

FUEL CELLS

Paper Fuel Cell Electrodes

W. A. BARBER and N. T. WOODBERRY, *Electrochem. Technol.*, 1965, 3, (7-8), 194-198

Waterproofed and platinised acrylic paper forms large-area, uniform electrode sheets with performance in H_2-O_2 fuel cells equal to Pt black on a metal screen.

TEMPERATURE MEASUREMENT

Differences between the Thermodynamic Scale and the International Practical Scale of Temperature from 0°C to -183°C

C. R. BARBER and A. HORSFORD, *Metrologia*, 1965, 1, (3), 75-80

The thermodynamic scale was realised at 0 to -183°C using an He constant volume gas thermometer, against which Pt resistance thermometers were calibrated to relate accurately the resistance of pure Pt and the thermodynamic temperature. The IPTS, as defined by the Pt resistance thermometer using the Callendar-Van Dusen equation, errs by -0.036° at $-80^\circ C$,

$+0.023^\circ$ at $-150^\circ C$, estimated limit of error on these differences $\pm 0.006^\circ C$.

On the Accuracy of Temperature Measurement with the Platinum Resistance Thermometer

V. V. SYCHEV and N. I. GORBUNOVA, *Teplofiz. Vysokikh Temp.*, 1965, 3, (4), 632-637

Theoretical methods are developed which enable the limits of absolute error to be calculated in the range 0-600°C.

High Voltage Cathode Temperature Measurement

R. L. FORGACS, B. A. PARAFIN, and E. EICHEN, *Rev. Sci. Instrum.*, 1965, 36, (8), 1198-1203

The temperature measurement and control system of the high voltage cathode of an emission microscope operating to 1300°C, 50 kV consists of a Pt:10% Rh-Pt thermocouple, a recorder, controller and power amplifier. The voltage is $\sim 10^9$ greater than the permissible thermocouple voltage error but this has negligible effect on the indicated temperature. The thermocouple unit is enclosed in a shield at cathode potential. Accuracy is $\pm 5^\circ C$.

NEW PATENTS

METALS AND ALLOYS

Palladium-Silver Alloys

E. I. DU PONT DE NEMOURS & CO
British Patent 1,004,652

Pd-Ag alloys in powder form are produced by preparing a solution containing Ag and Pd nitrates and precipitating a finely divided alloy of Pd-Ag by the addition of a reducing agent, e.g. H_3PO_2 , which reduces both metals.

Noble Metal Alloy Having High Specific Electrical Resistance

DEUTSCHE GOLD- UND SILBER-SCHNEIDANSTALT
British Patent 1,005,292

An alloy of high specific electrical resistance consists of 18-75 wt. % Au, 20-75 wt. % Pd, 2-15 wt. % Fe and 0.4-5 wt. % of one or more of Al, B, Ga or In, any balance being unavoidable impurities.

Ruthenium Alloy of Improved Workability

THE INTERNATIONAL NICKEL CO.
U.S. Patent 3,194,657

The workability of Ru metal is improved by permeating the molten Ru in a non-oxidising medium with 0.005-5 wt. % Zn, Bi, Cd, Ba, Ge, Hf or a lanthanide, maintaining the metal in a molten

state until it is substantially quiescent and free from gas evolution and then allowing it to solidify.

Self-lubricating Bearing

THE BOEING CO.
U.S. Patent 3,199,934

The running surface is a composite of 7.5-22.5 % Ag, 7.5-22.5 % Pt, 26.25-78.25 % MoS_2 , 5.45-22.75 % PbO and 0.4-1.2 % SiO_2 .

Palladium Alloy Permeable to Hydrogen

NIPPON JUNSUISO K.K. *German Patent* 1,199,242
The alloy consists of Pd with 2-40% of at least one Group IB element and at least 0.1-20% of other Group VIII elements, e.g. Pd with 20-30% Ag, 3-10% Au and 0.1-5% Ru.

ELECTROCHEMISTRY

Use of Platinum Anodes in Electrolytic Processes

MONSANTO CO. *U.S. Patent* 3,193,475
A Pt anode and a Hg cathode have been used in various reactions involving the electrolytic

coupling of olefines, hydrodimerisation of olefinic compounds, electrolysis of acrylamide and the production of adiponitrile.

Electrode for Electrolytic Processes

KREBS & CO. *French Patent* 1,393,029

Electrode, particularly for electrolytic Cl_2 production, consists of a suitable conductor or semi-conductor coated with a resistant-surface, e.g. Ti "activated" by Pt or Ir.

ELECTRODEPOSITION AND SURFACE COATINGS

Chemical Plating Process

SPERRY GYROSCOPE CO. LTD.

British Patent 1,003,575

Articles are chemically plated with metals by immersing the hot article in a solution of PdCl_2 in a mixture of about 40% acetone and methyl salicylate, butyl formate or acetaldehyde, drying it and then immersing in the usual chemical plating bath.

Plating Bath and Process for Platinum Plating

ASAHI KASEI KOGYO KABUSHIKI KAISHA

British Patent 1,003,634

Pt is electrodeposited by using a plating bath formed by dissolving NH_4 , K or Na sulphamate in an aqueous NH_3 solution of dinitrodiamino platonic acid and carrying out the plating at about 3.0 V and pH 6.5-8.0.

Protection of Metallic Surfaces

JOHNSON MATTHEY & CO. LTD.

British Patent 1,003,848

Tarnishable and/or oxidisable metal surfaces are protected against external corrosive influences by electrodepositing a $15-50 \times 10^{-6}$ inch layer of Pt and then electrodepositing $15-500 \times 10^{-6}$ inch thick layer of Rh.

Protection of Metallic Surfaces

JOHNSON, MATTHEY & CO. LTD.

British Patent 1,003,849

Oxidisable metal surfaces are protected against corrosion and tarnishing by first electrodepositing on them a $15-50 \times 10^{-6}$ inch thick film of Pt and then $15-500 \times 10^{-6}$ inch thick Au film.

Process for Electrodeposition of a Platinum Coating and Electrolyte for the Application of Said Process

COMPTOIR LYON-ALLEMAND, LOUYOT & CIE.

British Patent 1,005,478

Up to 15μ Pt layers are electrodeposited on non-ferrous metals, e.g. Au, Ag, Ni, Cu, various alloys, etc., by using as electrolyte an aqueous solution containing 5-100 g/l fluoboric acid and 5-60 g/l Pt (II) salt or Pt (II) diaminodinitrite.

CATALYSIS

Production of Carbonyl Compounds

THE DISTILLERS CO. LTD. *British Patent* 1,001,539

Carbonyl compounds are produced by reacting a 5-8C olefine for 2 min to 5 h at 20-120°C with a mixture of a Pt group metal compound, CuCl_2 , H_2O and a Cu salt of mono- or di-halo-acetic acid.

Catalytic Oxidation of Waste Gases

UNIVERSAL OIL PRODUCTS CO.

British Patent 1,002,337

A waste gas stream containing combustible materials is purified by passing it, together with O_2 , at a suitable temperature through a bed of particulate material consisting of Al_2O_3 supporting 0.05-0.2 wt. % Pt and comprising 70-92 wt. % Al_2O_3 particles composited with 0.013-0.1 wt. % Pt and 8-30 wt. % evenly dispersed Al_2O_3 particles composited with 0.1-5.0 wt. % Pt.

Preparation of Alcohols

SHELL INTERNATIONALE RESEARCH MIJ. N.V.

British Patent 1,002,429

Alcohols are produced by reacting an olefinic compound with H_2 and CO in an alkaline medium at 150-190°C, 400-700 psig and in the presence of a catalyst comprising a complex of Co, Ru or Rh with at least one P-containing ligand.

Conversion Process and Catalysts

UNION OIL COMPANY OF CALIFORNIA

British Patents 1,002,921-2

A catalyst system for the hydrocracking of high-boiling mineral oil fractions comprises (i) a partially decationised zeolitic aluminosilicate molecular sieve supporting a minor amount of Group VI B or VIII metal, e.g. 0.05-2 wt. % Pt or Pd and (ii) a relatively inert, powdered, refractory material of greater particle size than the crystal size of the molecular sieve.

3, 4 - Dihydro-benzoxazinones-(1,3,2) and Processes for the Production of Such Compounds

E. MERCK A.G.

British Patent 1,003,113

3,4-Dihydro-benzoxazinone-(1,3,2) is produced by reacting 2-hydroxy-benzyl halide with a salt of cyanic acid and treating the product with H_2 in the presence of supported Pd or PtO catalyst. See also 1,001,114.

Separation by Selective Catalytic Chemical Reactive Process

SOCONY MOBIL OIL CO. INC.

British Patent 1,003,251

A catalyst for the continuous separation of chemical compounds of different molecular shape but similar chemical reactivity, consists of crystalline aluminosilicate zeolite of 5-13 Å pore size activated by Ag or Pt group metal.

Selective Hydrocracking

SOCONY MOBIL OIL CO. INC.

British Patent 1,003,252

A catalyst for the selective hydrocracking of hydrocarbons consists of 5–13 Å crystalline aluminosilicate zeolite whose pores support catalytically active Ag, Ni, Co or Pt group metal.

Production of Organic Esters

THE DISTILLERS CO. LTD. *British Patent* 1,003,347

Organic esters are produced by reacting an olefine and an organic carboxylic acid at 50–250°C, 1–50 atm. and in the presence of a supported Pt group metal catalyst.

Tetracycline Derivatives

AMERICAN CYANAMID CO. *British Patent* 1,003,474

6-Deoxy or 6-deoxy-6-demethyl tetracycline derivative containing a 7 and/or 9 amino group is prepared by reacting the corresponding 7 and/or 9 nitro derivative with 1 mole carbonyl compound and H₂ in the presence of a supported Pt or Pd catalyst.

Producing Vinyl Esters of Fatty Acids

ASAHI KASEI KOGYO KABUSHIKI KAISHA

British Patent 1,003,499

Vinyl esters of fatty acids are produced by flowing a mixture of an O₂-containing gas, a saturated fatty acid and C₂H₄, at 50–300°C, over a catalyst consisting of an inert carrier supporting (i) at least one metal or oxide selected from Cu, Ag, Zn, Cd, Sn, Pb, Cr, Mo, W, Fe, Co or Ni and (ii) at least one metal selected from Pd, Pt, Rh, Ru or Ir.

Substituted Amines and Processes for Their Production

E. MERCK A.G.

British Patent 1,003,687

New techniques for the production of substituted amines involve a hydrogenolysis stage in the presence of supported or unsupported noble metal catalysts, e.g. Pt, PtO, Pd black, etc.

Hydrocarbon Reforming and Catalyst therefor

IMPERIAL CHEMICAL INDUSTRIES LTD.

British Patent 1,003,702

A catalyst suitable for steam reforming of hydrocarbons is produced by forming a mixture of an inert refractory oxide carrier, 1–10 wt.% alkali metal oxide or hydroxide and catalytic amount of Ni, Co, Ru, Rh, Pd, Os, Ir, Pt or their mixtures and heating at 600–900°C to increase its resistance to leaching with H₂O.

Process for Hydrogen Treatment of Hydrocarbon

GULF RESEARCH & DEVELOPMENT CO.

British Patent 1,003,715

Residual petroleum fractions are subjected to destructive hydrogenation by contacting them and

H₂ at 371–441°C and 35–280 kg/cm² with a catalyst consisting of activated Al₂O₃ supporting minor amounts of Pt, Pd or their oxides or sulphides.

Benzophenones and Derivatives thereof

F. HOFFMANN-LA ROCHE & CO. A.G.

British Patent 1,003,936

Benzophenone derivatives are produced by reacting a 2-halo-benzophenone with benzylamine and then dibenzylating the product by hydrogenolysis in the presence of a Pd metal catalyst.

Manufacture of Naphthalene Derivatives

IMPERIAL CHEMICAL INDUSTRIES LTD.

British Patent 1,005,024

The compounds A.CHOH.CH₂.NRR', where A is a naphthalene nucleus, R is H or Me and R' is 1–4C alkyl are produced by reducing A.CO.CH(OH)₂ in the presence of R"NRR' (where R" is H₂ or a hydroxy radical) and preferably by catalytic hydrogenation in the presence of Pt or Raney Ni.

See also 1,005,025–7.

Process for the Preparation of 2,6-Dichlorobenzonitrile

SHELL INTERNATIONALE RESEARCH MIJ. N.V.

British Patent 1,005,067

2,6-Dichlorobenzonitrile is produced by passing a mixture of substituted 2,6-dichlorobenzenes, NH₃ and O₂ or O₂-containing gas over a C supported Pd or Ni catalyst maintained at 150–500°C.

Oxidation of Mercurous Salts

IMPERIAL CHEMICAL INDUSTRIES LTD.

British Patent 1,005,658

Hg(I) salts are oxidised to Hg(II) salts by reacting them with O₂ in an acidic aqueous medium and in the presence of Ag, Au, Pt or Pd supported catalyst, e.g. on SiO₂.

Catalytic Conversion of Hydrocarbons

STANDARD OIL CO.

British Patent 1,006,291

Hydrocarbons are converted to lower boiling materials by contacting them at 200–2,000 psig and 400–1,000°F with H₂ in the presence of a catalyst composite comprising highly porous acid-treated Al₂O₃ or its mixture with a refractory metal oxide, 0.5–5 wt.% Ni, Co, Pt or Pd and 0.01–1 g atoms Group VA element/1 g atom Group VIII metal.

Dehydrogenation Catalysts

SINCLAIR RESEARCH INC. *U.S. Patent* 3,190,932

Methylcyclopentane is converted to C₆H₆ by passing it, together with molecular H₂, at 800–1,000°F and 150–400 p.s.i.g. in contact with a catalyst consisting of 0.01–2 wt.% Pt group metal, preferably Pt supported on activated Al₂O₃.

Reduction of Isonicotinic Acid Esters

PMC CORP. *U.S. Patent 3,192,220*

Lower alkyl esters of isonicotinic acid are reduced to the corresponding piperidine derivatives by reacting the ester with H_2 at 35–150°C and up to 500 p.s.i.g. in the presence of a supported catalyst providing 0.005–2 g Pd/mole of the ester.

Improved Hydrocarbon Cracking Catalysts

SOCONY MOBIL OIL CO. INC.

U.S. Patent 3,193,491

Improved, solid, porous hydrocracking catalysts are produced by mixing aqueous solutions of Zr, Al and alkali metal silicate salts, allowing the mixture to set to a hydrosol, transforming it into a hydrogel and then combining it with a Group VI or VIII metal, oxide or sulphide, such as Pt, Pd or Mo.

Platinum Group Metal Hydrogenation Catalysts

MERCK & CO. INC. *U.S. Patent 3,193,550*

Reduced cephalosporin C derivatives are produced by reducing an alkyl ester derivative of N-acyl cephalosporin C in a solvent medium with H_2 at 25–100°C, 1000–30,000 p.s.i. and in the presence of a Pt metal catalyst.

Dehalogenated Platinum-on-Alumina Reforming Catalysts

AIR PRODUCTS & CHEMICALS INC.

U.S. Patent 3,194,755

Improved catalysts are produced by forming sorptive Al_2O_3 particles free from Cl_2 , impregnating them with aqueous H_2PtCl_6 acid solution to produce a Pt content of 0.1–2 wt.%, treating them with gaseous H_2S at 60–140°F and then with NH_3 , washing to reduce Cl_2 content and drying.

Rhodium Hydrogenation Catalyst

ABBOTT LABORATORIES INC. *U.S. Patent 3,194,839*

Aromatic amino compounds are produced by hydrogenating the corresponding nitro compounds at 25–150°C, 1 atm to 150 p.s.i. and in the presence of an almost spent Rh catalyst.

Regeneration of Platinised Carbon Catalysts

HALCON INTERNATIONAL INC.

U.S. Patent 3,194,843

Spent Pt catalysts on C supports are regenerated by leaching with H_2O until the leachings are free from organic material and then separating the regenerated material.

Olefin Hydrogenation in Alkyl Benzene Production

ESSO RESEARCH & ENGINEERING CO.

U.S. Patent 3,196,174

In the production of biodegradable perhydro-bis-(isoprenyl)-alkyl aryl sulphonates, the reduction of the olefines is accomplished using Pd/C.

Reductive Cyclohexylamine Production

ABBOTT LABORATORIES *U.S. Patent 3,196,179*

Aniline is reduced to cyclohexylamine at elevated temperature and pressure over a Rh catalyst, preferably supported elemental Rh.

Catalytic 2-Oximinoindane Reduction

CUBA CORP. *U.S. Patent 3,196,181*

The reduction of this compound to 2-aminoindane is catalysed by a non-pyrophoric supported Pd catalyst in the presence of H_2SO_4 and acetate ions.

Improving Front End Octane Rating

SOCONY MOBIL OIL CO. INC. *U.S. Patent 3,198,728*

Naphtha containing naphthenes and paraffins are treated first over a Pt group metal reforming catalyst and then over a non-noble metal sulphide dehydrogenating catalyst.

Loading Molecular Sieves with Catalysts

UNION CARBIDE CORP. *U.S. Patent 3,200,082*

Catalytic metals, including Au, Ag, and Pt metals, are more easily loaded on to zeolites in the form of their complexes with amines, e.g. tetramine platinumous chloride. See also 3,200,083.

Deoxytetracycline Production

CHAS. PFIZER & CO. INC. *U.S. Patent 3,200,149*

The hydrogenation of 6-deoxy-6-dimethyl-6-methylene-tetracyclines over Pt metal catalysts produces the 6-epi-6-deoxytetracyclines.

Dehydrogenation Catalysts

CENTRE NATIONAL DE LA RECHERCHE SCIENTIFIQUE

French Patent 1,391,114

Alkylanthraquinones are dehydrogenated to alkenylanthraquinones by contacting them at 400–600°C and in an inert gas atmosphere with a Pt, Pd or their oxides catalyst.

Production of Formyl Carboxylic Acids

SOC. D'ELECTROCHIMIE, D'ELECTROMETALLURGIE ET DES ACIERIES ELECTRIQUES D'UGINE

French Patent 1,394,863

These γ -formyl carboxylic acids are produced by reduction of the corresponding unsaturated acids over a Pd catalyst.

Catalytic Olefine Oxidation Process

FARBWERKE HOECHST A.G.

German Patent 1,197,071

The oxidation of olefines to aldehydes, ketones or acids over a supported $PdCl_2/CuCl_2$ catalyst system is effected in a plurality of catalyst beds in sequence using H_2O injection for cooling.

Olefin Purification Catalysts

GIRDLER-SUDCHEMIE KATALYSATOR G.m.b.H.

German Patent 1,198,809

Acetylenes and dienes are removed from an

olefine feedstock by selective hydrogenation over a catalyst comprising 0.027-0.09 wt.% Pd and 0.07-0.09 wt.% Cr on a support having pores not larger than 800 Å.

Production of Vinyl Esters

CONSORTIUM FÜR ELEKTROCHEMISCHE INDUSTRIE
G.m.b.H. *German Patent 1,198,813*

The production of vinyl esters from C_2H_4 and carboxylic acids in the presence of Pt metal catalysts is improved by cyclically varying the pressure.

FUEL CELLS

Fuel Cell Electrode

LEESONA CORP. *British Patents 1,001,523-5*

A fuel cell electrode consists of a porous hydrophobic polymer structure, the side thereof facing the gas or liquid feed being in intimate contact with a porous ion exchange resin film and the side facing the electrolyte being coated with Ag, Au, Ru, Rh, Pd, Os, Ir, Pt or some other catalytically active metal.

Carbon-supported Fuel Cell Catalyst Electrode

ESSO RESEARCH & ENGINEERING CO.
U.S. Patent 3,196,050

A C catalyst is produced by impregnating a C electrode with an aqueous solution of a catalyst metal, e.g. Pt, and then heating it at 600-900°F in a CO atmosphere to remove O_2 and then at 1000-1800°F in an H_2 atmosphere to reduce the metal.

Fuel Cell Oxygen Electrode

LEESONA CORP. *German Patent 1,198,430*

A porous electrode is made from an alloy of Pd with 0.25-10% Ag, especially 1.5% Ag.

ELECTRICAL AND ELECTRONIC ENGINEERING

Metal-Glass Resistance Material

BECKMAN INSTRUMENTS INC.
British Patent 1,002,791

A resistance device comprises a high temperature resistant, electrically non-conductive base having fired on it a resistance layer formed of 50-84 wt.% glass and 16-50 wt.% finely dispersed alloy of Ir with at least one metal selected from Au, Pd or Ag.

Metal-Glass Resistance Materials

BECKMAN INSTRUMENTS INC.
British Patent 1,002,793

A metal-glass resistance material designed to be fired on to a non-conductive base member consists of a finely divided mixture of 60-99 wt.% glass and 1-40 wt.% Ru or its alloy with Au, Pd, Pt or their mixtures.

Solion

GENERAL ELECTRIC CO. *British Patent 1,003,529*

An integrator assembly for solion devices comprises an envelope containing the electrolyte, a Pt metal anode, a pair of porous graphite cathodes placed on each side of the anode and connectors projecting beyond the envelope.

Methods of Manufacturing Semiconductor Devices

MULLARD LTD. *British Patent 1,004,048*

Good ohmic and p-n junctions are produced in semiconductor bodies consisting of a III-V or substituted III-V semiconductor compound by using alloys of 0.5-5 wt.% Pt and 95-99.5 wt.% Sn.

Electrical Capacitors

E. I. DU PONT DE NEMOURS & CO.

British Patent 1,004,653

A Pd-Ag alloy metallising composition, e.g. for stencil application, for use in capacitor electrode production, comprises 30-60% Pd-Ag alloy powder, which contains 95-60% Pd and 5-40% Ag, dispersed in 70-40% of an inert vehicle, preferably an organic vehicle.

Electrical Components

DAYSTROM INC. *British Patent 1,006,238*

A composite resistance unit is made by depositing on the non-conductive substrate a catalytic film of Ni, Fe, Co, Ru, Ph, Pd, Os, Ir or Pt, depositing a thin film of electrically resistive material, e.g. Ni and finely divided P, by means of a chemical reduction process and then heat-treating the unit at 250-650°F for several hours to modify its temperature coefficient of resistance and to stabilise its resistance value.

Electrical Conductor

IMPERIAL CHEMICAL INDUSTRIES LTD.
British Patent 1,006,394

An electrical conductor comprises a casing of Ti or its alloy with up to 14 wt.% Zr or up to 5 wt.% Pt, Rh or Ir, coated on its internal surface with an adherent film of Cu or Ni and a tightly fitting Cu, Al, Fe or steel core fixed by means of a fusible metal or metal alloy solder.

Electric Discharge Tube Grid

NORTH AMERICAN PHILIPS CO.
U.S. Patent 3,200,284

The grid has a base of Mo, an intermediate layer of Fe, Co or Ni and an outer layer of Pt.

Condenser Dielectric Electrode

ELECTRO-MATERIALS CORP.
French Patent 1,393,294

An electrode is produced from a paste of crude ceramic dielectric mixed with a high conductivity metal in powder form which represents 20-80% of the solids. Suitable metals include Ag, Pd, Pt, Rh and Ir.