

obtained using intermediate metal thermocouples are in accord with practical observations and with theory. The results obtained for the 13½ lb castings were unsatisfactory with both types of thermocouple due to the response time of the instruments being too long for the rapid solidification which occurs. Recalibration of the intermediate metal thermocouples after use showed no serious error due to contamination of the wires by steel from the castings.

#### **Design of an Immersion Thermocouple and its Application in Open-Hearth Steelworks**

H. LÖSCHER, *Neue Hütte*, 1958, 3, (7), 416-425

A review of literature on the developments in design of immersion thermocouples and sheaths for use in the steel industry. The thermocouples 6%Rh-Pt/30%Rh-Pt and Pt/10%Rh-Pt are described. The dependence of the mean temperature increase of the bath on the admission temperature and heat supply is discussed. The improvement of steel quality by observing the correct tapping and casting temperatures is described.

#### **Error in Temperature Measurement due to the Interdiffusion at the Hot Junction of a Thermocouple**

A. J. MORTLOCK, *J. Sci. Instruments*, 1958, 35, (8), 283-284

Theoretical expressions are derived for the error in e.m.f. due to interdiffusion for the case where one arm of the thermocouple is a pure metal and

the other is an alloy of this metal. The error is less when the thermocouple leads are anti-parallel than when they are parallel. For the normal parallel arrangement a Pt/13%Rh-Pt thermocouple in a gradient of 10°C/cm may, according to the equations derived, have an error of up to 1.3°C at all temperatures in the normal operating range following a heat treatment equivalent to 100 days at 1500°C.

#### **On the Accuracy of Temperature Measurement of Molten Steel with Immersion Thermocouples of Various Types**

A. N. GORDOV and N. N. ERGARDT, *Zavods. Lab.*, 1957, 23, (6), 727-730. (In Russian)

The thermocouples in use in the Russian steel industry at present are Pt/10% Rh-Pt and W/Mo. Errors obtained with the latter are due to oxidation and crystallisation on immersion and amount to about ±20°C. The noble metal thermocouples which are used give errors since the Pt is not sufficiently pure and the sheathing is ineffective. The use of spec. pure Pt and effective sheathing is recommended. Work is described on the thermocouples 6%Rh-Pt/30%Rh-Pt and 13%Rh-Pt/30%Rh-Pt which are more stable at high temperatures than Pt/10%Rh-Pt. They have sufficiently high e.m.fs. for technical use in the temperature range 1500-1700°C. The 6%Rh-Pt/30%Rh-Pt couple seems the most promising. A method of calibrating shallow-immersion thermocouples for the temperatures met with in industry is described.

## **NEW PATENTS**

#### **Catalytic Reforming of Hydrocarbon Mixtures**

N. V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ  
*British Patent 794,650*

Hydrocarbon mixtures of the gasoline type with a naphthene content of less than 30% by weight are reformed by contacting the mixture, together with an 8-fold molar proportion of hydrogen, at at least 10 atm. A 1% platinum-on-alumina catalyst activated with halogen is used.

#### **Hydrogenation of Petroleum Fractions**

ESSO RESEARCH AND ENGINEERING CO. *British Patent 794,809*

Petroleum fractions, containing aromatic compounds boiling in the range of 300-650°F are upgraded by hydrogenation at a temperature of 500-675°F, a pressure of 100-1,000 p.s.i.g. in the presence of 2,000-15,000 standard cubic feet of hydrogen per barrel of feed in contact with a catalyst comprising platinum supported on eta alumina. 0.001-5% (preferably 0.05-1%) by

weight of platinum is used. Chloroplatinic acid or platinum sulphide is preferred. Method of making catalyst is described.

#### **Catalytic Processes**

THE M.W. KELLOGG CO. *British Patent 794,915*

A catalyst of platinum- or palladium- on alumina is used in a process for the catalytic converting or reforming of hydrocarbons in which the reactant material is passed at reaction temperature and pressure through a fixed bed of the catalyst along paths of equal length and flow resistance.

#### **Higher Fatty Alcohol**

CALIFORNIA RESEARCH CORP. *British Patent 795,181*

A ruthenium-containing hydrogenation catalyst is used in a process for the production of higher fatty alcohols from fat.

#### **Manufacture of Aromatic Hydrocarbons**

FARBWERKE HOECHST A.G. *British Patent 795,235*  
Aromatic hydrocarbons containing a substantial

amount of para-xylene are prepared by heating 2:4:4-trimethylpentene at elevated temperatures in the presence of a catalyst comprising an oxide of chromium, molybdenum or tungsten and containing 0.1-5% of palladium or platinum.

### Manufacture of Hydrogen Peroxide

COLUMBIA-SOUTHERN CHEMICAL CORP. *British Patent 795,272*

A catalyst of palladium, rhodium or platinum supported on alumina is used as a hydrogenation catalyst in the manufacture of hydrogen peroxide by hydrogenation of an anthraquinone.

### Isoxazolidone Derivatives

F. HOFFMANN-LA ROCHE & CO. A.G. *British Patent 795,459*

A highly active hydrogenation catalyst, e.g. platinum or palladium, is used in the second step of a process of making 4-amino-isoxazolidone-(3) and 5-alkyl derivatives thereof by (1) esterifying an  $\alpha$ -amino- $\beta$ -hydroxypropiohydroxamic acid derivative of stated general formula, (2) hydrogenolysing the resulting ester and (3) treating the resulting hydroxamic acid with a basic agent, e.g. gaseous ammonia.

### Conversion of Hydrocarbons

N.V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ *British Patent 796,048*

Platinum in the form of wire or gauze or supported on silica or alumina is used as a catalyst in a hydrocarbon conversion process involving the contacting of a mixture of a hydrocarbon containing at least 2 carbon atoms and hydrogen iodide in vapour phase at 300-800°C with the catalyst to form a mixture of the hydrocarbon and iodine, which mixture is then subjected to a temperature of 300-800°C to convert the hydrocarbon and iodine to a different hydrocarbon containing a new carbon-to-carbon linkage, and hydrogen iodide.

### Catalysts

CALIFORNIA RESEARCH CORP. *British Patent 796,175*

A catalyst comprising one or more catalytic metals dispersed on the surface of a porous support of surface area of at least 25 sq. m/g is made by wetting the support with a dispersion in a liquid carrier of an active catalytic metal chelate or chelates having an amino acid, e.g. ethylene-diamine tetraacetic acid, as the chelating agent, drying and calcining to decompose the chelate. Platinum chelate may be used, preferably free from halide. Alumina, silica-alumina etc. may be used as the support.

### Refining and Reforming of Hydrocarbon Oils

UNIVERSAL OIL PRODUCTS CO. *British Patent 796,393*

A hydrogenation catalyst of platinum or palladium

either alone or composited with silica, alumina, magnesia or zirconia, is used in a combination process of refining and reforming hydrocarbon oils containing poisonous gasoline hydrocarbons.

### Field Emission Electron Gun

M. VON ARDENNE *British Patent 796,424*

A field emission electron gun of high brightness comprises a principal cathode of tantalum or magnesium, an auxiliary electrode of platinum surrounding the principal cathode and an anode arranged adjacent to the cathode.

### Manufacture of Hydrogen Peroxide

COLUMBIA-SOUTHERN CHEMICAL CORP. *British Patent 796,461*

A catalyst composed of a Group VIII metal, supported on an alkaline earth metal phosphate, e.g. calcium phosphate, is used in a process of manufacturing hydrogen peroxide involving hydrogenation of an anthraquinone compound. The catalyst metal is preferably palladium.

### Reductive Alkylation

UNIVERSAL OIL PRODUCTS CO. *British Patent 797,224*

N-alkylated aromatic amines are produced by reacting in a reaction zone hydrogen, an aromatic compound, having amino- and/or nitro-substitution, and a ketone or aldehyde in the presence of a reduction alkylation catalyst at 230-350°F and a total pressure of 100-3,000 lb/sq. in. A platinum alumina catalyst is used.

### New Amines

E.I. DU PONT DE NEMOURS & CO. *British Patent 797,241*

N:N<sup>1</sup>-bis [(1-amino-cycloalkyl) methyl] alkylene diamines are prepared by catalytically hydrogenating N:N<sup>1</sup>-bis [(1-nitrocycloalkyl) methyl] alkylene diamines, using a supported platinum, palladium or rhodium catalyst.

### Novel Amines

E.I. DU PONT DE NEMOURS & CO. *British Patent 797,244*

Bis (1-aminocycloalkyl-methyl) amines are prepared by catalytically hydrogenating bis (1-nitrocycloalkylmethyl) amines, using a supported platinum, palladium or rhodium catalyst.

### Hydrogenation of Nitrosamines

HERCULES POWDER CO. *British Patent 797,483*

A hydrazine is produced by reacting a nitrosamine with hydrogen at above atmospheric pressure and in the presence of particulate catalytic material (Group VIII metal, preferably palladium, platinum, rhodium or iridium). The catalyst has a specific surface area of at least 15 sq. m per gram. Platinum or palladium supported on carbon may be used.

### Variable Electric Resistor Device

G. V. PLANAR LTD. *British Patent 797,676*

In a variable electric resistance of the stepped or discontinuous type comprising a conducting film, e.g. a gold-platinum alloy arranged in a meandering pattern, photographically produced, on a base, the track of the contact wiper is reinforced by electrically conducting material arranged in a number of discrete areas so as to overlay and/or underlay portions of the conducting film. The reinforcement may consist of rhodium, gold, silver or copper or a composite of any of them. The contact wiper surface preferably consists of rhodium.

### I.C.E. Exhaust Purifier

OXY-CATALYST INC. *British Patent 797,777*

Exhaust gases of internal combustion engines operated on leaded gasoline are catalytically oxidised by passing them over a bed of pellets of catalyst of average dimension of 1/32–3/8 inch. The temperature and velocity of the gases is maintained such that the average temperature of the bed is 700–1400°F, but not over 1500°F under normal operating conditions. Decrease of activity of the catalyst due to accumulation of lead compounds is thus much less drastic. The catalyst is platinum-on-activated alumina.

### Preparation of Ketoximes

E.I. DU PONT DE NEMOURS & CO. *British Patent 797,985*

A ketoxime is prepared by the catalytic hydrogenation of a nitro-cyclo alkane or secondary nitro alkane. The catalyst may consist of any of the following: a mixed palladium-platinum-ferric oxide catalyst supported on acetylene black; mixed palladium-ferric oxide on acetylene black; palladium on acetylene black, carbon, calcium sulphate or alumina; platinum on carbon.

### Preparation of Deuterated Hydrocarbon Materials

N.V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ *British Patent 798,030*

Wholly or partly deuterated hydrocarbon material is made by flowing normally liquid or solid hydrocarbon material downwardly over the surface of catalyst material in a reaction zone, held at elevated temperature and pressure, and passing a gas, i.e. deuterium and/or deuterium compounds into the zone at the same time, the catalyst promoting the exchange of deuterium atoms for hydrogen atoms in the hydrocarbon material. Platinum or palladium catalyst supported on silica gel pumice etc. or an alumina catalyst may be used.

### Manufacture of Anthracenes

IMPERIAL CHEMICAL INDUSTRIES LTD. *British Patent 798,167*

A catalyst consisting of platinum on alumina or

palladium on carbon may be used in a process of manufacturing anthracene by cyclisation of diarylmethanes of specified structure by heating the diarylmethanes in the absence of oxygen and in the presence of the catalyst.

### Spinning Nozzles

COURTAULDS LTD. *German Patent 1,012,427*

Each spinning opening in a spinning nozzle for the production of artificial fibres is located in a recess, formed on the outer side of the relatively thick nozzle base, of such shape that the angle between the base of the recess and the wall of the opening is not greater than 90°, the depth of the recess is not greater than 0.038 mm and the openings are several times longer than the depth of the recesses. The nozzles may be made of platinum or an alloy thereof with rhodium, iridium or palladium.

### Method of Flameless Decomposition of Gaseous Hydrocarbons to CO and Hydrogen

BADISCHE ANILIN & SODA FABRIK A.G. *German Patent 1,013,631*

The catalyst employed in the first stage of a method of flameless decomposition of gaseous hydrocarbons consists of a heat-resistant inert granular material carrying 0.003–0.15 wt.% of platinum.

### Platinum Alloy

JENAER GLASWERK SCHOTT & GEN. *German Patent 1,014,331*

A platinum alloy contains 0.02–0.2%, preferably 0.03–0.05%, tellurium and/or selenium and remainder platinum. The alloy is readily workable. Suitable for glass melting apparatus, spinning nozzles etc.

### Electrolyte for the Galvanic Deposition of Tungsten and/or Molybdenum

N.V. PHILIPS GLOEILAMPENFABRIEKEN. *German Patent 1,014,406*

A vessel or tank made of platinum group metal is used for the electrolyte for the galvanic deposition of tungsten and/or molybdenum.

### Protecting Metal Surfaces against Corrosion

A. HUET *German Patent 1,019,138*

A coating for protecting metal surfaces, such as heat exchangers, against corrosion consists of fine glass particles of spherical shape to which may be added catalytically active particles, such as platinum sponge.

### Production of Platinum Metal-Alumina Catalysts

ESSO RESEARCH & ENGINEERING CO. *German Patent 1,020,609*

In the production of platinum metal catalysts by impregnating alumina carriers – which are pro-

duced by hydrolysing aluminium alcoholate in the presence of a peptising medium, extruding the dried alumina hydrosol and calcining the product - with solutions of platinum metal compounds, a large amount of water is circulated by agitation of the carrier and to which the solution of platinum metal compound is slowly added in predetermined amount, the circulation being continued for at least half an hour and the catalyst finally separated, dried and calcined.

### Hydroisomerisation Process

GULF RESEARCH & DEVELOPMENT CO. *U.S. Patent* 2,831,908

A gasoline boiling range hydrocarbon charge comprising at least 85 vol % n-pentane and remainder other open chain paraffinic hydrocarbons is contacted in admixture with hydrogen (100-400 standard cu. ft. of hydrogen per barrel of hydrocarbon in the charge) with a supported platinum catalyst (0.1-5% by weight of platinum) at 600-900°F, a pressure of 20-2,000 lb/sq. in. gauge and at a charge liquid hourly space velocity of 8-12 vol per vol of catalyst per hour.

### Platinum Catalyst

SHELL DEVELOPMENT CO. *U.S. Patent* 2,834,740

A supported platinum-containing catalyst (0.1-1% platinum on a chloride-free carrier) is made by impregnating the carrier with an aqueous solution of tetrammine platinous hydroxide, drying and calcining to decompose the compound to the metal.

### Preparation of Diethyl Ketone

ESSO RESEARCH & ENGINEERING CO. *U.S. Patent* 2,839,580

Diethyl ketone is prepared by reacting ethylene, carbon monoxide and a hydrogen donor in the presence of a catalytic amount of a rhodium-containing catalyst capable of being dissolved in the reaction mixture under the conditions of the reaction, which include temperature of 100-300°C and pressures of 1,500-3,000 p.s.i.g.

### Spinnerets

AMERICAN ENKA CORP. *U.S. Patent* 2,839,783

A spinneret for high pressure melt-spinning of high polymeric substances consists of a base metal, e.g. stainless steel, spinneret element having holes therein and provided with noble metal inserts in the holes in the bottom plate of the spinneret. The holes include two portions of different conicity, that portion of the hole in direct contact with the insert having a conicity of 1°-6°. The inserts are formed of a 70% gold and 30% platinum alloy.

### Catalysts

STANDARD OIL CO. *U.S. Patent* 2,840,527

An alumina-based hydroforming catalyst of improved activity is made by commingling a platinum compound with alumina in amount of 0.05-1% by wt. of platinum based on dry  $Al_2O_3$ , drying and calcining to a water content of 1-30% by wt., dry basis, introducing into the dried and calcined mixture 0.001 mole per mole of dry  $Al_2O_3$  of aluminium sulphate, -nitrate, -chloride, -bromide, or -iodide and then removing any added solvent and calcining.

### Reforming Catalyst

HOUDRY PROCESS CORP. *U.S. Patent* 2,840,528

In the preparation of dual-function reforming catalyst granules consisting of 0.1-2% of platinum on a silica-alumina or alumina carrier having controlled acidic function distributed over a large surface area in excess of 5 sq./m/g, granular particles of the carrier are impregnated with chloroplatinic acid, the granules are dried to a water content below 1 mg/sq.m. of surface area and subjected to a gaseous sulphiding agent in excess of 1/6th the weight of the platinum content of the granules and in the absence of steam, whereby at least part of the chloroplatinate is sulphided. The particles are then subjected to a hydrogen-containing gas stream at 500-1000°F to form the platinum catalyst.

### Platinum-alumina Catalyst

UNIVERSAL OIL PRODUCTS CO. *U.S. Patent* 2,840,532

Alumina is impregnated with a homogeneous aqueous solution of a water-soluble platinum compound and an acidic compound selected from nitric acid, sulphuric acid, phosphoric acid, aluminium nitrate, acetic acid, oxalic acid, formic acid and propionic acid in an amount to give the solution a pH of below 2.5.

### Preparation of 4-Ketooctanedioic Acids

E.I. DU PONT DE NEMOURS & CO. *U.S. Patent* 2,840,608

A palladium catalyst is used in a process of preparing 4-ketooctanedioic acids by reacting hydrogen in an acidic medium of pH less than 2 and in contact with the catalyst, with a dilactone of specified general formula.

### Hydrogenation Process

E.I. DU PONT DE NEMOURS & CO. *U.S. Patent* 2,840,609

A platinum catalyst is used in the preparation of 1,8-octanedioic acid by hydrogenating, in solution in a polar organic solvent and in contact with the catalyst, a dilactone of specified general formula.