

## CATHODIC PROTECTION

### Anode Design for Shipboard Cathodic Protection

R. L. BENEDICT, *Materials Protection*, 1965, 4, (12), 36-38

Effective protection depends on the shape and voltage of the anodes and on their area. They should be located where mechanical abrasion of paint is unlikely to occur. Platinised Ti, and Pb anodes with Pt micro-electrodes are most effective and should last the life of the ship.

## GLASS TECHNOLOGY

### A Wide Range (up to $10^{10}$ P) Rotating Cylinder Viscometer

A. NAPOLITANO, P. B. MACEDO and E. G. HAWKINS, *J. Res. N.B.S., Sect. A, Phys. Chem.*, 1965, 69A, (5), 449-455

Three techniques enable the range of this viscometer (described in *Platinum Metals Rev.*, 1963, 7, (2), 54-55) to be extended up to  $10^{10}$ P. At  $10^0 - 10^8$ P the outer Pt cylinder is rotated at constant speed and the torque measured on the inner Pt bob. From  $10^{4.5}$  to  $10^{7.5}$ P the bob is rotated through an angle and timed as it returns to its zero position. From  $10^{5.5}$  to  $10^{10}$ P the bob is driven through an angle at constant torque and is timed as it does so.

## NEW PATENTS

### METALS AND ALLOYS

#### Hydrogen Diffusion Tubes

JOHNSON, MATTHEY & CO LTD.  
*British Patent* 1,009,326

A closing plug for sealing the open end of Pd or Pd/Ag alloy  $H_2$  diffusion tube comprises a body formed of a material having approximately the same coefficient of thermal expansion and is dimensioned to give a tight fit and has a projecting, threaded spigot of smaller diameter than the tube and used to form a means for attachment of or for stabilising an internal support for the tube.

### ELECTROCHEMISTRY

#### Activated Platinum Electrodes

J. BISHOP & CO. *U.S. Patent* 3,202,594  
The overvoltage of electrodes coated with a Pt group metal, in particular Pt, is lowered by contacting the coating with an alkali metal amalgam, removing the alkali metal from the Hg film and heating the electrode so that Hg and Pt metal react and finally Hg is distilled off leaving an activated Pt group metal coating.

## TEMPERATURE MEASUREMENT

### Reference Tables for Platinum-40% Rhodium/Platinum-20% Rhodium Thermocouples

R. E. BEDFORD, *Rev. Sci. Instrum.*, 1965, 36, (11), 1571-1580

Tables in the range 0-1880°C are based on calibrations in air of ten thermocouples from four wire lots at the freezing points of Zn, Sb, Ag and Au; the melting points of Pd and Pt by the wire method, and from comparison with standard Pt:10%Rh-Pt and 5%Rh-Pt:20%Rh-Pt thermocouples at 0-1750°C. Test thermocouples are intercompared at 0-1850°C. A.P.H. Report No. 1292 gives more detailed tables.

### Measurement and Control of Gas Temperature in the FINGAL Process

M. N. ELLIOTT and J. R. GROVER, *Br. Chem. Engng.*, 1965, 10, (12), 846-849

The design of a bare wire Pt:13% Rh-Pt thermocouple is described for measurement of off-gas temperatures in the FINGAL process, developed at Harwell for the incorporation of highly radioactive fission product wastes in glass. A similar thermocouple is used with a suction-type pyrometer for gas temperatures in the process vessel.

### Silver Oxide-Palladium Electrode

GENERAL MOTORS CORP. *U.S. Patent* 3,212,934

An electrochemical secondary battery cell comprises a Zn cathode, an alkaline electrolyte and an Ag anode having 1-1.5 wt.% Pd alloyed with it.

### ELECTRODEPOSITION AND SURFACE COATINGS

#### Electrodeposition of Palladium

TECHNIC INC. *British Patent* 1,014,045  
Heavy, clean stress-free and bright electroplated Pd films are formed by using an aqueous electrolyte bath containing 2-10 g/l Pd as its chelate with N,N'-cycloalkane diamine tetraacetic acid and a buffer maintaining a pH 4-12.

#### Improved Method of Coating Graphite or Like Elements and Products Obtained by Such Method

STE. NATIONALE D'ETUDE ET DE CONSTRUCTION DE MOTEURS D'AVIATION  
*British Patent* 1,016,309

C elements, e.g. graphite, are protected against thermal shock by applying on them a fixative

intermediate layer of Ta, Zr, Mo, Hf, Nb, Ti, W or their carbides, nitrides or borides and 1-50 wt. % Ru, Rh, Pd, Os, Ir or Pt and then depositing by plasma jet spraying a coating of a selected refractory material.

### Method of Electroplating Palladium

WESTERN ELECTRIC CO. INC.

*British Patent 1,017,950*

Pd films are electrodeposited by using an aqueous solution having a pH 7-9 and containing 30-60 g/l dichlorodiamine Pd(H), 0.1-1.0 g/l selenous acid and NH<sub>3</sub> for pH adjustment and operating at 15-160 A/ft<sup>2</sup> cathodic current density.

### Fluidised Bed Plating with Noble Metals

ETHYL CORP.

*U.S. Patent 3,202,537*

Metal or ceramic substrates are coated with Pt group and other transition metals by contacting the substrate with a fluidised bed of solid, heat-decomposable, C-containing compound of the plating metal and ensuring that the temperature of the substrate is sufficient to decompose the plating metal compound.

### Electrodeposition of Platinum and Palladium

JOHNSON, MATTHEY & CO. LTD.

*U.S. Patent 3,206,382*

A bath for the electrodeposition of Pt or Pd on to Au, Ag, Cu, Ni, Ti, etc., has a pH below 2, is operated at 30-70°C and 5-25 A/ft<sup>2</sup> and contains 5-20 g/l M<sub>2</sub> (M<sup>1</sup>(NO<sub>2</sub>)<sub>2</sub>R<sub>4-a</sub>) or M<sub>2</sub> (M<sup>1</sup>(NO<sub>2</sub>)<sub>2</sub> R<sup>1</sup> (4-a)/2), where M is H, NH<sub>4</sub>, alkali or alkaline earth metal, M<sup>1</sup> is Pt or Pd, R is OH or monovalent acid radical, R is a divalent acid radical and a is 1-3.

### Electrodeposition of Iridium

U.S. SECRETARY OF THE ARMY

*U.S. Patent 3,207,680*

An excellent electrodeposited film of Ir is formed on a Cu, Ni, Nb or Mo substrate by connecting the substrate as a cathode in an electrolytic bath maintained at 25-90°C and containing X<sub>2</sub>IrY<sub>6</sub>, where X is H, Na or K and Y is Cl, Br or I and passing a current of 20-120 A/ft<sup>2</sup>.

### Deposition of Palladium

JOHNSON, MATTHEY & CO. LTD.

*Canadian Patent 722,528*

A plating bath for the immersion of electroless deposition of Pd on a metal base comprises an aqueous solution of a complex nitrito-palladite compound.

## CATALYSIS

### Preparation of Palladium Catalysts and Catalysts Obtained thereby

SNIA VISCOSA S.P.A.

*British Patent 1,007,359*

Pd/C catalysts are produced by adding to a

suspension of C in an aqueous solution of a Pd salt at least an equimolar amount of an aqueous solution of an alkaline hydroxide and then reducing the precipitated hydroxide to Pd metal by treating it with an excess of H<sub>2</sub> at 50-100°C.

### Unsaturated Carboxylic Acids

IMPERIAL CHEMICAL INDUSTRIES LTD.

*British Patent 1,007,707*

An unsaturated carboxylic acid is produced by reacting a  $\pi$ -allylic complex of PdCl<sub>2</sub> with CO at 10-150°C and 200-1000 atm and then hydrolysing the product at 100°C or above.

### Process for Preparing Aminophenols

UNIVERSAL OIL PRODUCTS CO.

*British Patent 1,009,024*

Aminophenols are produced by reacting a nitro-substituted phenol with H<sub>2</sub> in an aqueous alkaline medium at 25-100°C, 1-35 atm and in the presence of Pt, Pd, Rh, Pt or Pd black or Pt or Pd oxide supported on a porous material.

### Catalytic Production of Hydrogen Cyanide

E. I. DU PONT DE NEMOURS & CO.

*British Patent 1,009,137*

HCN is produced by reacting NH<sub>3</sub>, O<sub>2</sub> and an hydrocarbon gas at 1000-1200°C in the presence of Pt gauze catalyst at least partially insulated against heat losses by a refractory fibre of Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>, modified SiO<sub>2</sub> or K titanate and positioned upstream of the catalyst.

### Process for the Preparation of Ketene Polymers

SHELL INTERNATIONALE RESEARCH MIJ. N.V.

*British Patent 1,009,910*

Polymeric ketenes are produced by contacting the monomer at 10-15°C with a catalytic amount of PdCl<sub>2</sub> or (Ar<sub>2</sub>X)<sub>2</sub>PdCl<sub>2</sub>, where Ar is aryl and X is P, As or Sb.

### Production of Unsaturated Organic Esters

THE DISTILLERS CO. LTD. *British Patent 1,010,548*

Esters of unsaturated alcohols are produced by reacting at 30-200°C and 2-100 atm an olefine and a gas containing molecular O<sub>2</sub> with a solution containing an amine carboxylate, a carboxylic acid and 0.1-4 wt. % Pt group metal compound, e.g. PdCl<sub>2</sub> or PdAc<sub>2</sub>, in the absence of a redox system.

### Selective Hydrogenation Procedure and Catalyst therefor

CHEMETRON CORP.

*British Patent 1,011,270*

A catalyst for the selective hydrogenation of acetylenic and diolefinic hydrocarbons comprises 0.01-0.5 wt. % each of Pd and Cr taken in 2:1 to 2:3 wt. ratio on an Al<sub>2</sub>O<sub>3</sub> carrier having a pore volume of surface pores of 0.0-0.4 cm<sup>3</sup>/g and the

surface pores having a threshold diameter of less than 800Å.

### Production of Phenol

HALCON INTERNATIONAL INC.

*British Patent 1,011,432*

A catalyst for the production of phenol by vapour phase dehydrogenation of a fraction containing cyclohexanol, cyclohexanone, their lower alkyl derivatives or mixtures, comprises up to 5 wt.% Pt and an active C support which has been treated with 0.1-19% aqueous HCl, and then washed until the washings have a pH 5.3-7.0.

### Production of Aldehydes and Alcohols

THE DISTILLERS CO. LTD.

*British Patent 1,012,011*

Saturated aldehydes and/or alcohols are produced by reacting at 90-300°C, 25-300 atm and in the liquid phase, an olefine, Co and H<sub>2</sub> in the presence of a catalyst comprising a synthetic molecular sieve zeolite supporting a Pt group metal, preferably Rh or Ru.

### Dehydrogenation Process and Catalyst for Use therein

HALCON INTERNATIONAL INC.

*British Patent 1,013,115*

A dehydrogenation catalyst particularly suitable for phenol production is produced by dissolving 0.5-5 wt. parts Pt compound in H<sub>2</sub>O, mixing the solution with 100 wt. parts activated C, refluxing for at least 10 min., separating the catalyst and drying it.

### Production of Aminophenols

MONSANTO CHEMICALS LTD.

*British Patent 1,014,968*

Aminophenol or aminophenolate is produced by contacting an aqueous solution of an alkali metal nitrosophenate with H<sub>2</sub>, at 5-120°C, 200-400 psig and in the presence of a Pt or Pd catalyst.

### A Treatment Process for Silicon Compounds

TEXAS INSTRUMENTS LTD.

*British Patent 1,015,604*

PCl<sub>3</sub> is removed from halogen substituted derivatives of silane by contacting the latter for over 1 h with adsorbent Al<sub>2</sub>O<sub>3</sub> coated with 0.1-25 wt.% PtCl<sub>4</sub>.

### Oxidation, Reduction Catalysts

JOHNSON, MATTHEY & CO. LTD.

*British Patent 1,016,058*

Catalysts for oxidation, reduction and electrochemical processes are produced by forming a mixture of Pt and Ru compounds, fusing it with NaNO<sub>3</sub> to form an intimate mixture of 90-99 wt.% Pt oxide and 1-10 wt.% Ru oxide and then applying it on a suitable catalyst carrier or porous electrode matrix.

### Palladium-on-Carbon Hydrogenation Catalyst

U.S. SECRETARY OF AGRICULTURE

*U.S. Patent 3,206,440*

Vinyl esters of maleic anhydride adducts have been characterised by reduction in glacial CH<sub>3</sub>COOH in the presence of Pd/C as hydrogenation catalyst.

### Platinum Group Metal Isomerisation Catalysts

SINCLAIR REFINING CO. *U.S. Patent 3,206,525*

4-9 C paraffins are isomerised by contacting them at 500-750°F and an elevated pressure with H<sub>2</sub> and activated Al<sub>2</sub>O<sub>3</sub> supporting 0.1-2 wt.% Pt group metal and NH<sub>4</sub>BF<sub>4</sub> taken in an 0.001-0.3:1 molar ratio with respect to Al<sub>2</sub>O<sub>3</sub>.

### Noble Metal Hydrogenation Catalysts

UNION OIL COMPANY OF CALIFORNIA

*U.S. Patent 3,208,931*

Wax of desired colour stability is produced from wax bottoms or petrolatum by contacting them with H<sub>2</sub> at 700-775°F, 500-1500 psig and in the presence of a supported Group VIB and/or VIII metal, metal oxide or sulphide and then hydrogenating the product at 450-600°F in the presence of a supported Pt, Pd, Rh, Ir or Ru catalyst.

### Platinum Catalysts for Reductive Alkylation

UNIVERSAL OIL PRODUCTS CO.

*U.S. Patent 3,209,030*

A catalyst for the reductive alkylation of organic compounds having an amino or nitro substituent comprises a suitable support, preferably Al<sub>2</sub>O<sub>3</sub>, 0.01-25 wt.% Pt, 0-8 wt.% halogen and 0.05-5 mol.% S.

### Gas Oil Hydrocracking Process

SINCLAIR RESEARCH INC. *U.S. Patent 3,210,264*

A combined process for hydrocracking gas oil boiling at 460-650°F and reforming naphtha has, as its first stage, contacting the naphtha with a supported Pt metal reforming catalyst at 800-1000°F and 150-250 psig in the presence of H<sub>2</sub>.

### Supported Noble Metal Catalyst

PHILLIPS PETROLEUM CO. *U.S. Patent 3,210,296*

A high surface area active catalyst is produced by impregnating Al<sub>2</sub>O<sub>3</sub> with a 1-50 wt.% solution of PtCl<sub>4</sub> in ether, draining, drying and calcining at 200-400°C in the presence of H<sub>2</sub> to give a Pt metal content of 0.05-1 wt.%.

### Platinum Group Metal Catalysts

PHILLIPS PETROLEUM CO. *U.S. Patent 3,213,150*

Os, Ir, Ru, Rh, Pd or Pt supported on the usual carriers are used as catalysts for the demethylation

of a H<sub>2</sub> stream, e.g. a reformer off-gas stream, and subsequent hydrogenation reactions.

### **Palladium Catalysts in the Production of Unsymmetrical Hydrazines**

AEROJET-GENERAL CORP. *U.S. Patent 3,214,474*  
Unsymmetrical lower alkyl hydrazines are produced by reducing a nitroso amine with H<sub>2</sub> in aqueous solution in the presence of colloidal Pd or finely divided Pd on usual carriers, e.g. SiO<sub>2</sub>, CaCO<sub>3</sub>, BaSO<sub>4</sub>, C, etc.

### **Platinum Gauze Catalyst in the Production of Hydrogen Cyanide**

E. I. DU PONT DE NEMOURS & CO.  
*U.S. Patent 3,215,495*

An improved process for the production of HCN is operated at 1000–1200°C and utilizes NH<sub>3</sub>, O<sub>2</sub> and hydrocarbon gas reacted in a bed containing a Pt gauze catalyst.

## **FUEL CELLS**

### **Process and Apparatus for the Generation of Electrical Power**

VARTA A.G. *British Patent 1,010,635*  
A cell for the production of electrical energy comprises loose heaps of Ag, Pt or Pd catalysts or such catalyst impregnating a support and placed between screens or sieves. The partial oxidation products of a saturated or unsaturated aliphatic hydrocarbon are then fed to a 10 wt.% Pd fuel cell electrode, whilst the O<sub>2</sub> electrode is composed of Raney Ag.

### **Improvements Relating to Fuel Cells**

COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION *British Patent 1,013,703*  
An ion exchange membrane for a fuel cell is produced by treating the membrane with a reducing agent, and then with an aqueous solution of one or more noble metal salt, preferably Pt, thus forming a film of finely divided and catalytically active metal.

### **Palladium Paste-form Fuel Cell Electrode**

AIR PRODUCTS AND CHEMICALS INC. and NORTHERN NATURAL GAS CO. *U.S. Patent 3,212,936*  
A fuel cell electrode is produced by impregnating activated C powder with a Pd salt optionally mixed with a transition metal, ageing the paste, treating it with KOH and using a solid C electrode to support the paste. See also 3,212,937.

### **Platinum-coated Screen Electrodes**

AIR PRODUCTS AND CHEMICALS INC. and NORTHERN NATURAL GAS CO. *U.S. Patent 3,215,562*  
Fuel cell electrodes comprise a number of interconnected, conductive mesh screens made of a corrosion-resistant alloy and coated with catalytically active Pt.

## **GLASS TECHNOLOGY**

### **Mica-Metal Bonds**

OWENS-ILLINOIS INC. *U.S. Patent 3,206,355*  
An integral bond is formed between mica or glass-bonded mica and steel or 1:1 Pt:Pd alloy by bringing them in contact with powdered, devitrifiable glass, fusing the glass and then cooling slowly to cause devitrification and bonding.

### **A Platinum Liner for a Glass Furnace**

BAUSCH & LOMB INC. *U.S. Patent 3,206,295*  
A glass furnace with refractory-lined melting and fining chambers has the interconnecting throat and the fining chamber lined with Pt.

### **Platinum Articles for Glass Furnaces**

COMPTOIR LYON-ALEMAND LOUYOT & CIE. *U.S. Patent 3,210,167*  
Articles which come into contact with molten materials at high temperatures, e.g. molten glass, comprise a layer of Pt in contact with the material, a layer of an alloy of Pt with Ir, Ru or Os and a layer of Pt-Rh alloy.

## **ELECTRICAL AND ELECTRONIC ENGINEERING**

### **Electrode Structures for Use in Electric Discharge Devices**

THE M-O VALVE CO. LTD. *British Patent 1,007,860*  
A wire for the manufacture of grid electrodes for electric discharge devices has a refractory metal core which is coated with a layer of small Pt particles of dull grey appearance and a second layer of partly reduced TiO<sub>2</sub>.

### **Electrolytic Capacitors**

PHILIPS ELECTRONIC AND ASSOCIATED INDUSTRIES *British Patent 1,010,096*  
An electrolytic capacitor is manufactured by assembling a Ta or Nb anode provided with a dielectric oxide film formed by electrolytic oxidation, a Ag container cathode and an electrolyte containing 0.05–2 wt.% of at least one Pt group metal salt, whereby a spongy layer of such Pt group metal is deposited as a coating upon the cathode.

### **Noble Metals in Resistor Compositions**

E. I. DU PONT DE NEMOURS & CO. *U.S. Patent 3,207,706*  
A resistor composition consists of (i) 35–85% of a frit having 24–34% ZnO, 18–25% SiO<sub>2</sub>, 22–30% B<sub>2</sub>O<sub>3</sub>, 3–7% Al<sub>2</sub>O<sub>3</sub>, 4–10% Na<sub>2</sub>O, 4–6% ZrO<sub>2</sub>, 0–4% CaO, 0–4% P<sub>2</sub>O<sub>5</sub>, more than 1% and less than 2% total of PbO, Sb<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, NiO and Fe<sub>2</sub>O<sub>3</sub> and less than 0.1% other alkali metal oxides and (ii) 15–65% Ag and Pd or PdO so that the Ag:Pd ratio is 3:2 to 2:3.