

A review of refractory oxides suitable for the protection of thermocouples under the conditions of this application is given.

Temperature Error Associated with Imbedded Thermocouples

A. R. THOMAS, B. SCHURIN and J. C. MORRIS, *Rev. Sci. Instruments*, 1958, **29**, (11), 1045-1046

The error in surface temperature of a thin plate in a vacuum as measured by an embedded Pt/10%Rh-Pt thermocouple is investigated experimentally and theoretically.

Immersion Thermocouple Practice

H. V. SCHUBERT, *Iron Steel Eng.*, 1959, **36**, (1), 91-97

A detailed description is given of the basic design, construction, application and maintenance of an immersion thermocouple assembly. The subjects are dealt with from a purely practical viewpoint. Full details are given for taking a temperature reading and analysing whether or not the result obtained is acceptable. Great importance is attached to thermocouple maintenance and details are given for checking the couple together with probable causes of any defects.

NEW PATENTS

Manufacture of Sulphanilamide

F. HOFFMANN-LA ROCHE & CO. A.G. *British Patent* 804,036

A noble metal hydrogenation catalyst is used in a process for the manufacture of 5-sulphanilamido-3,4-dimethyl-isoxazole by reduction of P, P¹ [bis [3,4-dimethyl-isoxazolyl-(5)]-amino-sulphonyl]-azobenzene in a highly acidic liquid medium. The catalyst may be Pd-C or PtO₂.

Uranium Compounds

UNITED KINGDOM ATOMIC ENERGY AUTHORITY
British Patent 804,062

Uranium salts of deuterated higher aliphatic acids are made by treating a uranium salt of a higher aliphatic acid with deuterium in the presence of a hydrogenation catalyst, e.g. Pt.

Alloy

ENGELHARD INDUSTRIES INC. *British Patent* 804,404

A high resistivity high strength alloy suitable for electric resistance wires consists of 20-62% Pd (preferably 30-50%), 28-70% Au and more than 5% up to 18% Fe (preferably 7-12%). The resistivity of the heat-aged alloy is 450-1200 ohms/mil foot. Pt or Rh (up to 10%) may be included.

New Indole Derivatives

SOC. DES USINES CHIMIQUES RHONE-POULENC
British Patent 804,786

Adam's PtO₂ is used as catalyst in a process involving catalytic hydrogenation for the manufacture of specified indole derivatives.

Catalysts

F. HOFFMANN-LA ROCHE & CO. A.G. *British Patent* 804,788

A catalyst is composed of Pd sponge deposited on a substrate of a mixture of Zn(OH)₂ and Fe(OH)₃ and/or ZnCO₃ and FeCO₃ in the ratio

2-9 gram atoms of Pd to 1 of Zn and 1 of Fe. The catalyst is made by treating an aqueous solution of the component ions with an alkaline material and a reducing agent.

Production of Motor Gasoline

THE BRITISH PETROLEUM CO. LTD. *British Patent* 805,045

A high octane gasoline is made by thermally reforming a mixture of a light straight-run gasoline and a "platinum reformat".

Purification of Benzene

THE BRITISH PETROLEUM CO. LTD. *British Patent* 805,050

Benzene is separated from mixtures of benzene and non-aromatic hydrocarbons which form azeotropes with the benzene by catalytically reforming the mixture in the presence of hydrogen at 850-1050°F and pressure of 50 to 1000 p.s.i.g. followed by fractional distillation of the product to obtain a benzene fraction. A Pt-Al₂O₃ catalyst is used.

Preparation of Hydrogen Peroxide

COLUMBIA-SOUTHERN CHEMICAL CORP. *British Patent* 805,101

Hydrogen peroxide is prepared by successively hydrogenating a quinone in a solvent medium and in the presence of a catalyst, e.g. Pd-Al₂O₃, oxidising the product to regenerate the quinone and generate H₂O₂, extracting the peroxide at a temperature below the hydrogenation temperature and heating the solvent from the extraction to the hydrogenation temperature.

Hydroforming of Naphthas

ESSO RESEARCH & ENGINEERING CO. *British Patent* 805,111

A gasoline fraction is produced by hydroforming a naphtha fraction in the presence of hydrogen

and a catalyst and passing the total effluent without cooling to a dealkylation zone, where it is heated, the total normally liquid product recovered thereby having a greater vol.% of distillate boiling at 230°F than could be obtained from the effluent from the hydroforming zone. A Pt hydroforming catalyst is used.

Catalytic Reduction of Aromatic Dinitro Compounds

GENERAL ANILINE & FILM CORP. *British Patent* 805,249

Reference is made to the use of Pt group metal catalysts, preferably Pd or Pt, supported or unsupported, in a novel process for the catalytic hydrogen reduction of aromatic dinitro compounds to form the corresponding diamines.

Fluorination Catalyst

THE DOW CHEMICAL CO. *British Patent* 805,503

A fluorination catalyst is made by saturating a porous activated γ - Al_2O_3 in an aqueous solution of a halide of, *inter alia*, Pd to incorporate 0.1–10% by weight based on the metal of the halide, and then passing a stream of anhydrous gaseous HF into contact with the Al_2O_3 until it becomes dry.

Preparation of Liquid Hydrocarbon Mixtures

N.V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ *British Patent* 805,530

Liquid hydrocarbon mixtures for use in motor fuels are produced by catalytically reforming a straight-run gasoline or fraction thereof with a Pt catalyst on an acidic carrier and distilling the reformate to separate one or more heavy fractions boiling above 160°C at least one of which is thermally reformed. A catalyst containing 0.76% Pt, 0.22% Cl and 0.38% F, all by wt. on an Al_2O_3 support is referred to.

Phosphate Derivatives of Steroids

MERCK & CO. INC. *British Patent* 805,828

Phosphate derivatives of steroids of given general formula are hydrogenated in the presence of a catalyst and a tertiary amine to form the corresponding tertiary amine salt of 3-ethylene-deoxy-11-(keto or β -hydroxy)- Δ^5 -pregnenc-17 α ,21-diol-20-one-21-phosphate. The catalyst used is pre-reduced PtO_2 .

Preparation of Cyclopentene

ESSO RESEARCH & ENGINEERING CO. *British Patent* 805,865

Cyclopentene is prepared by hydrogenating cyclopentadiene dimer in the presence of a PtO_2 catalyst at 25–150°C to give dihydro-dicyclo-pentadiene and cracking the latter at 350–600°C. Hydrogen pressure used is 50–200 p.s.i.g.

Preparation of Sulphur Oxyfluorides

E. I. DU PONT DE NEMOURS & CO. *British Patent* 805,874

A reactor tube of Pt or Pt-lined Ni is used in the preparation of sulphuryl and/or thionyl fluoride by pyrolysis of a sulphur oxy-fluoride, having in its molecule hexavalent S and two F atoms and of higher molecular weight than the desired end product, at between 200°C and 1800°C.

Spot-welded Joints

SIGNAL STAT CORP. *British Patent* 805,871

A high carbon spring steel member is welded to a cold rolled mild steel member by forming a first spot-weld between the superimposed members, placing against the spring steel member a piece of Ni, Pt or an alloy of Ni or Pt and spot-welding this piece to the members by a second spot-weld adjacent the first weld.

Hydrogenation of Saccharides

ENGELHARD INDUSTRIES INC. *British Patent* 806,236

A polyhydric alcohol is made by treating an aqueous solution of a saccharide with hydrogen in the presence of a Ru-containing catalyst. The catalyst consists of Ru metal or RuO_2 or Ru mixed with another Pt group metal and preferably carried on a support, e.g. C or Al_2O_3 .

New Basic Steroid Derivatives

F. HOFFMANN-LA ROCHE & CO. A.G. *British Patent* 806,581

An Adam's PtO_2 hydrogenation catalyst and a Pd-C catalyst are used in a process for the manufacture of 17 [piperidyl-(2¹)-methyl]-androstane-diol-(3 β , 17 β) and 1¹-methyl and 1¹-ethyl derivatives thereof.

Preparing Esters of Unsaturated Carboxylic Acids

E. I. DU PONT DE NEMOURS & CO. *British Patent* 806,800

Esters of unsaturated carboxylic acids are prepared by reacting allene and a monohydric alkanol or cycloalkanol with CO at a pressure of at least 100 atm and a temperature of 120–250°C in contact with at least 1% (by wt of the allene) of a Ru carbonyl catalyst.

Printed Circuits

BRITISH INSULATED CALLENDER'S CABLES LTD. ET AL. *British Patent* 806,977

A printed circuit is made by mechanically roughening the surface of an insulating base of resin-bonded fibrous material, activating part or all of the surface by treatment with a solution containing a soluble salt of a noble metal, treating the activated surface with a reducing agent to precipitate the noble metal and finally treating the base with a hot solution containing a soluble Ni salt, hypophosphite ions and a buffer under

such conditions that the Ni (containing Ni phosphide) is deposited on the surface. An aqueous solution of PdCl_2 of a strength of 100-500 p.p.m. is preferably used.

Manufacture of Novel Ketones

F. HOFFMANN-LA ROCHE & CO. A.G. *British Patent* 807,009

Novel ketones of specified general formula are prepared by catalytically hydrogenating a compound of given formula under conditions of temperature and pressure allowing of hydrogenation of the carbon-to-carbon double bond or bonds in the nuclear substituent or substituents of the compound. The catalyst employed is a Pd catalyst, preferably a reduced Pd- CaCO_3 catalyst. A temperature of 20-30°C and a hydrogen pressure of not over 30 p.s.i.g. is employed.

Reforming Catalysts

W. R. GRACE & CO. *British Patent* 807,272

A gasoline reforming catalyst is made by impregnating a base, formed of silica gel partially deactivated to a surface area of 400-625 sq.m/g by heat treatment and modified by the addition before, during or after deactivation, of 0.1-1 wt.% Al_2O_3 , with an aqueous solution of a Pt or Pd compound in amount to form a final catalyst containing 0.1-1 wt.% of Pt or Pd, drying and converting the adsorbed compound to the metal. An aqueous solution of chlorplatinic acid may be used.

Purification of Commercial Hydrogen

ENGELHARD INDUSTRIES INC. *British Patent* 807,584

Carbon monoxide and/or CO_2 present in commercial hydrogen is/are removed by contacting the hydrogen with a Rh or Ru catalyst (0.01-2% of the metal), supported on a water-insoluble dehydrated metal oxide, e.g. Al_2O_3 at a reaction temperature of at least 120°C until the CO and CO_2 content is completely converted to methane. The process may also be used expressly for the production of methane.

New Indole Derivatives

IMPERIAL CHEMICAL INDUSTRIES LTD. *British Patent* 807,877

New indole compounds of given formula are made by de-alkylating the corresponding 5-alkyloxy-indole compounds of given formula by means of hydrogen in the presence of a hydrogenation catalyst, preferably Pd-C or Pd- BaSO_4 .

Novel Pyridones

ROCHE PRODUCTS LTD. *British Patent* 808,045

The use of a Pd-C hydrogenation catalyst is referred to in the manufacture of 4-methyl-5-alkyl-pyridone (2).

Conversion of Ethanolamine into Ethylene Diamine etc.

THE DOW CHEMICAL CO. *British Patent* 808,114

Pt- or Pd- Al_2O_3 or finely divided SiO_2 may be used in the conversion of ethanolamine into ethylene diamine, piperazine and diethylene triamine by passing a stream of ethanolamine and NH_3 under pressure over a fixed bed of the catalyst maintained at elevated temperature.

Use of Palladium Alloys for Electric Interrupter Contacts

E. DURRWACHTER *German Patent* 1,022,385

Covers a modification of the alloy of 0.1-25% Pb and/or 0.1-20% Sn, remainder Pd, see German Patent 961,762, for use as an electric interrupter contact in which, in place of, or in addition to, Pb and/or Sn, one or more of the metals In, Ga, Tl, or Ge is included, the total content of Pb, Sn and addition metals being from 0.1-20%, preferably 5-15%.

Apparatus for the Automatic Firing of Gas Burners

W. C. HERAEUS G.m.b.H. *German Patent* 1,028,055

Refers to the use of a Pt or other Pt metal catalyst, in the form of wire gauze, in an apparatus for the automatic firing of gas burners, e.g. propane or butane gas burners.

Separation of Metal Salts from Aqueous Solutions

ROHM & HAAS G.m.b.H. *German Patent* 1,028,551

In the process of separating noble metal salts, e.g. Au and/or Pt salts, from aqueous solutions according to German Patent 965,635 in which the metal salts are contacted with the products resulting from the polymerisation of HCN, the products enriched with the salts are treated with a mixture of an excess of solvent, miscible with water, e.g. acetone and aqueous HCl.

Galvanic Deposition of Platinum on a Metal Object

N.V. PHILIPS GLOEILAMPENFABRIEKEN *German Patent* 1,033,478

In a method for the electrodeposition of Pt on a metal object using a Pt containing electrolyte from which Pt is deposited in the form of a layer of Pt black, which is sintered at about 1000°C and converted to Pt, the object is brought at least once, with the employment of a relatively high current density, into an electrolyte bath consisting of a solution of 150-300 g chlorplatinic acid (H_2PtCl_6) and 1-15 g of mercuric chloride (HgCl_2) per litre.

Production of Complex Platinum- and Palladium-Hydroxides

ATLANTIC REFINING CO. *German Patent* 1,033,646

Complex Pt- or Pd-hydroxides are produced by

passing a dilute aqueous solution of a Pt or Pd complex salt over an anion exchange resin, which is in the hydroxyl state.

Catalyst

HOUDRY PROCESS CORP. *U.S. Patent 2,857,336*

A hydrocarbon conversion catalyst is made by treating calcined activated γ - Al_2O_3 particles with acetic acid, washing with a limited amount of water to leave a significant amount of acid adsorbed on the particles, drying at less than 300°F and impregnating the particles with a Pt compound. Platinum is thereby distributed throughout the thickness of the Al_2O_3 particles in such manner as to resist migration to the surfaces thereof.

Preparation of Catalyst

E.I. DU PONT DE NEMOURS & CO. *U.S. Patent 2,857,337*

A hydrogenation catalyst is made by heating a solution of PdCl_2 in an alkali metal bicarbonate system (wt. ratio of bicarbonate to Pd being from 5 : 1 to 85 : 1) to 90 – 95°C in the presence of an inert carrier for at least 15 min.

Production of Cyclohexanone

ALLIED CHEMICAL CORP. *U.S. Patent 2,857,432*

Cyclohexanone is made by continuously passing hydrogen into a liquid body of a mixture of cyclohexanone and phenol, having dispersed in it powdered Pd catalyst at an elevated temperature so as to hydrogenate the phenol to cyclohexanone, continuously passing a gas through the body to remove cyclohexanone as vapour free of catalyst, to maintain the concentration at 30–80% by wt.

Reforming Process

THE M. W. KELLOGG CO. *U.S. Patent 2,860,102*

Discloses a novel reforming process in which a light hydrocarbon oil is contacted with a fluidised mass of finely divided Pt catalyst under reforming conditions and which includes regeneration of the contaminated catalyst.

Thermocouple

H. NISHIMURA *U.S. Patent 2,861,114*

A thermocouple consists of Pt and a Pt-Mo-W alloy containing 0.5–4% Mo and 3–6.5% W. The sum of the Mo and W is always less than 7%.

Reforming

SINCLAIR REFINING CO. *U.S. Patent 2,861,942*

A process of reforming olefin-free hydrocarbons of gasoline and naphtha boiling ranges in the presence of hydrogen and a Pt metal reforming catalyst and utilising in series catalyst beds maintained under reforming conditions in a number of adiabatic reaction zones, with a heating zone before each reaction zone, includes the steps of passing the heated feed to a reaction zone at an inlet temperature of 875 – 975°F , heating the

effluent from a reaction zone and passing it to a subsequent reaction zone at a temperature at least 25° below the temperature of the preceding reaction zone.

Catalyst Preparation

W. R. GRACE & CO. *U.S. Patent 2,861,958*

A silica gel base reforming catalyst is made by impregnating silica gel with an aqueous solution of aluminium chlorplatinate in amount to give 0.05–5 wt.% Pt and 0.01–1 wt.% Al_2O_3 , drying and converting the adsorbed Pt compound to Al_2O_3 and metallic Pt.

Hydroforming Catalyst

ESSO RESEARCH & ENGINEERING CO. *U.S. Patent 2,861,959*

A hydrocarbon conversion catalyst consists of 0.1–1% by wt. of metallic Pt finely dispersed on an Al_2O_3 carrier, promoted by the addition of less than 0.5% by wt. MoO_3 . The amount of oxide used is below the minimum at which it would, by itself, form an effective catalyst with the Al_2O_3 .

Hydrocarbon Conversion Process and Catalyst

STANDARD OIL CO. *U.S. Patent 2,861,960*

A Pt- Al_2O_3 catalyst is made by mixing with Al_2O_3 a solution of a co-ordination compound of a Pt halide and an organic compound containing an olefinic double bond, decomposing the Pt compound to deposit Pt in the Al_2O_3 and calcining.

Production of Dibenzyl Ethylenediamine

THE DISTILLERS CO. LTD. *U.S. Patent 2,862,969*

Dibenzyl ethylenediamine is prepared by hydrogenating dibenzylidene ethylenediamine with hydrogen in an inert solvent at 0–300 p.s.i. pressure and a temperature of 20 – 45°C in the presence of a Pd- Al_2O_3 catalyst in amount as to provide 0.25–1% by wt. of Pd with respect to the dibenzylidene ethylenediamine originally present.

Separating Hydrogen Isotopes

U.S. ATOMIC ENERGY COMMISSION *U.S. Patent 2,863,526*

A gaseous mixture of hydrogen and tritium is separated by contacting finely divided Pd with the mixture, which is taken up by sorption, and gradually heating the Pd to cause a fraction rich in tritium to be given off first, other evolved fractions being collected, as they are formed.

Production of Dihalobenzene

ALLIED CHEMICAL CORP. *U.S. Patent 2,866,828*

A Pt or Pd catalyst may be used in the preparation of metadihalobenzene by reacting 1, 2, 4 homotrihalobenzene with hydrogen in the presence of the catalyst.