

Al, however, no breakdown occurred over a period of 707 hr. This establishes that a considerable part of the insulation ageing occurs because of the penetration of Ag from the anode into the ceramic. For reliable operation for long periods at increased or high temperatures the anode, at least, should be made of burnt-in Pt.

### Rhodium Plating Techniques

R. R. BENHAM and I. J. WARWICK, *Brit. Commun. & Electronics*, 1958, 5, (4), 266-269

The uses of Rh plating in the electronics industry are described. The high wear and corrosion resistance and constant resistivity of Rh deposits make them particularly suitable as a contact material. An outline of the plating procedure is given.

## TEMPERATURE MEASUREMENT

### The Accurate Measurement of Temperature

J. A. HALL, *Research*, 1958, 11, (4), 147-151

The International Temperature Scale and some of the most accurate temperature measuring instruments are described. The platinum resistance thermometer can be used in the temperature

range  $-182.97^{\circ}\text{C}$  to  $1062^{\circ}\text{C}$ ; up to  $630.5^{\circ}\text{C}$  it has a reproducibility better than  $0.01^{\circ}\text{C}$ . Mercury thermometers have the advantage of simplicity in use but are not very accurate unless special precautions are taken. Pt/Rh-Pt thermocouples can be used in the range  $400-1700^{\circ}\text{C}$ , with an accuracy from  $\pm 1^{\circ}\text{C}$  at  $1063^{\circ}\text{C}$  to  $\pm 5^{\circ}\text{C}$  at  $1600^{\circ}\text{C}$ . Other thermocouples are also described. Above the range of rare-metal thermocouples a disappearing filament pyrometer is used, but this is only accurate when the emissivity of the surface is known. With a black-body source accuracies of  $\pm 10^{\circ}\text{C}$  at  $1600^{\circ}\text{C}$  can be obtained.

### Immersion Thermocouple Sheaths

O. M. MARGULIS, K. G. ROMANCHENKO and I. A. GETMAN, *Stal'*, 1957, (8), 714-715

Research was carried out on the development of sheaths capable of repeated use in liquid steel temperature measurement. In general ceramic sheaths were found to crack at the liquid metal-air interface. This cracking can be prevented if the sheaths are covered with a protective coating of a composition based on magnesite or on  $\text{ZrO}_2$  stabilised with CaO. Sheaths prepared in this way can be used for several brief immersions or for a prolonged immersion of up to 4 hrs.

## NEW PATENTS

### Supported Platinum Catalysts

W. R. GRACE & CO. *British Patent* 790,431

A catalyst is prepared by commingling alumina shapes with an aqueous chlorplatinic acid-hydrogen halide solution 0.1-1.0 normal with respect to the hydrogen halide, drying at  $200-400^{\circ}\text{F}$  and then converting the adsorbed platinum to the metallic state. The hydrogen halide may be hydrofluoric acid or hydrochloric acid. Suitable for reforming of gasoline.

### Catalytic Reforming

SOCONY MOBIL OIL CO. INC. *British Patent* 790,476

A reforming catalyst having a surface area of from 650-100 g/sq metre comprises platinum and/or palladium in amount from 0.05-2% by weight deposited on silica which contains alumina in amount from 0.25-7% (based on weight of catalyst), the amount being correlated with the surface area to define a point falling within an area given on an accompanying graph.

### Manufacture of a Cyclic Ketone

F. HOFFMANN-LA ROCHE & CO. A.G. *British Patent* 790,607

A platinum hydrogenation catalyst is used in a process involving the oxidation of 2,6,6-trimethyl-cyclo-hexen-(1)-one-(4) with an organic peracid,

hydrolysing the product, followed by isomerisation to form 2,6,6-trimethyl-hexan-dione-(1,4), which is then reduced with hydrogen in the presence of the catalyst to give 2,6,6-trimethyl-cyclo-hexanone-(1)-ol-(4).

### New Carotenoid Compounds

F. HOFFMANN-LA ROCHE & CO. A.G. *British Patent* 790,614

A palladium/calcium carbonate catalyst is used as a hydrogenation catalyst in a process for the manufacture of 4,4<sup>1</sup>-disubstituted-15,15<sup>1</sup>-dehydro-beta-carotenes.

### New Carotenoid Compounds

F. HOFFMANN-LA ROCHE & CO. A.G. *British Patent* 790,615

A palladium/calcium carbonate hydrogenation catalyst is used in a process for the manufacture of 4,4<sup>1</sup>-diketo-15,15<sup>1</sup> dehydro-beta-carotene and 15,15<sup>1</sup> monocis, 4,4<sup>1</sup>-diketo beta-carotene.

### Di (2,2,2-Trifluorethyl) Acetal of Acetaldehyde

AIR REDUCTION CO. INC. *British Patent* 790,824

Di (2,2,2-trifluorethyl) acetaldehyde acetal is heated in the presence of a dehydration catalyst to produce 2,2,2-trifluorethyl vinyl ether. The

catalyst may be platinised asbestos containing 10% by weight of platinum.

### Purification of Aliphatic Acids

THE DISTILLERS CO. LTD. *British Patent* 791,044  
Impure aliphatic monocarboxylic acids of 2-4 carbon atoms produced by the liquid phase oxidation of paraffinic hydrocarbons of 4-8 carbon atoms and containing certain odorous substances and materials of a reducing nature are purified by subjecting the acids to hydrogenation in the presence of a platinum group metal catalyst. The catalyst may comprise 0.1 part by weight of Adam's platinum oxide, or granular carbon impregnated with 1% by weight of palladium or silica impregnated with 1% palladium may be used.

### Desulphurisation of Petroleum Fractions

SOCONY MOBIL OIL CO. INC. *British Patent* 791,071  
A sulphur-containing petroleum fraction is desulphurised by contacting it with a catalyst comprising 0.05% to 20% by weight of a metal of the platinum or palladium series, i.e. having an atomic number of 44-46 or 76-78 inclusive, supported on an inert carrier, e.g. alumina, silica, magnesia, charcoal or coke at 500-850°F in the presence of hydrogen under a pressure of 100-3,000 lb/sq in (gauge) at a liquid hourly space velocity of 0.1-10 V/V/hr and with a hydrogen to hydrocarbon molar ratio of 1-80. Platinum or palladium is preferred.

### Combined Hydrocracking and Hydroforming Process

SOCONY MOBIL OIL CO. INC. *British Patent* 791,072  
In a process of catalytic conversion of petroleum hydrocarbons, a stock of heavy petroleum fractions is hydrocracked in the presence of a catalyst composed of 0.05-20% by weight of a metal of the platinum or palladium series (atomic number 44-46 or 76-78 inclusive), deposited on a synthetic composite of solid oxides of at least two elements of Groups IIA, IIIB, IVA and IVB of the Periodic Classification, such as silica-alumina at 400-825°F, the heavy naphtha produced is then reformed in the presence of a platinum or palladium reforming catalyst to produce a gasoline.

### Catalytic Hydrocracking

SOCONY MOBIL OIL CO. INC. *British Patent* 791,073  
Discloses the hydrocracking step of No. 791,072 above, using a catalyst of platinum- or palladium-on silica-alumina with or without fluorine.

### Production of Compounds of Amides of Lysergic Acid

CILAG LTD. *British Patent* 791,173

Dehydro compounds of ergotamine, ergotoxine, ergocristine, ergotinine, ergosine and sensibamine or addition products or salts thereof are produced

by hydrogenation thereof with hydrogen in the presence of a catalyst composed of finely divided platinum or rhodium deposited on a carrier, e.g. carbon, at atmospheric pressure or a pressure not exceeding 10 atmospheres, hydrogenation being effected initially at room temperature and completed at a temperature not over 100°C.

### Novel Esters

F. HOFFMANN-LA ROCHE & CO. A.G. *British Patent* 791,177

A lead-inhibited palladium catalyst, e.g. lead-inhibited palladium-calcium carbonate catalyst, is used as a hydrogenation catalyst in one stage of a process of producing new esters of specified general formula useful as odour-imparting agents in perfumes and scents.

### Hydantoin Derivatives

MERCK & CO. INC. *British Patent* 791,327

A palladium-on-charcoal hydrogenation catalyst is used in the preparation of a 5-(delta-halo-butylidene) hydantoin in which the halogen is other than fluorine, e.g. bromine or chlorine.

### Hydantoin Derivatives

MERCK & CO. INC. *British Patent* 791,328

5-(delta-aminobutylidene) hydantoin is contacted with hydrogen in the presence of a catalyst of noble metal or noble metal oxide, e.g. platinum oxide, to produce 5-(delta-aminobutyl) hydantoin.

### Preparation of 3,3,3-Trimethylisopropanol

RHEINPREUSSEN A.G. FÜR BERGBAU & CHEMIE  
*British Patent* 791,542

3,3,3-trimethylisopropanol is prepared by hydrogenating 3,3,3-trimethylolacetone at normal or elevated pressure and normal or elevated temperature in the presence of a hydrogenating catalyst, e.g. platinum oxide or platinum black.

### Alcohol Hydrofinishing

ESSO RESEARCH & ENGINEERING CO. *British Patent* 791,747

Ethyl alcohol obtained from the hydration of ethylene and having a permanganate time of at least 10 minutes is purified by treatment in liquid phase with hydrogen in the presence of a platinum catalyst, preferably for less than 24 hours and at a temperature below the boiling point of the alcohol.

### Cathode Ray Tubes

EGYESULT IZZOLAMPA ES VILLA-MOSSAGI RESZVENY-TARSASAG *British Patent* 792,046

A cathode ray tube has a supply cathode comprising a closed supply chamber containing an activator, part of the chamber wall consisting of a porous sintered body, the pores of which form the only passages for the activator to the outer surface of the cathode. At least part of the surface of the body is made of a metal of the platinum

group or an alloy thereof and adapted to form an alloy with an alkali metal or alkaline earth metal. The activator is an alloy containing an alkali or an alkaline earth metal.

### Combustible Gas-leak Detectors

NORTH THAMES GAS BOARD *British Patent* 792,101  
A platinum filament or a filament of a less noble metal coated with spongy platinum is used as a catalyst in a gas-leak detector. The catalyst filament, which is included in a Wheatstone Bridge network, becomes, in the presence of gas and air, heated and varies in resistance.

### Hydrogenation of Alkali Metal Salts of Dicyclopentadiene Dicarboxylic Acids

ESSO RESEARCH & ENGINEERING CO. *British Patent* 792,218

A hydrogenation catalyst of Adam's platinum oxide or palladium may be used in a process for hydrogenating a water-soluble alkali metal salt of a dicyclopentadiene dicarboxylic acid.

### Preparing Aviation Fuels

N.V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ *British Patent* 792,643

A mixture of liquid hydrocarbons is prepared by separating from a naphthenic straight run gasoline a fraction having an initial boiling point of about 65°C and a final boiling point of 130–140°C, catalytically reforming the fraction in the presence of a platinum catalyst and mixing the resulting reformat with all or part of the fraction boiling up to about 65°C.

### Preparation of Catalyst Particles

ESSO RESEARCH & ENGINEERING CO. *British Patent* 792,645

Platinum group metal-alumina catalysts are made by submerging preformed shaped bodies of adsorptive alumina in a large amount of water, circulating water through a bed of such bodies, slowly adding a solution of a platinum group metal compound to the water in sufficient amount to incorporate the required amount of platinum group metal into the support and continuing the circulation so as more completely to impregnate the support, after which the bodies are separated, dried and calcined. Chloroplatinic acid may be used.

### Hydroforming Processes

ESSO RESEARCH & ENGINEERING CO. *British Patent* 792,806

A petroleum hydrocarbon is contacted in a hydroforming zone under hydroforming conditions with a hydrogen-containing gas and a hydroforming catalyst supported on an alumina base containing a major amount of eta alumina and obtained by hydrolysing an aluminium alcoholate with 1–10 volumes, based on the alcoholate of ammonium hydroxide solution containing 1.8–3.4% by weight of ammonia. Hydrolysis is carried out at 35–100°F, the re-

sultant hydrous alumina slurry being then aged for ½ hr to 15 hrs, the alumina separated from the slurry and calcined at 400–1250°F. The catalyst metal used is platinum.

### Capacitors

G. V. PLANER LTD. *British Patent* 792,835

A capacitative element consists of a composite body with at least two closely adhering electrodes. The body is composed of a ceramic, e.g. a fired alkaline earth titanate, in which are dispersed particles of a metal with a melting point above the forming temperature of the ceramic; the body as a whole constitutes an electrical insulator and has an apparent permittivity of 750 or over. The metal is preferably a platinum group metal or an alloy containing gold, e.g. a gold-platinum alloy. Platinum or palladium powders of relatively fine particle size are referred to.

### Hydrogenation of Alkylanthraquinones

E. I. DU PONT DE NEMOURS & CO. *British Patent* 793,004

In the production of hydrogen peroxide with the use of an alkylanthrahydroquinone produced by hydrogenation of alkylanthraquinones, as an intermediate by the process of Patent No. 686,574 using a metallic palladium-on-activated alumina catalyst in the hydrogenation of the alkylated anthraquinone, optimum catalyst performance is obtained by maintaining the water content of the mixture in the hydrogenator within the range corresponding with relative humidities in the gas phase of the mixture of from 40–98%.

### Production of Amines

IMPERIAL CHEMICAL INDUSTRIES LTD. *British Patent* 793,716

A 5% platinum-on-charcoal catalyst is used in the production of 4-sec-butylamino-diphenylamine by reacting 4-amino-, 4-nitro or 4-nitrosodiphenylamine with hydrogen and methyl ethyl ketone in the presence of the catalyst.

### Desulphurisation of Hydrocarbons

ESSO RESEARCH & ENGINEERING CO. *British Patent* 793,817

A sulphur-containing hydrocarbon is desulphurised by contacting it in the vapour phase with 100–10,000 standard cubic feet of hydrogen per barrel of liquid hydrocarbon at 340–940°F and a pressure of 15–600 p.s.i.g. in the presence of a platinum-type hydrogenation catalyst. The catalyst preferably contains 0.05–2 wt% of platinum, supported on alumina, silica-alumina, silica-magnesia or activated charcoal.

### Hydroforming

ESSO RESEARCH & ENGINEERING CO. *British Patent* 793,924

Gasoline is produced by hydroforming a naphtha feed, separating C<sub>9</sub> and heavier hydrocarbons from the product, thermally hydrodialkylating the C<sub>9</sub> and heavier components to form C<sub>7</sub> and

C<sub>8</sub> hydrocarbons, hydrofining at least part of the latter and recycling the hydrofined product to the hydroformer. A platinum-on-alumina catalyst is used.

### Electric Capacitor

AEROVOX CORP. *British Patent* 794,056

A ceramic capacitor comprises a ceramic core having an inner electrode of non-oxidising refractory metal bonded to and covering the length thereof, but leaving at least one end surface exposed. This electrode is covered with a dielectric film which leaves exposed the end surface of the electrode remote from the exposed end of the core, this film being covered with an outer electrode. The refractory metal is preferably platinum or palladium and is applied in the form of the resinate.

### Treating Noble Metal Catalysts

HOUDRY PROCESS CORP. *British Patent* 794,349

Platinum group metal on alumina catalysts are activated in the presence of iron by treatment, in the absence of normally liquid hydrocarbons and carbon, with a gas mixture consisting mainly of hydrogen and comprising hydrogen halide in an amount not greater than 15% of the hydrogen halide partial pressure necessary for the formation of iron halide at the treating conditions to increase the halide content of the catalyst to at least 0.05, but not more than 2% halogen.

### Metallisation of Ceramics

GENERAL ELECTRIC CO. NEW YORK *U.S. Patent* 2,820,727

A refractory body having a high alumina content is metallised by applying to the body one or more layers of a mixture of alumina and Fe<sub>2</sub>O<sub>3</sub> or Cr<sub>2</sub>O<sub>3</sub> (Al<sub>2</sub>O<sub>3</sub> = 30–90% by weight of the mixture) and then a layer of platinum or iridium. The coated body is then heated in an oxygen atmosphere to 1200–1600°C for platinum or 1200–1750°C for iridium to cause the oxide-alumina coating to react with the body and form a bond between it and the metal coating.

### Production of Cyclohexylamine

ABBOTT LABORATORIES *U.S. Patent* 2,822,392

Cyclohexylamine is prepared by passing continuously countercurrently aniline and hydrogen, in the absence of a solvent, once through a reaction zone containing a ruthenium catalyst maintained at a temperature of 200–250°C and a pressure of 250–10,000 lb/sq in. The rate of flow of the aniline through the catalyst is 0.5–3.5 volumes of feed per hour per volume of catalyst.

### Production of Hydroxylamine Salts

BADISCHE ANILIN & SODAFABRIK A.G. *U.S. Patent* 2,823,101

Hydroxylamine salts are formed by reduction of nitric oxide with hydrogen in an acid medium in

the presence of a catalyst prepared by adding to a platinum metal catalyst at least one compound of arsenic, antimony, bismuth, sulphur, selenium and/or tellurium; the compound is reduced under the reaction conditions to the respective element.

### Hydrogenation of Nitro Compounds

E. I. DU PONT DE NEMOURS & CO. *U.S. Patent* 2,823,235

A hydrogenation catalyst for use in the reduction of organic nitro compounds comprises an oleophilic carbon black support having an oil absorption factor of at least 200 and a surface area of 20–100 m<sup>2</sup>/g, on which is deposited 0.1–10% by weight of the support of palladium or platinum.

### Manufacture of Alumina-platinum Catalysts

UNIVERSAL OIL PRODUCTS CO. *U.S. Patent* 2,826,555

A platinised alumina catalyst is prepared by commingling with an aqueous chloroplatinic acid solution containing an appreciable amount of chloroplatinous acid, an amount of oxidising agent, not much in excess of that required to oxidise the chloroplatinous acid, and oxidising the latter with it. The oxidising agent comprises a mixture of hydrochloric acid and hydrogen peroxide. The treated solution is then composited with alumina, in an amount to form a final catalyst containing 0.01–10% by weight of platinum and the composite is calcined at 500–1200°F.

### Manufacture of Trichloro- and Tetrachlorobenzenes

ETHYL CORPORATION *U.S. Patent* 2,826,617

Chlorinated benzenes are recovered from a waste tetrachlorobenzene chlorination by-product mixture containing a large proportion of pentachlorobenzene by reducing the materials to lower chlorinated benzenes by reacting the mixture with hydrogen at above 250°C while in contact with a hydrogenation catalyst to reduce the pentachlorobenzene by an amount greater than the net reduction of the tetrachlorobenzene. The catalyst used may be platinum or palladium.

### Preparation of Hydroxylamine

SPENCER CHEMICAL CO. *U.S. Patent* 2,827,363

Hydroxylamine is prepared by bringing together an aqueous solution of nitric acid, molecular hydrogen and an active rhodium catalyst at a temperature ranging from above the freezing point of the mixture up to 170°C. The hydrogen is provided at the catalyst-acid interface in time to prevent dissolution of the catalyst and is maintained thereat during the reaction, the hydroxylamine so produced being recovered.