

meter is reading too high or too low. The resistance elements are mounted in the glass reaction vessel by means of tubes protruding to the inside.

#### **Intercomparison of Eleven Resistance Thermometers at the Ice, Steam, Tin, Cadmium and Zinc Points**

E. H. MCLAREN, *Canad. J. Physics*, 1959, **37**, (4), 422-432

Eleven standard thermometers comprising three different types of construction were compared. Temperature coefficients determined from measurements at the ice, steam and Zn points were used to calibrate the thermometers for measurements at the Sn and Cd points. The precision of measurement was  $\sim 0.0002^{\circ}\text{C}$  at each fixed point. No significant error in the readings with any thermometer was observed. Since the quadratic resistance-temperature relations were used in these calculations their applic-

ability to Pt resistance thermometers in this range is verified. The results also indicate the reliability of the liquidus points of In, Sn and Cd as precision alternatives to the steam point on the International Temperature Scale.

#### **Equipment Used for Vacuum Melting and Casting in the Iron and Steel Industry**

A. S. D. BARRETT and M. E. HARPER, *J. Iron Steel Inst.*, 1959, **191**, (4), 366-374

Vacuum metallurgy is being used in the production of specialised alloys. The development of a 56 lb furnace for research and pilot-plant work and one of 5 cwt for production work is described. Equipment used in the operation and control of such furnaces is outlined. Temperature measurement of the melt is carried out by means of two 5% Rh-Pt/20%Rh-Pt immersion thermocouples. Using molybdenum-alumina sheaths up to 30 dips can be achieved without replacement of the sheath.

## **NEW PATENTS**

#### **Production of Tetracyclonitrile**

AMERICAN CYANAMID CO. *British Patent* 808,701

Tetracyclonitrile is prepared by reducing chlor- or brom-tetracyclonitrile by hydrogenation in the presence preferably of a Pd-charcoal catalyst and an inert organic solvent such as methyl "Cellosolve" (RTM).

#### **Electrodeposition of Rhodium**

THE MOND NICKEL CO. LTD. *British Patent* 808,958

In order to reduce or prevent cracking of a rhodium deposit, deposited from an aqueous electrolyte composed mainly of rhodium sulphate, rhodium phosphate, rhodium fluoborate or a mixture of any of them, selenic acid or an alkali metal salt thereof is added to the electrolyte, preferably in the amount of 0.4 to 1 g/l.

#### **Combustible Gas Indicator**

MINE SAFETY APPLIANCES CO. *British Patent* 809,215

In a combustible gas indicator comprising a detector filament heated electrically and electrical means responsive to the increase in temperature of the filament, the filament is formed of a coil of Pt having a narrow band at the mid-section surrounded by a thin adherent coating of glass of melting point above the operating temperature of the coil.

#### **Organochlorosilanes**

MIDLAND SILICONES LTD. *British Patent* 809,317

Reference is made to the use of activated Pt on

charcoal (5% by wt. of Pt) in the preparation of organochlorosilanes.

#### **Reduction of Carbonyl Compounds**

SOC. DUMONT FRERES ET CIE. *British Patent* 809,339

A carbonyl compound, i.e. aldehyde, hydroxyaldehyde, ketone or hydroxyketone, is reduced to form the corresponding alcohol by catalytic hydrogenation in the presence of a non-soluble anion-exchange resin. Pd or Pt oxide may be used as catalyst.

#### **Preparation of Carboxylic Acid Esters**

E. I. DU PONT DE NEMOURS & CO. *British Patent* 809,496

Carboxylic acid esters are made by reacting carbon monoxide, a non-aromatic unsaturated hydrocarbon (each carbon atom bonded directly to only one other carbon atom) and a monohydric primary or secondary alcohol in the presence of a catalyst comprising a combination of an alcohol-soluble salt of Sn or Ge with an alcohol-soluble salt of a Group VIII noble metal at 30-325°C and pressure of 100-1000 atm. The catalyst may comprise tin chloride and platinum chloride or chloroplatinic acid. Palladium chloride or ruthenium dioxide may also be used.

#### **Upgrading of Gasoline**

UNIVERSAL OIL PRODUCTS CO. *British Patent* 809,635

Reference is made to the use of a supported Pt catalyst in a process for upgrading gasoline. A

catalyst comprising a composite of  $\text{Al}_2\text{O}_3$ , 0.2–1% by wt. Pt and 0.2–1% by wt. halogen is preferred.

### Solder Alloys and Soldering

PHILIPS ELECTRICAL INDUSTRIES LTD. *British Patent 810,076*

A ductile soldering material for use in soldering together W parts consists of an alloy of Co with Pd and/or Au, which can be represented by a point on the straight line, which, in the ternary Au–Co–Pd alloy diagram, joins the point corresponding to an alloy of 40% by wt. of Co and 60% by wt. of Pd to the point corresponding to an alloy of 90% by wt. of Au and 10% by wt. of Co.

### Dealkylation of Aromatic Hydrocarbons

THE COAL TAR RESEARCH ASSOCIATION *British Patent 810,751*

Alkyl aromatic hydrocarbons are dealkylated by leading the hydrocarbon as a vapour, together with hydrogen, over a catalyst, comprising active C, preferably promoted with 1–5% of Pt or Pd, at a temperature of 450–700°C.

### Electrolytic Coating of Metal with Platinum

PHILIPS ELECTRICAL INDUSTRIES LTD. *British Patent 810,818*

A metallic object, e.g. a Mo wire, is electrolytically coated with a layer of Pt by depositing Pt black on the wire from an electrolyte consisting of an aqueous solution of 150–300 g. of  $\text{H}_2\text{PtCl}_6$  and 1–15 g of  $\text{HgCl}_2$  per l and then sintering to convert to Pt metal.

### Hydrogenation of Acetylenic Carbinols

F. HOFFMANN-LA ROCHE & CO. *British Patent 810,913*

Ethylenic carbinols of given general formula are prepared by hydrogenating acetylenic carbinols of given formula in solution in 5–25% of their wt. of quinoline in the presence of a Pd hydrogenation catalyst which selectively influences the hydrogenation of an acetylenic linkage to an ethylenic linkage. A 5% Pd– $\text{CaCO}_3$  catalyst is referred to.

### Amino Acid Derivatives

PARKE DAVIS & CO. *British Patent 811,104*

A noble metal, e.g. Pd catalyst, is used in a process for producing 4-(4-diazo-3-oxobutyl)-oxazolidine-2,5-dione, which includes subjecting Y-benzyl-N-carboxyglutamate anhydride to hydrogenolysis in the presence of the catalyst to form N-carboxyglutamate anhydride.

### Electrolytic Condenser

STANDARD TELEPHONES & CABLES LTD. *British Patent 811,331*

An electrolytic, e.g. Ta, condenser in which one electrode only is formed, has, in order to enlarge

the surface of the non-formed electrode, those parts of the electrode in contact with the electrolyte coated with a spongy layer, including a Pt group metal. Pt black is preferred.

### Dilactones

E. I. DU PONT DE NEMOURS & CO. *British Patent 811,498*

A dilactone of given general formula prepared by reacting an acetylenic compound, in an inert organic liquid reaction medium free of active hydrogen, with carbon monoxide, is subjected to catalytic hydrogenation in the presence of a Pd catalyst to produce the corresponding hexahydro compound, or a Pt catalyst to produce a suberic acid.

### Production of High Purity Hydrogen

UNION OIL COMPANY OF CALIFORNIA *British Patent 811,749*

A Pt catalyst is used in the hydrogenation to methane of residual carbon monoxide obtained in the purification of hydrogen contaminated with carbon monoxide.

### Reforming Catalyst

W. R. GRACE & CO. *British Patent 811,775*

A gasoline reforming catalyst is made by contacting amalgamated Al with an aqueous hydrogen peroxide solution (1–4%  $\text{H}_2\text{O}_2$ ), separating the thus formed  $\text{Al}_2\text{O}_3$  from the supernatant liquid and impregnating it with a solution of chloroplatinic acid and fluosilicic acid in amount to give 0.1–1% Pt, 0.01–1% F and 0.01–1%  $\text{SiO}_2$  in the final catalyst, and converting to Pt.

### Catalytic Hydrogenation of Acetylene

ENGELHARD INDUSTRIES INC. *British Patent 811,820*

The selective hydrogenation of acetylene in a gaseous mixture is effected in the presence of a catalyst composed of 0.001–0.1% of Pd on activated  $\text{Al}_2\text{O}_3$ . Rh or Ru may be included as promoter.

### Non-metallic Ferromagnetic Materials

E. I. DU PONT DE NEMOURS & CO. *British Patent 811,963*

Reference is made to the use of a flexible Pt tube in a process for preparing a complex oxide of Mn, Co and Ni.

### Supported Catalysts

THE DISTILLERS CO. LTD. *British Patent 812,382*

A supported catalyst is made by treating solid mildly alkaline support material with a solution of a Pt group metal compound in an organic solvent to deposit the oxide of the metal on the support and then reducing the oxide to the metal. The use of palladous chloride and alumina, magnesia, titania or chromia as the support is preferred.

### Isomerisation of Hydrocarbons

UNIVERSAL OIL PRODUCTS CO. *Belgian Patent* 563,491

The isomerisation of hydrocarbons is effected in the presence of a catalyst comprising a refractory metal oxide, a Pt group metal and a combined halogen representing an amount of F between 2 and 5% by wt.

### Production of Hydrogenation-Dehydrogenation Catalyst from Reforming Catalysts

HOUDRY PROCESS CORP. *German Patent* 1,033,641

A method of producing a non-acid monofunctional hydrogenation-dehydrogenation catalyst from a reforming catalyst composed of a major amount of activated  $\gamma$ - $\text{Al}_2\text{O}_3$  and 0.2-1.9% of metallic Pt and halide consists in treating the reforming catalyst in a gaseous atmosphere containing water vapour and a reducing agent and in the absence of an oxidising agent at normal pressure up to a pressure of 10 atm and a temperature of 370-540°C for at least one hour.

### Manufacture of Supported Platinum Catalyst

UNIVERSAL OIL PRODUCTS CO. *German Patent* 1,035,106

In the manufacture of supported Pt catalyst containing 0.01-2% of Pt and, if desired, 0.1-8% of combined Cl, in which the fired  $\text{SiO}_2$ -free carrier material is obtained by dehydrating metal hydroxides and, after impregnation with a Pt solution is heated to above 260°C, the porous carrier particles of more than 0.75 mm diameter, fired at 425-700°C before impregnation, are impregnated with a solution containing the Pt compound and an added basic nitrogen compound and the impregnated particles are heated in an oxygen-containing gas at 260-595°C.

### Catalyst Gauze

W. C. HERAEUS G.m.b.H. *German Patent* 1,037,426

A catalyst gauze particularly for chemical reactions in the gas- or vapour phase consists of interwoven threads formed from wires of catalytically active metals and/or alloys, preferably Pt or Pt alloys.

### Manufacture of Palladium Catalysts

R. KUHN *German Patent* 1,037,427

In the manufacture of Pd catalysts on alkaline earth sulphate carriers from a Pd salt and alkaline earth compounds for use in catalytic hydrogenation in the liquid phase a solution of palladium chloride in dilute sulphuric acid is added to an alkaline earth hydroxide solution, preferably barium hydroxide, and the resultant brown-coloured deposit consisting essentially of alkaline earth sulphate and palladium oxyhydrate separated.

### Production of Composite Metals

VACUUMSCHMELZE A.G. *German Patent* 1,040,873

In the production of multiple metals of which

one component is a Mn alloy and the other component or components is/are base metal alloys, the juxtaposed surfaces are provided with a thin protective layer of metal, the oxide decomposition temperature of which is lower than the plating temperature and the components are united by pressure at a temperature below the melting temperature of the layer. A Pt group metal may be used.

### Selective Hydrogenation

OLIN MATHIESON CHEMICAL CORP. *U.S. Patent* 2,866,794

A 3,6-endoxy-3,6-dihydrophthalate is selectively hydrogenated by reacting a diester of 3,6-endoxy-3,6-dihydrophthalic acid with hydrogen in the presence of a catalyst comprising Pd, Pt or Rh in a liquid solvent consisting of a chlorinated lower alkane, a chlorinated cycloalkane or a chlorinated aromatic hydrocarbon, and recovering the 3-6-endoxy-3, 4, 5, 6-tetrahydrophthalate formed.

### Aromatising a Naphtha

THE STANDARD OIL CO. *U.S. Patent* 2,870,084

Petroleum naphtha is reformed by contacting it at 800-1200°F at a total pressure of 0-100 p.s.i.g. at a hydrogen partial pressure of 10-18 p.s.i. absolute with a co-precipitated alumina-chromia catalyst containing 70-82 mol % alumina, 30-18 mol % chromia and 0.001-1 mol %, based on weight of catalyst, of platinum oxide or palladium oxide.

### Hydrogenation of Phosphatides

AMERICAN LECITHIN CO. INC. *U.S. Patent* 2,870,179

Phosphatides are hydrogenated at 75-80°C and at 50-150 atms in the presence of finely divided Ni and Pt as a catalyst.

### Difluorochloroacetaldehyde

ALLIED CHEMICAL CORP. *U.S. Patent* 2,870,213

$\text{CClF}_2\text{CHO}$  is made by subjecting gaseous  $\text{CClF}_2\text{CoCl}$  to the action of a reactive amount of hydrogen at 110-210°C and in the presence of Pd-activated C catalyst.

### Fabrication of Neutron Sources

U.S. ATOMIC ENERGY COMMISSION *U.S. Patent* 2,870,339

A source of neutrons is prepared by heating a sealed container containing an amount of polonium-210 and B powder coated with Ag or Pt to above 450°C for a predetermined time to effect combination of the Po with the coating material.

### Synthesis of $\alpha$ -Methylene Carboxylic Acids

E. I. DU PONT DE NEMOURS & CO. *U.S. Patent* 2,871,262

Esters of  $\alpha$ -methylene carboxylic acids are pre-

pared by reacting allene and an alkanol or cycloalkanol with carbon monoxide at 120–250°C and in contact with ruthenium carbonyl.

### Producing Articles for Enamelling

INLAND STEEL CO. *U.S. Patent 2,872,352*

Iron or steel having pickle lag is prepared for enamelling by pickling the metal in an aqueous bath of mineral acid to remove from the surface thereof an amount of metal at least equivalent to the predetermined pickle lag, the bath containing an adherence-promoting metal, which may be Pt, Pd, Ir or Rh, and depositing this adherence-promoting metal on the Fe or steel during pickling in amount sufficient to produce satisfactory enamel adherence.

### Copper Sensitisers

SYLVANIA ELECTRIC PRODUCTS INC. *U.S. Patent 2,872,359*

An insulator substrate is sensitised by immersion in an alkaline solution containing a hydroxide complex of Pt,  $(Pt(OH)_6^{-2})$  or of Pd,  $(Pd(OH)_6^{-2})$  and electrolessly depositing Cu on the so-treated substrate.

### Preconditioning of Catalysts

STANDARD OIL CO. *U.S. Patent 2,876,195*

A Pt- $Al_2O_3$  catalyst to be used in the hydroforming of petroleum naphthas to produce a gasoline having an F-1 octane number above 90 is preconditioned by exposing it, when fully active, to contact under hydroforming conditions with a hydrocarbon stock free of substances deleterious to the catalyst for a time sufficient to deposit 0.1–10% of coke thereon, thereby substantially improving the activity maintenance of the catalyst.

### Desulphurising of Petroleum Fractions

ESSO RESEARCH & ENGINEERING CO. *U.S. Patent 2,876,196*

A petroleum fraction boiling at 100–450°F and containing less than 0.3 wt. % of S is desulphurised by partially vaporising the fraction and passing it through a bed of finely divided Pt impregnated on a suitable carrier and maintained at 300–500°F and a pressure from atm. to 100 p.s.i.g. The fraction is passed through the bed until the latter contains not more than 2 atoms of S per atom of Pt. Hydrogen is excluded from the reaction, the sole reactants being Pt and the S compounds and hydrocarbons in the fraction.

### Purification of Benzene

SOCONY MOBIL OIL CO. INC. *U.S. Patent 2,876,268*

Thiophenic materials are removed from a benzene containing such materials by contacting the benzene with a catalyst formed of 0.1% to 1% by wt. of catalyst of Pt deposited on  $Al_2O_3$  in the presence of hydrogen in amounts, in terms of mole ratio of hydrogen to hydrocarbon, of 4 to

12 at pressures of 200 p.s.i.g. to 1200 p.s.i.g. at a liquid hourly space velocity of 0.5 to 10 and a temperature of 450–550°F. The temperature is from 500–525°F at an hourly space velocity of 1 and a pressure of 1000 p.s.i.g.; below 500–525°F with a velocity lower than 1 and a pressure greater than 1000 p.s.i.g. and above 500–525°F with a velocity greater than 1 and a pressure below 1000 p.s.i.g.

### Composite Electrodes

SIEMENS & HALSKE A.G. *U.S. Patent 2,876,400*

A transistor comprises a crystal and an electrode in point contact therewith, the electrode consisting of a core of one or more of the metals, Nb, Ta, Fe, Cu, Sn, Zn, P, Be or an alloy thereof, on which is a coating of Rh, Pd, Ir and/or Pt or an alloy or alloys thereof.

### Semi-conductor Devices

RADIO CORP. OF AMERICA *U.S. Patent 2,877,396*

A semi-conductor device includes a body of crystalline semi-conductive material having upon a surface thereof a large area rectifying electrode to which is bonded an electrical lead composed of a Pt-coated Mo wire.

### Reforming Catalyst

UNIVERSAL OIL PRODUCTS CO. *U.S. Patent 2,878,192*

A catalyst is made by impregnating  $Al_2O_3$  with a solution of chloroplatinic acid, hydrogen fluoride and nitric, sulphuric or phosphoric acid. The concentration of the acid and the impregnating temperature are such that substantially no reaction takes place between the  $Al_2O_3$  and the acid during impregnation. The amount of hydrogen fluoride is such as to form in the final catalyst 0.1–8% by wt. of combined F. The amount of acid is sufficient to lower the pH of the solution to below 2.5.

### N,N<sup>1</sup>-alkylene-bis-(3-hydroxypiperidine) Compounds

U.S. VITAMIN CORP. *U.S. Patent 2,878,254*

N,N<sup>1</sup>-alkylene-bis-(3-hydroxypiperidine) is prepared by hydrogenating a N,N<sup>1</sup>-alkylene-bis-(3-oxypyridyl) betaine in a solvent in the presence of Rh as a catalyst.

### Hydroforming Catalyst

ESSO RESEARCH & ENGINEERING CO. *U.S. Patent 2,881,131*

A naphtha is subjected to elevated temperature and pressure in the presence of added hydrogen and a catalyst made by impregnating acid washed and dried activated C with a Pt-containing solution sufficient to deposit up to 1.2% Pt, drying, thoroughly mixing in anhydrous aluminium chloride and heating the mixture to 900°F for 1 h. to produce a dry catalyst containing up to 2% aluminium chloride.