO, NO_x and hydrocarbon gases and its light-off temperature for the gases, especially CO, is much lower than a prior art Pt-Rh catalyst.

Dehydrocyclisation of Paraffins

UOP

U.S. Patent 5,384,038

A hydrocarbon feedstock is reformed to obtain an aromatics-rich product by contact with a bed of catalyst particles having a surface layer of uniformly dispersed Pt and Pd on a support of a non-acidic large pore molecular sieve, such as zeolite L, and an inorganic oxide binder, such as SiO₂. The specified Pt and Pd metal distribution improves catalyst activity and selectivity to aromatics, and enhances catalyst stability when small amounts of S are in the feed.

Hydrogenation of Aromatics and Olefins

SHELL OIL CO.

U.S. Patent 5,391,291

A hydrocarbon feedstock containing components of boiling point $\geq 125^\circ\text{C}$ is hydrogenated by contacting with H₂ and a Pt group metal, such as 0.05–3 wt.% Pt and Pd on zeolite at $\geq 150^\circ\text{C}$. A treatment of diesel fuel feeds containing aromatics and olefins, converted to saturates and saturated aromatics to acyclics, is also claimed, but at $\geq 225^\circ\text{C}$. The catalyst hydrogenates aromatics and olefins while minimising hydrocracking.

Catalyst for Chlorine Dioxide Generation

AMTX INC.

U.S. Patent 5,391,533

A catalyst system comprising a porous sintered Al₂O₃ and SiO₂ agglomerate catalyst substrate, with Al₂O₃ particles joined to other Al₂O₃ particles via point bonding silicates, is produced by compressing a particulate material at low pressure. The Pt group metal, preferably Pd, is supported on the substrate. The catalyst is useful in ClO₂ generation. The system creates a unique microstructure with a unique surface area which contributes to maintaining constant conversion efficiency and high chemical activation levels.

Low Pressure Manufacture of Cyclohexane Dicarboxylate Ester

EASTMAN CHEM. CO.

U.S. Patent 5,399,742

Manufacture of dimethyl cyclohexane dicarboxylates in a continuous process comprises feeding H₂ gas with a liquid mixture of the dimethyl cyclohexane dicarboxylate product and the corresponding dimethyl benzene dicarboxylate to a hydrogenation zone containing at least one fixed bed of Pd/Al₂O₃ catalyst. H₂ and a liquid product containing the dimethyl cyclohexane dicarboxylate product are removed. Conversions of > 99% are obtained with space-time yields of > 1000 g/l h. 1,4-Cyclohexane dicarboxylate is an intermediate in the production of cyclohexane dimethanol, which is used as a monomer in the preparation of condensation polymers.

Improved Preparation of Acetic Acid

SHOWA DENKO K.K.

Japanese Appl. 6/293,695

Preparation of acetic acid (1) comprises reaction of ethylene with O₂ and H₂O in the gaseous phase over Pd-V-H₃PO₄ catalyst prepared by treating supported Pd catalyst containing one or more metals (2) selected from Cr, V, Mo and W with H₃PO₄. The catalyst is prepared by supporting Pd containing salts and (2)-containing salts on SiO₂, Al₂O₃, etc. The process is performed in the gaseous phase, and (1) is prepared more economically than from liquid phase reactions.

Combustion Catalyst for CH₄-Type Fuel

DENRYOKU CHUO KENKYUSHO

Japanese Appl. 6/296,866

Rh is supported on a catalyst surface containing Pd or PdO (Rh:Pd = 1–25%) to form a combustion catalyst for CH₄ type fuel. The catalyst has good durability. In an example, 12 g/l Pd and γ -Al₂O₃ are supported on a cordierite honeycomb support, immersed in 2.5% Rh nitrate solution, ion exchanged to precipitate Rh and thermally treated at 700°C for 30 min to form catalyst with Rh:Pd of 3%.

Purification Catalyst for Exhaust Gas

NISSAN MOTOR CO. LTD.

Japanese Appl. 6/296,869

A catalyst, composed of Pd, activated Al₂O₃ and CeO₂ containing Fe and Zr, Co or Ni, and Li, Na, K, Ba or Sr as additive, is used to purify hydrocarbon, CO and NO_x in exhaust gas. In an example, activated Al₂O₃ supporting 2% Pd and CeO₂, containing Fe and Zr, were mixed and wet ground using HNO₃ to form a slurry, coated on a cordierite support, dried and burned at 400°C for 1 h, to form the catalyst.

2,3-Dihydro-1H-indene Derivative

OTSUKA PHARM. CO. LTD. *Japanese Appl.* 6/298,711
Preparation of optically active 2,3-dihydro-1H-indene derivative (1) and its salts (2) comprises catalytic hydrogenation of 2,3-dihydroindene derivative (3) or its salts in the presence of PtO₂ or Pd/C catalyst. Preferably, (3) is hydrogenated in suitable solvent such as lower alcohols at -20°C to 100°C under 1–50 atm for 0.5–10 h. A specific optically active stereoisomer of (1) and (2) with high optical purity can be prepared selectively by using these catalysts. (1) and (2) are useful as materials for drugs in treating hypoxia, etc.

Preparation of Chloroformic Acid Esters

UBE IND. LTD. *Japanese Appls.* 6/306,016–17
Preparation of chloroformic acid ester (1) comprises contact reacting Cl₂, CO and nitrous acid ester in the presence of a solid catalyst supporting a Pt group metal, such as Pd/Al₂O₃, and maintaining 0.1–100 moles of nitrous acid ester to 1 mole of Cl₂. The esters (2) may also be prepared by reacting CO with nitrite ester and HCl or nitrosyl chloride with a Pt group metal catalyst. (1) and (2) are starting materials for chemical manufacture. The one-step method forms esters selectively. The process may be performed without using toxic phosgene.

Catalytic Reduction of Nitrogen Oxides

AGENCY OF IND. SCI. & TECHN. *Japanese Appl.* 6/327,980
The catalyst substrate is coated with an undercoat containing 100 pts. wt. of a Pt group metal, such as Pt, Rh, Ir, Pd and Ru, 100–100,000 pts. wt. oxides of Ce, La, Nd, Ge or Ga, and preferably 0.0001–0.1 pts. wt. Au on a support and laminated with a top coat containing Al₂O₃, TiO₂, ZrO₂ or H type zeolite. NO_x is reduced efficiently and selectively. It can be used for purification of exhaust gases.

Decomposing Organic Halide

SUMITOMO METAL MINING CO. *Japanese Appl.* 6/335,619
Organic halide is removed from waste gas by contacting it with a Pt or Pt oxide loaded catalyst containing 0.1–5 wt.% Pt. The catalyst support is ZrO₂ prepared by reacting zirconyl nitrate with NH₃ water in the presence of organic halide containing gas, air and H₂O, followed by heating at 700–850°C. The organic halide, which contains Cl and/or F, such as freon or trichloroethylene, is decomposed. The catalyst is quite stable to concentrated HCl or HF, so it can be used over a long time.

Harmful Volatile Organic Halogen Treatment

KURITA WATER IND. LTD. *Japanese Appl.* 6/343,983
The treatment comprises contacting a gas containing volatile organic halogen compound with a reducing agent (H₂) in the presence of a catalyst supporting a metal, preferably, Pd, Pt, etc., in an amount of 0.1–10 wt.% based on the carrier, to decompose the gas. The reaction is controlled to 100–250°C. The carrier is preferably Al₂O₃, TiO₂, active C, etc. Compounds, such as trichloroethylene, etc., of boiling point < 150°C, are removed from the gas, making it harmless.

Highly Dispersed Platinum Catalyst

TANAKA KIKINZOKU KOGYO K.K. *Japanese Appl.* 7/8,807
Highly dispersed Pt catalyst is manufactured by reducing Pt ion using Pt dinitro diamino nitrate solution and ethanol as reducing agents. The catalyst is manufactured without using surfactant, so the process is simple and the cost is low.

Preparation of Cyclopentyltrichlorosilane

SHINETSU CHEM. IND. CO. LTD. *Japanese Appl.* 7/10,887
Cyclopentyltrichlorosilane is prepared by reacting cyclopentene with trichlorosilane in the presence of a Pt catalyst which includes H₂PtCl₆·6H₂O, Pt/C, etc., while introducing gaseous inorganic oxygen compounds into the reaction system. When solvent is used, hydrocarbon or chlorinated solvent is used in 0.1–100 times as much as the raw material cyclopentene. Cyclopentyltrichlorosilane, which is useful as high activity base material for organic Si compound and compounds with groups of organic Si, is prepared in markedly high yield > 90%.

Simultaneous NO_x Reduction and CO Oxidation

GAZ DE FRANCE *French Appl.* 2,707,075
A catalyst, for the reduction of NO_x to N₂ by CH₄ or any mixture containing CH₄ (such as natural gas) in an oxidising atmosphere, comprises a MFI-type zeolite exchanged with 0.3–2.0% of Pd, relative to total catalyst weight. Simultaneous oxidation of CO to CO₂ and CH₄ to CO₂ also occur. The catalyst can be used in an oxidising medium with CH₄, natural gas or petrol in the presence of H₂O.

HOMOGENEOUS CATALYSIS

Production of Thiophene Compounds

SUMITOMO CHEM. CO. LTD. *European Appl.* 633,263A
Production of thiophene compounds from alkyldiene derivatives comprises catalytically reducing them with H₂ in a solvent mixture of at least one alcohol and H₂O in the presence of a Pd catalyst soluble in organic solvents. Flocculent and adsorbent, to flocculate the Pd catalyst which is removed, are optionally added. High yields of thiophene can be obtained.

Isomerisation of External Olefins

INST. FRANCAIS DU PETROLE *European Appls.* 636,676–77A
Isomerisation of external olefins to more highly substituted and/or internal olefins in diolefin-free or diolefin-containing charge is carried out over a Pd containing catalyst pretreated with a solution of a S compound to give 0.05–10 wt.% S. The catalyst is then activated by treatment in a neutral or reducing atmosphere at 20–300°C, 1–50 bar and 50–600 volume of gas per volume of catalyst per hour and H₂ at 20–200°C and 1–50 bar. The catalyst gives 100% removal of diolefin and higher conversion of 1-olefins than untreated catalyst.

Process for Hydrosulphination of Olefins

HOECHST A.G.

European Appl. 641,755A

Hydrosulphination of olefins is carried out in the presence of a Pd(II) salt, such as PdCl₂, PdBr₂, etc., and at a temperature between the ceiling temperature of the SO₂ olefin copolymer system and 160°C. The sulphonic and sulphonic acids produced are useful as precursors for plant protection agents and pharmaceuticals, and as intermediates for reactive dyestuffs.

Acetic Acid Production

BP CHEM. LTD.

European Appl. 643,034A

Acetic acid production comprises carbonylation of MeOH, or a reactive derivative, using CO in a liquid reaction mixture containing acetic acid, an Ir catalyst soluble in H₂O, such as IrCl₃, IrI₃, etc., MeI, H₂O, methyl acetate, and Ru and/or Os as promoter, such as Os(III) chloride hydrate, OsO₄, etc., Ru(III)- and Ru(IV) chlorides, etc. Increased reaction rates of $\geq 96\%$ higher than prior methods, are obtained at relatively low CO partial pressures. When Ru is the promoter the volatility of Ir catalysts may be reduced, allowing operation at reduced Ir levels, which may reduce by-product formation.

Preparation of N-Vinyl Compounds

BASF A.G.

European Appl. 646,571A

N-vinyl compounds are prepared from NH compounds using acetylene at 50–250°C and 1–30 bar in the presence of a Pt group metal compound as catalyst. The acetylene is used with an inert gas in a volume ratio of 6:1–0.5:1. N-vinyl compounds can contain H, 1–20C alkyl, 5–8C cycloalkyl, 7–18C aralkyl or 6–18C aryl, or a saturated, unsaturated or aromatic heterocyclic ring, etc. The vinyl compounds can be used to produce polymers for clarifying liquids, film-formers in hair treating agents or in detergents. The process gives an improved space-time yield and better control of acetylene conversion.

Modified Pyrimidine Derivatives

UNIV. WASHINGTON STATE RES. FOUND.

World Appl. 94/29,279A

Modified pyrimidine derivatives for production of nucleic acid antibodies are prepared by reacting pyrimidine starting material with tetraalkyl Sn compound in the presence of a Pd_L catalyst, where L may be PPh₃, and isolating the modified pyrimidine. The catalyst is prepared in a solution of Pd(OAc)₂ and PPh₃ in a molar ratio of 3, in THF, MeCN, oxane, acetone, tetrahydropyran or morpholine. The catalyst improves yields of vinyl and aryl coupling products. The antibodies may have altered structures with increased capacity to bind target molecules.

Ethylenically Unsaturated Compounds

SHELL CANADA LTD.

World Appl. 95/5,354A

Hydroformylation of ethylenically unsaturated compounds by reaction with CO and H₂ uses a catalyst of Pt group metal cations, anions other than halide and bidentate ligands. Improved conversion rate, linearity of products and suppression of paraffins are obtained even at moderate temperature. Aldehydes and/or alcohols, such as propanol, are produced.

New Polyethers

EASTMAN CHEM. CO.

U.S. Patent 5,393,867

New polyethers, used in the production of surfactants are prepared by polymerising 3,4-epoxy-1-butene in the presence of a Pd(0) catalyst and nucleophilic initiator, or at –40 to +60°C in the presence of a Pd(0) catalyst, 2–4 trihydrocarbyl-phosphine ligands and an organic hydroxyl initiator. Polymers containing polyether residues can be produced.

Aryl-Aliphatic Carboxylic Acid Derivatives

ALBEMARLE CORP.

U.S. Patent 5,399,747

The preparation of aryl substituted aliphatic carboxylic acid derivatives comprises treating an aryl substituted aliphatic ether or thio ether and H₂O with CO at a pressure of ≥ 1 atm and 25–200°C in the presence of Pd(0) catalyst or a Pd compound where Pd has valence 1 or 2, mixed with a Cu compound, and at least one acid-stable ligand. Ibuprofen can be produced economically.

Chlorobutene Carbonylation Catalyst

DSM N.V.

U.S. Patent 5,399,753

A process for the preparation of 3-pentenoyl chloride (3PC) comprises treating the still heels obtained by the distillation of 3PC with a reaction mixture containing Pd catalyst residue with concentrated HCl acid at 100–120°C. The above products are combined with chlorobutene to form an organic phase and a HCl acid phase containing the Pd catalyst residue; the latter is combined with chlorobutene and distilled to an azeotropic mixture in a CO atmosphere at 70–75°C until only a chlorobutene phase remains, forming crotlyl Pd chloride, heating and recovering 3PC by distillation. Pd catalyst is recovered, recycled and re-used.

Fluorine-Containing Surface Active Agent

SHINETSU CHEM. IND. CO. LTD.

Japanese Appl. 71/793

Preparation of the F-containing surface active agent comprises batch-mixing organic Si compound with polyether compound in aprotic solvent in the presence of Pt-based catalyst followed by hydrosilylation. The agents are prepared without side reaction and with high reaction efficiency in a short period of time.

Production of Carbonyl Compounds

TOSOH CORP.

Japanese Appl. 7/10,797

Carbonyl compounds are produced quickly and efficiently from cyclic or straight chain olefins using a catalyst containing a Pd compound and a polyoxoanion group compound. At least a part of the counter-cation of the polyoxoanion group compound is greater than selected from Ni, Mn, La, Zn and Ce.

Production of Intermediates for Vitamins

RHONE POULENC NUTRITION ANIMALE

French Appl. 2,706,452

Production of intermediates for synthesis of vitamins A and E and carotenoids comprises: reacting an allylic compound (1) with a vinyl halide compound in a polar solvent in the presence of a base and Pd(OAc)₂, PdCl₂ or a Pd phosphine complex catalyst, and treating the product with a protonic acid. The base may be an alkali metal carbonate or acetate, or a Ag or Tl salt. (1) is vinyl- β -ionol, methylbutenol, linalool, etc.

Selective Oxidation of Organic Compounds

BAYER A.G. *German Appl.* 4,331,671
The selective oxidation of organic compounds by O₂ is performed in the presence of a Pd and Cu catalyst and CO. The process is useful for oxidising linear and cyclic 2-8C olefins to epoxides; aromatics to phenols, etc. With H₂O additions, olefins or acids give glycols or glycol esters; and cyclohexanone with NH₃ gives cyclohexanone oxime and/or ε-caprolactam; and linear ketones with NH₃ give azines.

FUEL CELLS

Solid Polymer Electrolyte for Fuel Cell

TANAKA KIKINZOKU KOGYO K.K. *European Appl.* 631,337A
A solid polymer electrolyte (SPE) composition comprises a solid polymer cation or anion exchange resin and 0.01-80 wt.% metal catalyst. The catalyst is Pt, Pd, Rh, Ir or Au. SPE is used as a fuel cell membrane. It produces H₂O and also retains the H₂O so that it has high ionic conductivity. The membrane depresses the crossover which improves cell performance.

Steam-Reforming Catalyst for Fuel Cells

TONEN CORP. *Japanese Appl.* 6/339,633
A steam-reforming catalyst for hydrocarbons for fuel cells contains a catalytically active component of a Pt group element in 0.1-10 wt.% with respect to the total amount of catalyst and with a specific surface area of 1-20 m²/g. The fuel cell is preferably molten carbonate and the fuel of LNG, LPG, etc. is reformed directly inside. Contamination by alkali carbonate via a gas phase route is inhibited and catalyst has longer life.

CHEMICAL TECHNOLOGY

Ruthenium Oxide Hydrate Powder

JAPAN ENERGY CORP. *Japanese Appl.* 6/345,441
Ru oxide hydrate powder is prepared by reducing a solution of Ru salts of valency > 4. An aqueous solution of Na₂RuO₄ and acetone were reacted, and uniform grain size powder was obtained. The powder is used to make thick film resistors. The uniform grain size RuO₂.nH₂O and RuO₂ powder is prepared with low cost materials, and without crushing apparatus.

ELECTRICAL AND ELECTRONIC ENGINEERING

Soft Magnetic Material Composition

READ RITE CORP. *European Appl.* 642,183A
A soft magnetic alloy contains 82.7-84.7 at.% Ni, 15.3-17.3% Fe and Rh in an at.% defined by Rh = 11.63 (Ni:Fe atomic ratio)-49.53 (1). Also claimed is a magnetoresistive read transducer with a magnetoresistive layer and at least one soft magnetic layer as above. Reduction of the corrosion rate of Ni-Fe alloy while maintaining low magnetostriction comprises simultaneous deposition of Ni, Fe and Rh in amounts defined by (1). The alloy has a high resistivity and has an improved signal:noise ratio.

Magnetic Recording Medium

VICTOR CO. OF JAPAN *Japanese Appl.* 6/309,647
A medium for high density recording comprises laminations on a glass substrate of a pre-coat film, an undercoat layer of Cr and a magnetic film of Co-Cr-Ta-Pt alloy. The medium has ≥ 2200 Oe of coercivity. The magnetic film has lower X-ray diffraction intensity of Cr(200) and Co(110) on each Cr crystal and Co crystal lattice plane than when both planes are oriented in parallel with the glass substrate plane.

Recording Medium with High Coercive Force

SHOWA DENKO K.K. *Japanese Appl.* 7/14,712
A magnetic recording medium consists of a non-magnetic base layer, a non-magnetic metal layer and a magnetic layer containing 60-80 at.% Co, 5-20 at.% Cr and 2-25 at.% Pd, and a protecting layer. The magnetic recording medium has high coercive force.

Photomagnetic Recording Medium for Rewritable Photomagnetic Disk

SANYO ELECTRIC CO. LTD. *Japanese Appl.* 7/21,601
The medium comprises a lamination of a substrate, a first magnetic layer of Pd/Co laminate film with coercivity Hc-1 and Curie temperature Tc-1, and a second magnetic layer of Pt/Co laminate film having coercivity Hc-2 and Curie temperature Tc-2, where Hc-1 > Hc-2 and Tc-1 < Tc-2. The medium is used for a rewritable photomagnetic disk and light modulation overwriting; it has high density write character.

Photomagnetic Recording Medium

TOSOH CORP. *Japanese Appl.* 7/29,230
The medium comprises magnetic-bonding lamination of an artificial lattice film made of a layer based on Co or Co-Ni alloy and a layer based on Pt, and rare earth transition metal amorphous alloy double layers with different Curie temperatures. The medium is suitable for write/read using a short wave laser beam and has 0.8 dB lower noise level.

MEDICAL USES

Age-Hardenable Gold Dental Alloy

NIPPON HASHITOKURYOKU K.K. *Japanese Appl.* 6/345,615
Au alloy comprises 68-80 wt.% Au, 15-30 wt.% unavoidable impurities, and preferably 0.1-1.0 wt.% Ir and 0.1-1.0 wt.% Ru. The alloy is solution annealed by heating to 650-700°C and cooled rapidly. The alloy has a hardness twice that of the initial hardness.

Use of Gold-Palladium Alloy for Dentistry

DEGUSSA A.G. *German Appl.* 4,324,738
High gold content Au-Pd alloy comprises 6-25 wt.% Pd, 0-12 wt.% Pt, 0-2 wt.% Ir, Rh and/or Ru, 0.7-5.8 wt.% Sn and remainder Au, for dental cast parts, which are optionally blended with ceramics. The alloys have improved corrosion resistance, good mechanical and high temperature stabilities, ductility, etc.

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