

Vacuum Radiation Furnace with Precise Control of Temperature Gradients for Crystal Growth by Sublimation

A. C. PRIOR, *J. Sci. Instr.*, 1961, **38**, (5), 198-201

The furnace, designed to operate between 700°C and 850°C, is described in detail. Temperature gradients in the centre of the furnace, which do not exceed about 0.1°C/cm, are measured by a five-fold differential system of 1% Rh-Pt:13% Rh-Pt thermocouples. A high degree of temperature uniformity and rapid response times are achieved by minimising the mass of heated material and by the use of water-cooled reflecting Ag surfaces for the walls. A regulating system controls the temperature to 0.1°C.

Effect of Pressure on E.M.F. of Thermocouples

F. P. BUNDY, *J. Appl. Phys.*, 1961, **32**, (3), 483-488

Thermal e.m.f.'s were measured for Pt, 10% Rh-Pt, Ni, Cu, Constantan, Alumel, Chromel, and 18% Mo-Ni at pressures up to 72 kbar for a

ΔT of 100°C. From these results, corrections due to pressure are deduced for common thermocouples made of pairs of these metals. Several thermocouple pairs were compared at pressures up to 58 kbar and temperatures up to 1200°C. Below 200° to 300°C, pressure deviations between them may be correlated with data on single metals taken at a ΔT of 100°C, but at higher temperatures the deviations decrease and generally reverse.

Thermocouple Calibration

ANON., *Instr. Practice*, 1961, **15**, (4), 428-431

Methods of calibration used at the National Bureau of Standards are described in detail. Base metal thermocouples are compared with a Pt resistance thermometer over the range -190° to 538°C and with a standard 10% Rh-Pt:Pt thermocouple up to 1100°C. Pt thermocouples are compared with the standard thermocouple, or are calibrated at fixed points on the International Temperature Scale. Thermocouple wires of various compositions and potentiometers also are calibrated.

NEW PATENTS

Electrical Resistance Alloy

JOHNSON, MATTHEY & CO. LTD. *British Patent* 861,646

A ductile stable high resistance alloy is composed of 16-90% palladium, 5-64% gold and 2.5-20% molybdenum apart from impurities.

Separating Palladium from other Platinum Group Metals

CANADIAN COPPER REFINERS LTD. *British Patent* 863,286

Palladium is separated from other platinum group metals by adding to an aqueous solution of compounds of palladium and other platinum metal or metals, maintained in an acid condition by the presence of a strong acid, a water-soluble iodide in amount at least stoichiometrically equivalent to the palladium present to form PdI₂ so as to precipitate the palladium as PdI₂, the solution containing a concentration of sulphur dioxide (0.3-4 g/l) sufficient to prevent the solution of PdI₂ by conversion thereof to PdI₄.

Separation of Platinum from other Metals

THE MOND NICKEL CO. LTD. *British Patent* 863,517

In the refining of platinum by precipitation as ammonium hexachloroplatinate from an acid chloride solution containing one or more other noble metals, the necessary ammonium ions are provided by hydrolysis of an amide in the

solution. Urea, acetamide or formamide may be used.

Catalytic Isomerisation

ENGELHARD INDUSTRIES INC. *British Patent* 863,688

A C₄ to C₆ *n*-paraffinic-containing hydrocarbon feed is isomerised by contacting it in vapour phase with a catalyst, containing 0.01-2% of a platinum group metal and 3-20% of boria supported on activated alumina, at 500-800°F, superatmospheric pressure and in the presence of hydrogen and while providing 1-25% by wt. based on the feed of a hydrogen halide (atomic number of halogen 35-85).

Isomerisation Catalyst

THE M.W. KELLOGG CO. *British Patent* 863,800

A catalyst for isomerisation processes is composed of an inorganic carrier, preferably alumina, rhodium, iridium, ruthenium or osmium, and at least 2% by wt. of a boron compound, preferably 5-25% of boron oxide. 0.05-5% by wt. of the platinum group metal is present.

Isomerisation Catalyst

THE M.W. KELLOGG CO. *British Patent* 863,801

A hydrocarbon is isomerised by subjecting it to contact under isomerisation conditions with a catalyst comprising platinum or palladium, alumina and at least 5% by wt. of boron oxide.

Platinum/Carbon Rods for Electron Microscopy

ASSOCIATED ELECTRICAL INDUSTRIES LTD. *British Patent 865,239*

Platinum/carbon rods are made by compacting a mixture containing by wt. 3-5 parts of platinum powder with 1 part of carbon powder, all the particles in the mixture being coated with organic compounds, and heating the so-formed rods to at least 500°C to remove oil (used as a die lubricant) and to decompose the organic compounds. Gum Arabic may be used, with linseed oil as lubricant.

Isomerisation of Paraffinic Hydrocarbons

ESSO RESEARCH & ENGINEERING CO. *British Patent 865,269*

Paraffinic hydrocarbons are isomerised without hydrocracking by contacting a mixture of a normal paraffin (5-8 carbon atoms in the molecule) and 1-15% by vol. based on the paraffin, of an aromatic hydrocarbon (in the gasoline boiling range) with a platinum catalyst at 600°-900°F in the presence of hydrogen to form an isomerised product. A 0.3-0.6 wt. % platinum-on-alumina catalyst is used.

Preparation of a Camphane Derivative

LEPETIT S.P.A. *British Patent 865,862*

3-methylaminoisocamphane is prepared by heating a mixture of 3-aminoisocamphane and a small excess over one equivalent amount of formaldehyde in a lower aliphatic alcohol at 100-125°C and pressure of 50 atms of hydrogen for 3-7 h in the presence of a palladium oxide catalyst. A palladium oxide-on-charcoal containing 4-6 wt. % of palladium is used in the ratio of 0.5-3 parts of palladium for 100 parts of 3-aminoisocamphane.

Removal of Acetylene from Air

ENGELHARD INDUSTRIES INC. *British Patent 866,816*

Acetylene is removed from air by passing a mixture of acetylene and air over a palladium-on-alumina catalyst at 100°-200°C and a pressure of about 1000 p.s.i.g.

Resistance Thermometers

ZAVODY PRUMYSLOVE AUTOMATISCHE N.P. *British Patent 867,431*

The supply conductors for the measuring resistors of a platinum resistance thermometer are formed of platinum-coated wire, preferably an iron-nickel alloy wire which imparts a total coefficient of thermal expansion to the conductors corresponding to that of hard glass materials used for sealing.

Electrolytic Apparatus

DAVID J. EVANS (RESEARCH) LTD. *British Patent 867,443*

An electrolytic cell includes two co-axial tube

electrodes of titanium, cooling water being supplied through the inner tube and electrolyte to the annular space between the tubes. One or both of the tubes is/are coated with platinum on the surface thereof contacting the electrolyte.

Palladium Catalyst

SOC. DES USINES CHIMIQUES RHONE-POULENC *British Patent 867,475*

A selective hydrogenation catalyst is formed of palladium and a stannous salt, e.g. the chloride, supported on a suitable carrier such as calcium carbonate. The tin to palladium atomic ratio is not less than 2 to 1. Made by mixing an aqueous solution of a palladium salt with the support material, reducing to metallic palladium and treating with an aqueous solution of stannous chloride in dilute hydrochloric acid. Intended for hydrogenation of acetylenic bonds to ethylenic bonds.

Catalysts

ENGELHARD INDUSTRIES INC. *British Patent 867,478*

In preparing a platinum-alumina catalyst by impregnating gamma alumina with platinum, the alumina is first contacted with water to rehydrate and convert 10-75% thereof to hydrous alumina, and the resulting hydrous alumina and gamma alumina mixture is then impregnated with the platinum.

Manufacture of Oil-soluble Petroleum Sulphonic Acids

LOBITOS OILFIELDS LTD. *British Patent 867,483*

Oil-soluble petroleum sulphonic acids are prepared by contacting a naphtha-containing petroleum oil with a hydrocarbon reforming catalyst (platinum-halogen-alumina) under dehydrogenating conditions to convert the naphthene components to aromatic hydrocarbons and then sulphonating the reformed oil to convert most of the aromatics to their sulphonated derivatives.

Metallising of Ceramics

COMPAGNIE FRANCAISE THOMSON-HOUSTON *British Patent 867,554*

In producing a joint between a ceramic body and a metal part by brazing or soldering the metal to a molybdenum layer sintered to the ceramic, the molybdenum is first coated with a layer of rhodium.

Reduction of Dextrose

ENGELHARD INDUSTRIES INC. *British Patent 867,689*

Dextrose is reduced to sorbitol by treating a solution thereof at super-atmospheric pressure with hydrogen in the presence of a catalyst composed of palladium and ruthenium (Pd=50-90% by wt. of the active metal) supported on carbon. A temperature of 90-130°C and pressure of 60-5000 p.s.i.g. are used.

Hydrocarbon Conversion Process

THE BRITISH PETROLEUM CO. LTD. *British Patent* 867,990

A hydrocarbon capable of molecular fission is passed over a catalyst consisting of a major proportion of alumina, a minor proportion of platinum and more than 6 wt.% of fluorine in combined form under conditions to cause fission of the hydrocarbon. The catalyst contains 0.01-1% of Pt based on weight of catalyst.

Hydrocarbon Conversion Process and Catalysts therefor

THE BRITISH PETROLEUM CO. LTD. *British Patent* 867,991

A catalyst is made by contacting an alumina gel (obtained by hydrolysis of an aluminium alkoxide) with hydrofluoric acid and with an aqueous solution of a water-soluble platinum-containing compound, drying and roasting (at 200-600°C), fluorine being added up to more than 6% based on total weight of catalyst. Tetra-amino platinous chloride is used. The alumina may first be peptised by addition of acetic acid.

Preparation of Disubstituted Nitrosamines

E. I. DU PONT DE NEMOURS & CO. *British Patent* 867,993

Palladium-on-charcoal or rhodium-on-charcoal may be used as catalyst in the preparation of nitrosamines by reacting nitric oxide with a secondary or tertiary amine in the presence of the catalyst at a temperature between 20°C and the decomposition temperature of the nitrosamine.

Contact Material

DEUTSCHE GOLD-UND SILBER-SCHNEIDANSTALT
German Patent 1,089,491

A contact material for low current contacts consists of an alloy of 25-35% Pd, 35-45% Ag, and 25-35% Au, preferably 30% Pd, 40% Ag and 30% Au.

Noble Metal Alloy as Material for Resistances

DEUTSCHE GOLD-UND SILBER-SCHNEIDANSTALT
German Patent 1,092,213

A palladium alloy containing uranium, rhenium and chromium, singly or together in proportions of 1-50%, preferably 3-40%, alloying metals and remainder palladium, is used as material for resistances, especially potentiometer wires. 50% of the rhenium, when present, may be replaced by vanadium. Up to 30%, preferably up to 15%, of the Pd may also be replaced by Au and/or Ag. The alloys have a low specific weight, small temperature coefficient, high fracture resistance and high specific resistance.

Isomerisation of Normal Paraffins

SINCLAIR REFINING CO. *U.S. Patent* 2,972,650

A method of isomerising C₈ to C₉ *n*-paraffins in a feed containing aromatics is effected in two

stages. The *n*-paraffins are first contacted with a first stage catalyst (0.01-2% platinum group metal + 3-20% boron on activated alumina) at 200°-750°F in the presence of free hydrogen and the resulting product is then contacted with a second stage catalyst (0.01-2% platinum group metal and 2-50% of an aluminium halide Friedel-Crafts component on activated alumina), at 150°-450°F in the presence of free hydrogen, and while providing 1-25% of a hydrogen halide.

Surface Treatment

ENGELHARD INDUSTRIES INC. *U.S. Patent* 2,973,283

Articles made of platinum-rhodium alloys are surface treated by coating first with platinum and then with gold, each coating being sufficiently thick to allow the formation of boundary zones of solid solutions. The article is then heated to diffusion temperature to harden the outer surface to a hardness at least equal to that of the Pt-Rh alloy by the formation of the solid solutions.

Catalyst for Hydrocyanic Acid Production

BERGWERKGESELLSCHAFT HIBERNIA A.G. *U.S. Patent* 2,975,144

A catalyst for use in the production of hydrocyanic acid by reaction in vapour phase of ammonia, a hydrocarbon and oxygen, is composed of platinum metals and is in the form of coils formed by spirally winding non-perforated foils of platinum metals (one at least of which is corrugated). The foils have a thickness of more than 0.02 mm, several coils being superimposed on one another to form the catalyst.

Hydroforming Catalyst

ESSO RESEARCH AND ENGINEERING CO. *U.S. Patent* 2,976,232

Hydrocarbon fractions boiling in the naphtha boiling range are hydroformed by contacting naphtha vapours mixed with hydrogen at 800-1000°F and pressure of 100-1000 p.s.i.g. with a catalyst composed of 0.01-5 wt.% of a platinum group metal combined with 0.05-5 wt.% of ceria supported on an alumina-containing carrier.

Compositions for Electrical Resistance Films

FAIRCHILD CAMERA AND INSTRUMENT CORP. *U.S. Patent* 2,978,314

A composition suitable for electrical resistance films is composed of an intimate physical mixture of finely divided particles consisting of 80% by wt. palladium and rhodium (weight proportion: 1.4 to 1.67 of palladium to 1 of rhodium) and balance gold.

Treatment of Gases

ENGELHARD INDUSTRIES INC. *U.S. Patent* 2,980,743

Carbon monoxide is removed from a mixture of itself with acetylene and oxygen by passing the mixture over a rhodium-containing catalyst at a temperature of 25°-150°C.