

## TEMPERATURE MEASUREMENT

### Two-Point Comparison

E. W. JONES, *Instrum. Control Syst.*, 1967, **40**, (1), 115-118

A two-point method for testing and calibration of Pt resistance thermometers gives accuracy of  $\sim 0.015^\circ\text{C}$  at  $-100^\circ\text{C}$  and  $\sim 0.006^\circ\text{C}$  at  $500^\circ\text{C}$  on the International Practical Temperature Scale.

### A Precision PtRh-Pt Thermocouple for Research and Industry

M. BEDUHN and W. HEYNE, *Feinger. Tech.*, 1967, **16**, (6), 257-260

Three East German research institutes have developed the "Model DAMW" Pt:10% Rh-Pt thermocouple instrument, which is suitable for both industrial and laboratory uses. Its construction, characteristics and calibration are described.

## NEW PATENTS

### METALS AND ALLOYS

#### Heat Treatment of Platinum-Cobalt Magnets

INTERNATIONAL NICKEL LTD.

*British Patent 1,067,054*

Remarkable magnetic properties can be produced in pure alloys containing 19.8-31.2% Co by subjecting them to a disordering treatment at a temperature above  $900^\circ\text{C}$  for 30 min to 1 h, cooling to  $630-750^\circ\text{C}$  at a rate of  $50-150^\circ\text{C}/\text{min}$ , cooling to room temperature, ageing at  $630-700^\circ\text{C}$  for 5 min - 2 h and then quenching to room temperature.

#### Tungsten-Ruthenium Alloy

U.S. ATOMIC ENERGY COMMISSION

*British Patent 1,070,114*

The high temperature strength of W can be improved by adding 1.1-12% Ru (based on the weight of the alloy). A preferred composition is 1.1 wt.% Ru and 98.9% W.

#### Alloys for Strain-Gauge Elements

KABUSHIKI KAISHA HITACHI SEISAKUSHO

*U.S. Patents 3,305,815-7*

In strain gauges the strain element consists of a binary alloy of Os with 90-99% Pt (815), a ternary alloy of 20-60 at.% Pt, 20-60 at.% Pd and 5-30 at.% Ir (816) and a ternary alloy of 15-80 at.% Pt, 15-80 at.% Pd and 2-15 at.% Mo.

#### Hydrogen Diffusion Tubes

JOHNSON, MATTHEY & CO. LTD.

*U.S. Patent 3,312,043*

A closing plug for sealing the open end of Pd or Pd-Ag alloy  $\text{H}_2$  diffusion tubes consists of material with approximately the same coefficient of thermal expansion and dimensioned to fit tightly with 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. This corresponds to *British Patent 1,009,326*.

### ELECTROCHEMISTRY

#### Electrode Boiler

IMPERIAL METAL INDUSTRIES (KYNOCHE) LTD.

*British Patent 1,068,732*

An electrode for an electrode boiler, e.g. for boiling  $\text{H}_2\text{O}$ , has the parts immersed in the electrolyte (at least) made of Ti or its alloys coated with Ir, Rh, Ir-Pt, Rh-Pt or Ir-Rh-Pt.

#### Production of Platinum and Palladium Oxides

JOHNSON, MATTHEY & CO. LTD.

*French Patent 1,458,185*

Oxidation of these metals is achieved by electrolysis of a Pt or Pd anode in a bath containing molten  $\text{NaNO}_3$  and an alkali metal halide. This corresponds to *Belgian Patent 664,526*.

### ELECTRODEPOSITION AND SURFACE COATINGS

#### Coating Titanium Surfaces

SOCIETE D'ELECTRO-CHIMIE DES ACIERIES

ELECTRIQUES D'UGINE *British Patent 1,069,005*

Process for coating Ti or its alloys with a metal of the Pt group. The metal to be coated is acid pickled and the Pt group metal deposited and then heated at  $150-300^\circ\text{C}$ .

#### Applying Designs to Metallic Bases

JOHNSON MATTHEY & CO. LTD.

*French Patent 1,455,917*

A decorating composition for application to a noble metal base consists of a metallising paste containing Au, Ag, Pt, Pd or alloys thereof.

#### Palladium Plating

JOHNSON, MATTHEY & CO. LTD.

*German Patent 1,239,159*

An aqueous neutral or alkaline Pd bath contains a Pd compound, e.g.  $(\text{PdNH}_3)_2(\text{NO}_2)_2$ , and a  $\text{NH}_4$  salt of a weak acid which does not form an

insoluble compound with the Pd compound, e.g. ammonium tartrate.

## BRAZING

### Brazing Alloys for Tungsten and Molybdenum

U.S. ATOMIC ENERGY COMMISSION

*U.S. Patent 3,312,539*

These metals and their alloys are brazed using alloys of 42–95 wt.% Mo, 5–44 wt.% Rh and up to 45 wt.% Re.

### Solder for Soldering Electrovacuum Instruments

B. E. KOVALEVSKII et al.

*U.S.S.R. Patent 186,836*

A solder of lower melting point which has increased strength and plasticity of the soldered joints has composition 8–12 wt.% Ge, 2–12 wt.% Pd, balance Cu.

## CATALYSIS

### Production of Saturated Aldehydes

WACKER-CHEMIE G.M.B.H.

*British Patent 1,065,628*

The partial hydrogenation of olefinically substituted aliphatic aldehydes at 70–140°C is catalysed by a mixture of metallic Pd and one or more of Cu, Ni and Co. Au or Ag may also be present.

### Silanes, their Quaternary Salts and Polymers

DOW CORNING CORP. *British Patent 1,066,346*

Haloether silanes are obtained by the Pt-catalysed co-reduction of a silane and a hydrocarbon ether.

### Production of Aromatic Dicarbonyls

SCHOLVEN-CHEMIE A.G. *British Patent 1,066,401*

The corresponding hydrocarbons are oxidised to hydroperoxides and then reduced to dicarbonyls using H<sub>2</sub> and Pt/Al<sub>2</sub>O<sub>3</sub>.

### Carbonylation of Olefinically Unsaturated Compounds

BADISCHE ANILIN- & SODA-FABRIK A.G.

*British Patent 1,066,772*

Carbonylation is catalysed by L<sub>m</sub>PdX<sub>n</sub>, where L is an organic phosphine or phosphite, NH<sub>3</sub>, an amine, a nitrile or an unsaturated hydrocarbon, X is an anion, m is 1–4, n is 1–2 and n + m is 2–6, e.g. Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub>.

### Vinyl Acetate Production

IMPERIAL CHEMICAL INDUSTRIES LTD.

*British Patent 1,067,850*

The catalyst for the reaction of C<sub>2</sub>H<sub>4</sub> with CH<sub>3</sub>COOH consists of a redox system and a Pd salt other than the fluoride.

### Regeneration and Reactivation of Supported Platinum Group Catalysts

SHELL INTERNATIONALE RESEARCH MIJ. N.V.

*British Patent 1,069,057*

Reforming catalysts are regenerated by treatment with Cl<sub>2</sub> or a compound which liberates Cl<sub>2</sub> on heating, followed by burning in a gas containing O<sub>2</sub> to remove C. The catalyst is then maintained at a temperature higher than burning temperature in a gas of higher O<sub>2</sub> content.

### Hydrocracking Process

ESSO RESEARCH & ENGINEERING CO.

*British Patent 1,071,467*

A crystalline aluminosilicate zeolite composited with a Pt group metal is used for hydrocracking of hydrocarbons.

### Production of Finely Divided Catalyst Layers on the Pore-free Surfaces of Hydrogen-absorbing Metallic Bodies

VARTA A.G.

*British Patent 1,071,503*

A metallic body is coated on one side with a catalyst layer and subjected to H<sub>2</sub> pressure, whilst on the other side (e.g. in a tube) a solution of the metal to be used for hydrogenation (e.g. Pd(NO<sub>3</sub>)<sub>2</sub>) is precipitated by diffusion of H<sub>2</sub> and reduction.

### Composite Catalysts

OFFICE NATIONAL INDUSTRIEL DE L'AZOTE

*British Patent 1,072,172*

A catalyst comprising 0.1–2.0% Pt, 8–50% Ni and/or Co and an Al<sub>2</sub>O<sub>3</sub> or MgO support. It is used in the methanation of CO by H<sub>2</sub> and in the reforming of hydrocarbons.

### Hydrocarbon Conversion Process

TEXACO DEVELOPMENT CORP.

*British Patent 1,072,620*

Mixtures of hydrocarbons (pentane, hexane, etc.) may be converted to highly branched hydrocarbon products by using a chloride-activated Pt/Al<sub>2</sub>O<sub>3</sub> catalyst.

### Preparation of Aryl Thiols

UNITED STATES RUBBER CO.

*British Patent 1,073,200*

PtS<sub>2</sub> is used as a catalyst for conversion of aryl sulphonic acids to aryl thiols by hydrogenation. This obviates the use of H<sub>2</sub>S and S which are needed with prior art processes using base metal sulphides.

### Preparing Catalysts

ESSO RESEARCH & ENGINEERING CO.

*British Patent 1,076,215*

A highly active catalyst is made by contacting a support with a salt solution of a catalytic metal, reducing the metal ions and subsequently removing the support to leave a finely divided metal.

Thus Pt metals, Ag and Au can be deposited singly or in mixtures on  $\text{CaCO}_3$  which is then removed.

### Dehydrogenation of Cyclo-octene

U.S. RUBBER CO. *U.S. Patent 3,305,593*  
1,5-Cyclo-octadiene is produced by heating cyclo-octene and a Rh salt, e.g.  $\text{RhCl}_3$ , in  $\text{CH}_2\text{OH}$ , at 50–150°C.

### Hydro dealkylation Catalyst

UNIVERSAL OIL PRODUCTS CO.  
*U.S. Patent 3,306,944*

The demethylation of alkyl aromatic hydrocarbons with Rh is catalysed by Rh deposited on an alkali metal-promoted metal oxide support, e.g.  $\text{Li}/\text{Al}_2\text{O}_3$ .

### Selective Hydrogenation of Hydrocarbons

MOBIL OIL CORP. *U.S. Patent 3,309,307*  
Dienes are selectively hydrogenated in the presence of olefines by using a Pd catalyst in the presence of either  $\text{CS}_2$  or  $\text{H}_2\text{S}$  below the desulphurisation temperature.

### Catalytic Hydrogenation of Paraffin Hydrocarbons

UNIVERSAL OIL PRODUCTS CO.  
*U.S. Patent 3,310,599*

A new composite catalyst, e.g. for producing iso- $\text{C}_4\text{H}_8$  from iso- $\text{C}_4\text{H}_{10}$ , 0.01–1.5%  $\text{Al}_2\text{O}_3/\text{Li}$ , 0.05–5% Group VIII metal and sufficient Te, Se or their compounds to give full cracking and isomerisation of the Group VIII metal. Pt and Pd are the preferred noble metals.

### Catalyst for Combining Hydrogen and Oxygen in Thorium Slurries

U.S. ATOMIC ENERGY COMMISSION  
*U.S. Patent 3,312,526*

A catalyst for reversing the radiolytic decomposition of  $\text{H}_2\text{O}$  in  $\text{ThO}_2$  slurries is produced by heating an aqueous  $\text{ThO}_2$  sol and platinic acid in a Th:Pt ratio of 2–3:1 until a flocculated suspension is formed. The suspended solids of platinised  $\text{ThO}_2$  are recovered and added to  $\text{ThO}_2$  slurries.

### Preparation of Butyrolactone

PETRO-TEX CORP. *U.S. Patent 3,312,718*  
Succinic anhydride is hydrogenated to butyrolactone in the presence of a suitable catalyst, e.g. a Pt metal, and silicotungstic acid at 200–300°C and at above 500 psig.

### Mono-oxonation Products of Cyclic Dimers and Trimers of Butadiene

CHEMISCHE WERKE HÜLS A.G.  
*U.S. Patent 3,312,742*

These butadiene-1,3 dimers and trimers are reacted with CO and  $\text{H}_2$  in the presence of a catalyst mixture consisting of (a) Co carbonyls or salts of fatty acids, Pd halides or finely divided

Pd and (b) a Cu chromite, Pt or Ag/Zn/Cr oxide to introduce a formyl group on a double bond.

### Production of 4-Halobutene-1

E.I. DU PONT DE NEMOURS & CO.  
*U.S. Patent 3,312,747*

Commercially valuable 4-chloro- and 4-bromobutene-1 are produced by dehydrohalogenation of 1,3-dihalobutane at 200–385°C in the presence of  $\text{Pd}/\text{Al}_2\text{O}_3$ ,  $\text{Rh}/\text{Al}_2\text{O}_3$ , ZnO or activated  $\text{Al}_2\text{O}_3$ .

### Platinum Double Bond Addition Catalyst

GENERAL ELECTRIC CO. (NEW YORK)  
*U.S. Patent 3,313,773*

The addition of a Si compound, having at least one atom of H attached to the Si atom, to a C–C unsaturated bond is catalysed by trimethyl platinum iodide or diplatinum hexamethyl.

### Hydrocracking Process

ESSO RESEARCH & ENGINEERING CO.  
*U.S. Patent 3,318,802*

The activity of a crystalline  $\text{SiO}_2\text{-Al}_2\text{O}_3$  zeolite containing a Pt group metal is increased by introducing a halogen containing compound into the hydrocracking zone.

### Production of Metallic Oxides

JOHNSON, MATTHEY & CO. LTD.  
*French Patent 1,458,185*

Oxides of Pt, Pd or mixtures of Pt and/or Pd with other metals are produced by electrolysis of a molten bath of an alkali metal nitrate and chloride with anodes of the requisite metals.

### Oxide Catalysts for Chemical Reactions

JOHNSON, MATTHEY & CO. LTD.  
*French Patent 1,458,671*

The catalyst is a homogeneous and intimate mixture, not merely a physical mixture, of 20–90%  $\text{PtO}_2$  and 10–80%  $\text{RuO}_2$  by weight.

### Chemical Reaction Catalyst

G. WILKINSON  
*French Patent 1,459,643 Italian Patent 748,928*

A catalyst for hydrogenation, hydroformylation and carbonylation consists of a platinum metal halide or pseudohalide complexed with an organic isocyanate or a Group VB or VIB compound, e.g.  $(\text{Ph}_3\text{P})_3\text{RhCl}$ . This corresponds to *Canadian Patent 745,663*.

### Catalyst Production

JOHNSON, MATTHEY & CO. LTD.  
*Dutch Appln. 66.17,004*

A general purpose chemical reaction catalyst is an intimate homogeneous mixture of a Pt metal oxide and a baser metal oxide, preferably in a ratio of 3:1 or higher. The base metal may be Fe, Co, Ni, Cu, etc., e.g. a mixture of Ni and Pt oxides. The mixture must not be a simple physical mixture.

## Olefin Isomerisation

JOHNSON, MATTHEY & CO. LTD.

*Italian Patent 743,469*

An improved catalyst especially for olefin isomerisation consists of a salt such as  $\text{RhCl}_3$  or  $\text{PdCl}_2$  dissolved in a virtually non-volatile hydroxylic compound, e.g. propylene glycol, optionally dissolved on an inert porous support. This corresponds to *French Patent 1,445,176*.

## FUEL CELLS

### Fuel Cells

TOKYO SHIBAURA ELECTRIC CO. LTD.

*British Patent 1,074,561*

A  $\text{H}_2$  electrode consists of a non-porous Pd or Pd alloy plate which is covered with Pd black on the surface exposed to  $\text{H}_2$ , and with black mixture of Pd and Pt on the other surface.

### Electrodes

ROBERT BOSCH G.m.b.H. *British Patent 1,074,862*

Porous sintered electrodes for fuel cells use Ni as frame metal, and Pt group metal or its Ni alloy as skeleton catalyst in which up to 20% Ti, V, Cr, Co, Mo, Ru or Ta is added.

### Hydrogen Fuel Cell Structure

UNION CARBIDE CORP. *U.S. Patent 3,307,977*

A heavy metal salt of at least one of Fe, Co, Ni, Mn, Cr, Cu, Ag, Au, V, Ti, Th, U and rare earth metals is used with an Al salt to coat a layer of spinel on a porous body (e.g. C). A Pt salt is then used to deposit a catalytic layer.

### Fuel Cell Electrode

AIR PRODUCTS & CHEMICALS INC. and NORTHERN NATURAL GAS CO. *U.S. Patent 3,309,231*

It has been found that when Pt is used with a Group IB or VIII noble metal in certain proportions, the noble metal acts synergistically to improve the activity of the Pt metal catalyst.

### Activation of Electrodes Containing Platinum or Palladium

UNION OIL CO. OF CALIFORNIA

*U.S. Patent 3,311,508*

Pt or Pd bonded to a fuel cell electrode is activated by exposure to a  $\text{H}_2$  atmosphere at room temperature. After activation the electrode must be exposed to an inert atmosphere to remove  $\text{H}_2$  traces before use in the presence of  $\text{O}_2$ .

## CHEMICAL TECHNOLOGY

### Process for Bonding Two Temperature-resistant Members

SOCIETE NATIONALE D'ETUDE ET DE CONSTRUCTION DE MOTEURS D'AVIATION *British Patent 1,071,179*

A bonding layer is interposed between the surfaces of the two members at least one of which is

made of graphite. This layer is a refractory material comprising the elements W, Mo, Zr, Hf, Ta, Ti and Ni; either singly or as mixtures and one or more of the Pt group metals.

### Production of Pure Hydrogen

J. F. MAHLER APPARARATE- & OFENBAU K.G.

*German Patent 1,238,884*

$\text{NH}_3$  is decomposed, in the absence of further catalysts, on a hot membrane containing Pd which allows the nascent  $\text{H}_2$  to pass through.

## ELECTRICAL AND ELECTRONIC ENGINEERING

### Semiconductor Devices

MULLARD LTD.

*British Patent 1,074,284*

A semiconductor device is made by alloying Bi and Pt to a semiconductor body consisting of at least two components, none of which is Bi. The amount of Pt is up to 10% of the alloy.

### Electrical Resistance Element

CTS CORP.

*U.S. Patent 3,304,199*

Resistance elements are produced by applying to a non-conducting substrate a mixture of 2-70 wt.% of a finely divided metal oxide selected from  $\text{RuO}_2$  and  $\text{IrO}_2$  and 98-30% powdered glass frit.

### Impedance Element with Alloy Connector

NYTRONICS INC.

*U.S. Patent 3,310,718*

Strong stable leads to miniaturised impedance components are produced using connectors (such as wire) made from wrought Ag-Pd alloy, particularly alloys with 3-20% Pd.

### Iridium Tip Electrode

CHAMPION SPARK PLUG CO. *U.S. Patent 3,315,113*

A tip electrode, especially for a sparking plug, is made from an Ir wire on which an outwardly extending shoulder is formed by melting the centre of the length of wire and pushing the ends inwards. This avoids mechanical working in which the metal is embrittled.

### Platinum Electrical Contacts

INTERNATIONAL BUSINESS MACHINES CORP.

*French Patent 1,458,861*

Contacts with ohmic properties stable at high temperatures are mixtures of 91-93 wt.% of a Pt group metal and 7-9 wt.% of C. The Pt group metal is suitably Pt.

### Ruthenium in a Glass Conductor

AIR REDUCTION CO. INC.

*French Patent 1,463,749*

A new form of electrical resistance consists of a mixture of Tl oxide and  $\text{RuO}_2$  intimately dispersed in a vitreous matrix. Preferably the  $\text{RuO}_2$  is 0.05-80% of the mixture. Au, Pt or Pd may also be present.