compound without hydrogenolysis of the -OH group and without change in rotation.

A Thermocouple Method of Studying Oxidation Reactions, Part I—Photosensitised Oxidation of Cyclohexene

J. C. ROBB and M. SHAHIN, *J. Inst. Petroleum*, 1958, **44**, (Sept.), 283-290

The non-stationary state of oxidation reactions is followed by measuring the rise in temperature of the reacting mixture using an apparatus incorporating a Pt/13%Rh-Pt thermocouple. The rate of oxidation is directly proportional to the rate of temperature rise. The method is used to study the kinetics of the photo-chemical oxidation of cyclohexene.

CATHODIC PROTECTION

The Anodic Polarisation of Lead-Platinum Bi-Electrodes

L. L. SHREIR and A. WEINRAUB, *Chem. & Ind.*, 1958, (Oct. 11), 1326-1327

Anodic polarisation of pure Pb anodes in chloride solutions results in formation of a voluminous deposit of $PbCl_2$. At 2.5 A/sq.dm the voltage increases rapidly and further electrolysis results in greater voltage increase and rapid corrosion of Pb. The insertion of a small micro-electrode of Pt into the Pb surface prior to polarisation results in the formation of an adherent coating of PbO_2 and this anode can be used for prolonged periods at high current densities. A Pb-Pt bi-electrode causes conversion of $PbCl_2$ to PbO_2 with a corresponding increase in voltage. The presence of Pt in the Pb surface is considered to increase the lead/interface potential to a value where $PbCl_2$, Pb^{2+} and Pb are oxidised to PbO_2. Bi-electrodes of Pb and Pb alloys with Pt may have practical application as inert anodes in cathodic protection and other electrolytic processes.

TEMPERATURE MEASUREMENT

The Use of Oxygen in a Modified Tilting Furnace. Roof Temperature Measurement

J. PURDIE, *J. Iron Steel Inst.*, 1958, **190**, (Sept.), 17-19

The thermocouples 5%Rh-Pt/20%Rh-Pt and 20%Rh-Pt/40%Rh-Pt were tested for use in oxygen-blown steel furnaces. The former can be used for short periods up to 1760° and the latter up to 1850°C. The best sheath materials were found to be Al_2O_3, MgO and spinel ($MgO.Al_2O_3$). Preliminary tests carried out with a 5%Rh-Pt/20%Rh-Pt thermocouple in a tilting furnace during normal working showed that the thermocouple must protrude $\frac{1}{2}$-1 in. inside the furnace roof. The best sheath material was spinel with an inner sheath of MgO which had a life of 6.5 days. With a 20%Rh-Pt/40%Rh-Pt couple in an 'Ajax' furnace during oxygen blowing the best results were obtained using a recrystallised Al_2O_3 sheath with the thermocouple protruding $\frac{1}{2}$ in. into the furnace. MgO and spinel sheaths were less satisfactory in this furnace due to spalling and increased attack by Fe_3O_4 in the furnace atmosphere. With the degree of control now possible the roof temperature rarely exceeds 1700°C so that 5%Rh-Pt/20%Rh-Pt couples may be used.

NEW PATENTS

Production of Higher Ketones

ESSO RESEARCH AND ENGINEERING CO. *British Patent* 798,838

Ketones are synthesised by contacting 2,3-epoxybutane or a saturated epoxide containing 5–16 carbon atoms in its molecule and having its oxygen atom joined to a secondary carbon atom, at an isomerisation temperature of 200–500°C with a Group I-B or VIII metal catalyst supported on activated C. The catalyst may contain 1–20 wt% of Pt or Pd.

Hydrogenation

ENGELHARD INDUSTRIES INC. *British Patent* 799,396

An unsaturated material is contacted under hydrogenating conditions with hydrogen and a Pt-calcined Al_2O_3 catalyst. The catalyst has a large pore, high surface area base structure composed of γ-Al_2O_3 modifications resulting from drying and calcining a mixture of precursor hydrous Al_2O_3 phases containing 65-95% of trihydrate, the amount of Pt being 0.1-1% by wt. in sufficiently divided form as to exhibit, by X-ray diffraction studies, the substantial absence of crystallites and crystals of size greater than 50Å.

Manufacture of Catalysts

UNIVERSAL OIL PRODUCTS CO. *British Patent* 799,459

An Al_2O_3 carrier is impregnated with a solution of a Pt compound and the composite is calcined, without the addition of halogen, at 250-595°C with a free oxygen-containing gas to which water has been added in amount to impart to the gas at least 15.78 g of water per kg of dry gas.

Production of Polyhydric Compounds

RUHRCHEMIE A.G. *British Patent* 799,753

Reference is made to the use of roughened Pt sheet of about 2 g wt for the purpose of decomposing excess H_2O_2 formed in a process of producing a polyhydric compound by hydroxylating an organic compound having an olefinic double bond with an aqueous solution of H_2O_2 at not less than 40°C in the presence of isopropyl formate.

Production of Hydrogen Peroxide

L'ETAT FRANCAIS *et al. British Patent* 799,756

In the production of H_2O_2, the catalyst comprises active catalyst material (reduced metal) uniformly distributed throughout a water-hardened catalytically inert and porous refractory cement having closed cells and an apparent density of 0.7–1.3 g/cc. The catalyst metal may be Pd.

Catalytic Conversion of Hydrocarbons

SOCONY MOBIL OIL CO. INC. *British Patent* 799,811

A catalyst consists of a mechanical mixture of a Pt-containing component deposited on an inert porous support and of an acidic component having a catalytic cracking activity. 0.1–2% by wt of Pt is used. The support may be silica gel and the acid component commercial silica-alumina cogel cracking catalyst. Used for hydrocarbon conversion processes.

Reforming Catalyst

W. R. GRACE AND CO. *British Patent* 799,827

A gasoline reforming catalyst is made by impregnating a silica gel base with an aqueous solution of aluminium chloroplatinate or a mixed solution of chloroplatinic acid and an Al salt, the anion of which is removed from the catalyst during subsequent conversion of the Pt compound to metallic Pt. The aqueous solution is sufficiently concentrated to form a final catalyst containing 0.05–5 wt% of Pt and 0.01–1 wt% of Al_2O_3. $AlCl_3$ may be used.

Hydrogenation Catalysts

E. I. DU PONT DE NEMOURS AND CO. *British Patent* 799,871

A hydrogenation catalyst comprises an oleophilic C support (oil absorption factor at least 200) and Pd and/or Pt deposited on its surface at a loading of about 0.1–10% by wt of the support. The Pd or the Pd component of the mixture is activated with a metal oxide, carbonate or hydroxide.

Hydrogenation of Dihydrophthalic Acid Esters

OLIN MATHIESON CHEMICAL CORP. *British Patent* 800,287

A 3,6-oxy-3,4,5,6-tetrahydrophthalic acid diester is produced by reacting a diester of 3,6-oxy-3, 6-dihydrophthalic acid with hydrogen in the presence of a hydrogenation catalyst and a solvent comprising a liquid chlorinated hydrocarbon. The catalyst used may be Pd or Pt on Al_2O_3 or C, Ru-Al_2O_3 or Rh.

Catalytic Reforming

SOCONY MOBIL OIL CO. INC. *British Patent* 800,339

A halogen-free reforming catalyst is composed of 0.05–2% by wt of Pt or Pd deposited on Al_2O_3 which has composited therewith 7–16% by wt of SiO_2 and which has an activity index of between 6 and 20. Method of reforming petroleum hydrocarbon fractions to produce high octane fuel also described.

Flux-free Bonded Assemblies

CORNING GLASS WORKS *British Patent* 800,519

The bond between a quartz and another inorganic part, e.g. SiO_2, includes a film of Pt on one part through which In is diffused into the part. The quartz part has a Au-Pt alloy adjacent to it.

Substituted Alkylamines

N. V. PHILIPS GLOEILAMPENFABRIEKEN *British Patent* 800,718

A noble metal hydrogenation catalyst, e.g. a Pd-C catalyst, is used at one stage in the preparation of novel substituted alkylamines.

Enrichment of Water in Deuterium Oxide

UNITED KINGDOM ATOMIC ENERGY AUTHORITY *British Patent* 800,730

A method of enriching water in deuterium oxide consists in bringing together water and deuterium-containing hydrogen in the presence as catalyst for the exchange reaction of *inter alia* Ru, Rh, Pd, Os, Ir or Pt.

Potentiometers

ENGELHARD INDUSTRIES INC. *British Patent* 801,034

A precision potentiometer has a resistance winding composed of Pd, with or without Pt, and 1–10% Cr and a brush member composed of a precious metal based alloy, e.g. 20% Ir-Pt or alloys of Pt with not more than 15% W or Mo.

Aromatisation of Light Naphthas

ESSO RESEARCH & ENGINEERING CO. *British Patent* 801,060

High octane number, highly aromatic products are obtained from hydrocarbon fractions boiling in the range of 110–250°F, by contacting the fractions, mixed with a hydrogen-rich gas, with a catalyst comprising a Pt metal-Al_2O_3 at 800–975°F and pressures below 125 p.s.i.g., maintaining contact for a time sufficient to produce a C_5 plus hydroformate having a Research clear octane number of about 80, separating the hydroformate from the normally gaseous materials, subjecting it to solvent extraction to obtain an aromatic-rich extract phase, and an aromatic-poor raffinate, which is recycled.

Preparation of Uranium Compounds

UNITED KINGDOM ATOMIC ENERGY AUTHORITY *British Patent* 801,380

A trivalent uranium compound is made by reducing a salt of uranium (valency of uranium higher than 4) by treating a solution of the salt with hydrogen in the presence of a Pt, Pd or Rh catalyst.

Manufacture of Aliphatic Dinitriles

THE GOODYEAR TIRE AND RUBBER CO. *British Patent* 801,406

The anodes of electrolytic cells used in the manufacture by electrolysis of aliphatic α, ω-dinitriles of given general formula have at least their surface composed of Pt or Ir.

Carbonylation Synthesis Reaction

ESSO RESEARCH & ENGINEERING CO. *British Patent* 801,734

Oxygenated compounds are produced by reaction of an olefin, carbon monoxide and hydrogen at temperatures of 60–240°C and pressures of 1,000–10,000 p.s.i.g. in the presence of a Rh-containing catalyst capable of being dissolved in the reaction mixture under the reaction conditions. Rh_2O_3, RhO_2 or $RhCl_3$ may be used, supported on an inert carrier.

Hydrogenation of Acetylene in Ethylene

THE DOW CHEMICAL CO. *British Patent* 802,100

The acetylene contained in a gaseous mixture of ethylene and acetylene is selectively hydrogenated by contacting the mixture together with hydrogen in proportion greater than 1 mole of hydrogen per mole of acetylene, with a catalyst containing an effective proportion of a material comprising 60–99 parts by wt of Pd and 40–1 parts of Cu, Ag or Au. The preferred catalyst contains 70–99 parts of Pd and 30–1 parts of Ag. Method of making the catalyst by impregnating a solid carrier with an aqueous solution of $Pd(NO_3)_2$ and $AgNO_3$ is described and claimed.

Production of Lubricating Oils

TEXACO DEVELOPMENT CORP. *British Patent* 802,194

Hydrocarbon lubricating oil stock is subjected in mixture with hydrogen and in the presence of a catalyst composed of cobalt molybdate or nickel sulphide and tungsten sulphide to hydrogenating conditions to convert it to an intermediate product of improved viscosity index, at least part of the product is then hydrogenated in the presence of a Pt catalyst, e.g. Pt, Al_2O_3 and a combined halogen, such as aluminium fluoride, at 400–650°F and a pressure of 750–5,000 p.s.i.g. to improve the oxidation stability.

Melting of Glass

CARL-ZEISS-STIFTUNG *British Patent* 802,310

In a glass melting process employing a tank furnace for melting and a separate homogeniser, the tube connecting the bottom of the furnace to the homogeniser is made of Pt, preferably surrounded by a ceramic casing. The tube is heated by passage of direct current through the metal part thereof.

Recovery of Uranium from Ores

COMMISSARIAT A L'ENERGIE ATOMIQUE *British Patent* 802,452

Uranium is recovered from an ore by forming, from the ore, a clear solution of a hexavalent uranyl-alkali metal or -ammonium carbonate solution, precipitating hydrated UO_2 from the solution by forming nascent hydrogen therein and separating this oxide. The nascent hydrogen is produced by subjecting the solution to electrolysis in the presence of a hydrogenation catalyst, e.g. Pt or Pd.

Hydroforming

ESSO RESEARCH & ENGINEERING CO. *British Patent* 802,943

In a hydroforming process, the fresh feed, comprising naphtha and hydroformate (the latter boiling above 120°F) is preheated to 800–1050°F and the hydrogen-rich recycle gas is preheated to 800–1400°F before charging to the reactor in which the catalyst, e.g. Pt, is fluidised; the reactor operates at 50–1,000 p.s.i.g. and a temperature of 750–1150°F.

Manufacture of Hydrogen Peroxide

LAPORTE CHEMICALS LTD. *British Patent* 803,121

Hydrogen peroxide is made by hydrogenating an anthraquinone sulphonic acid ester in solution in the presence of a hydrogenation catalyst, oxidising the reduced compound formed with gaseous oxygen, regenerating the original ester and forming H_2O_2, which is then separated. The catalyst is preferably Pd-activated Al_2O_3.

Production of Melamine

MONSANTO CHEMICAL CO. *British Patent* 803,195

A Pt or Pd catalyst may be used in the production of melamine by heating a mixture of HCN and NH_3 gas at 400–750°C and at atmospheric pressure in the presence of the catalyst.

Production of Nitric Acid

IMPERIAL CHEMICAL INDUSTRIES LTD. *British Patent* 803,211

Pt gauze catalyst is used in the production of HNO_3 by a method involving the oxidation of NH_3 resulting in the more efficient recovery of available heat.

Production of Hydrogen having Increased Deuterium Content

E. W. BECKER *British Patent* 803,274

Water or hydrogen of increased deuterium content is made by passing hydrogen and liquid water

counter-currently at high pressure and in the presence of a catalyst through two columns at different temperatures. Water passes first through the cold column and then through the hot column and the hydrogen *vice versa*. The catalyst is passed in a closed circuit dispersed in the water, preferably in the form of a Pt or Pd sol.

Composite Rod

ENGELHARD INDUSTRIES INC. *British Patent* 803,318

A composite rod or electrode is made by telescoping a refractory metal, e.g. W or Mo, core within a Pt metal sheath, so as to leave a portion of the latter extending beyond the core. This extension is then gripped and the sheath snug-fitted on the core. The extension is removed and the cross-sectional area of the rod reduced. Pt is preferably used to form the sheath.

Catalyst

THE BRITISH PETROLEUM CO. LTD. *Belgian Patent* 560,052

A catalyst for use in the conversion of hydrocarbons consists of a major proportion of Al, a small proportion of Pt and more than 3% by weight of combined F based on the total weight of the catalyst.

Isomerisation of Paraffinic Hydrocarbons

SHELL DEVELOPMENT CO. *U.S. Patent* 2,841,626

Paraffinic hydrocarbons are isomerised by contacting a feed of hydrocarbon having less than 7 carbon atoms in the presence of added hydrogen and 100 mole% of water vapour based on the hydrocarbon, with a Group VIII noble metal catalyst on an aluminous carrier and containing less than 0.1% of chlorine at a temperature of 480–890°F and a pressure of 1–100 atm.

Hydrogenation of Pyrazine

THE DOW CHEMICAL CO. *U.S. Patent* 2,843,589

Pyrazine, alkyl pyrazines, in which each alkyl substituent contains not more than 4 carbon atoms, and mixtures thereof are hydrogenated to corresponding piperazine products by the action of hydrogen in the presence of a hydrogenation catalyst, e.g. supported Rh or Pd at temperature between 125°C and 225°C and a pressure of 200–600 lb/sq. in.

Hydroforming Catalyst

J. A. HINLICKY *U.S. Patent* 2,844,542

A Pt-Al_2O_3 catalyst is made by combining in solution of an alcohol having 3–5 carbon atoms per molecule a minor amount of a Pt salt and a relatively large amount of an aluminium alcoholate and precipitating Pt from the solution by addition of NH_4OH, drying the deposit to obtain a hydrous Al_2O_3 having a finely divided Pt deposit colloidally dispersed throughout, which is reduced to Pt. 0.05–5% by wt Pt-Al_2O_3 is preferred.

Catalytic Hydrogenation

MONSANTO CHEMICAL CO. *U.S. Patent* 2,844,573

Polyvinyl chloride is catalytically hydrogenated by reacting it with hydrogen in the presence of a Pd catalyst under sufficiently high temperature and pressure and for long enough to replace at least 20% of the chlorine atoms with hydrogen atoms.

Joining Refractory Metals

SYLVANIA ELECTRIC PRODUCTS INC. *U.S. Patent* 2,844,868

Cathodes are made by coating a cylindrical metal base with a thin layer of Ru powder, wrapping a Mo mesh round the coated base and heating the assembly to 1150–1400°C to form a preliminary bond between the base and the mesh and finally heating the assembly to 1900–2100°C.

Fluid Hydroforming Process

ESSO RESEARCH & ENGINEERING CO. *U.S. Patent* 2,846,364

A fluidised bed comprising a mixture of a major proportion of finely divided Pt metal hydroforming catalyst and a minor proportion of inert heat transfer solid particles or shot is used in a process of reforming hydrocarbon fractions boiling within the gasoline range.

Reforming Catalyst

PHILIPS PETROLEUM CO. *U.S. Patent* 2,848,510

Fluid hydrocarbon material is catalytically converted to more valuable hydrocarbons by contacting the material with a catalyst comprising Pt or Pd (0.1–5 wt%), manganese oxide (1–20 wt%) and balance a cracking component under conversion conditions.

Catalytic Reforming Process

THE ATLANTIC REFINING CO. *U.S. Patent* 2,849,377

Describes improvements in the control of a process for catalytically reforming a hydrocarbon fraction boiling within the gasoline-kerosine range to increase the anti-knock value in the presence of hydrogen and a catalyst composed of an acidic metal oxide component impregnated with Pt or Pd.

Catalytic Reforming

SOCONY MOBIL OIL CO. INC. *U.S. Patent* 2,849,378

Hydrocarbon fractions having an initial boiling point of at least 60°F and an end boiling point of less than 450°F are reformed by contact with a catalyst of surface area varying between 650 sq.m/g and 100 sq.m/g and comprising Pt or Pd (0.05–2% by wt) deposited on SiO_2 that contains Al_2O_3 in amount of 0.025% to 7% based on the weight of catalyst, the relationship

between the amount of Al_2O_3 and the surface area defining a point on a graph.

Cyclisation of Aliphatic Hydrocarbons

THE M. W. KELLOGG CO. *U.S. Patent* 2,849,504

A naphtha fraction containing a straight chain hydrocarbon, i.e. a C_5 and/or a C_6 hydrocarbon, is subjected to separation to remove a straight chain hydrocarbon therefrom, the remainder is heated with a Pt catalyst under reforming conditions to produce a high octane gasoline. The straight chain saturated hydrocarbon is contacted with a 5–25% by wt Pt catalyst at 250–420°C, a pressure of 1–7 p.s.i.a. and a wt space velocity of 0.001–2 to produce a cyclopentane compound, which is then combined with the remainder of the naphtha fraction.

Catalysts

STANDARD OIL CO. *U.S. Patent* 2,851,399

A petroleum naphtha is contacted under hydroforming conditions with a catalyst composed of 0.05–1% by wt of Pt, based on dry Al_2O_3, and 0.01–1 atom of Se per atom of Pt, supported on Al_2O_3.

Purifying of Catalysts

U.S. ATOMIC ENERGY COMMISSION *U.S. Patent* 2,851,427

A finely divided Pt catalyst containing small amounts of chlorine is purified by treatment at elevated temperature with dry hydrogen and then with wet hydrogen having a hydrogen-water vapour ratio of 8:1. This treatment is repeated as required.

Reaction Process

UNION CARBIDE CORP. *U.S. Patent* 2,851,473

Organosilicon compounds are made by reacting an unsaturated organic compound containing at least one non-aromatic multiple carbon to carbon bond with a silane containing at least one silanic hydrogen bond, the reaction being catalysed with Pt-γ-Al_2O_3.

Preparation of Reforming Catalysts

W. R. GRACE & CO. *U.S. Patent* 2,852,472

A gasoline reforming catalyst is made by impregnating calcined Al_2O_3 with SiF_4 or fluosilicic acid, in amount to incorporate 0.03–3 wt% SiO_2 and 0.1–8 wt% F in the final catalyst, commingling the treated Al_2O_3 with an aqueous solution of chloroplatinic acid in amount sufficient to give 0.1–2% Pt in the catalyst, drying and converting to the metal.

Manufacture of Catalysts

ESSO RESEARCH & ENGINEERING CO. *U.S. Patent* 2,852,474

Catalysts are made by mixing particles of a dry adsorptive support with an aqueous solution of a Pt or Pd compound containing a small amount of a non-ashing, wetting and dispersing agent in amount sufficient to deposit 0.1–2 wt% of Pt or 0.5–5 wt% of Pd on the support and fixing the metal thereon.

Reforming Catalyst

W. R. GRACE & CO. *U.S. Patent* 2,853,456

A gasoline reforming catalyst is made by contacting amalgamated Al with a dilute aqueous solution of H_2O_2, separating the newly formed Al_2O_3 from the supernatant liquid, contacting it with a solution of chloroplatinic acid and fluosilicic acid in amount to form a final catalyst containing 0.1–1% Pt, 0.01–1% F and 0.01–1% SiO_2, drying and converting to metallic Pt.

Hydrocracking and Catalyst therefor

SOCONY MOBIL OIL CO. INC. *U.S. Patent* 2,854,401

A hydrocarbon charge is contacted in the presence of hydrogen with a catalyst consisting of a mechanical mixture of particles of less than 100 microns diameter of (1) a porous inert carrier on which is deposited 0.05–10% by weight of a Pt metal and (2) an acidic cracking component.

Catalytic Reforming

SOCONY MOBIL OIL CO. INC. *U.S. Patent* 2,854,403

A reforming catalyst is made by mixing particles of less than 100 microns diameter of Al_2O_3 and of an acidic SiO_2-Al_2O_3 cracking component, pelletising the mixture under pressure with a binder, removing the latter by combustion, contacting the composite with a solution containing Pt as an anion long enough to allow the pores of the composite to be filled, and, when the Pt reaches an adsorption equilibrium between Al_2O_3 and SiO_2, drying and reducing to the metal.

Catalytic Reforming

SOCONY MOBIL OIL CO. INC. *U.S. Patent* 2,854,404

A hydrocarbon mixture boiling in the gasoline range is reformed by contacting it under reforming conditions with a catalyst composed of a mechanical mixture of particles of less than 100 microns diameter of (1) a porous inert carrier carrying Pt metal in amount such that the final catalyst content of Pt is 0.05–5% by wt and (2) halogen activated η-Al_2O_3 (halogen content 0.1–8% by wt). The relative wt fractions of the two components are about 0.1 and 0.9. The catalyst has a dehydrogenation activity of at least 50 and an acid activity of at least 0.2.

Manufacture of Carbinols

THE DISTILLERS CO. LTD. *U.S. Patent* 2,854,487

A Pd-activated Al_2O_3 catalyst is used in a process for the manufacture of carbinols by reaction at 40–120°C of an organic hydroperoxide with hydrogen. 1–20% by wt, based on the wt of the hydroperoxide, of the catalyst is used.