FILM STUDY GROUP

-SUBJECT-INDEX AND REPORT

T.O.M. REEL NO. 38

Prepared by

CALIFORNIA RESEARCH CORPORATION

CALIFORNIA RESEARCH CORPORATION RICHMOND, CALIFORNIA-

INDEX OF TECHNICAL OIL MISSION

MICROFILM

REEL NO. 38

Prepared by California Research Corporation

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C. W. J. J. Merminan

CALIFORNIA RESEARCH CORPORATION RICHMOND, CALIFORNIA

ABSTRACT OF TECHNICAL OIL MISSION

MICROFILM

REEL NO. 38

Prepared by California Research Corporation

CALIFORNIA RESEARCH CORPORATION RICHMOND, CALIFORNIA

ABSTRACT OF TAC REEL NUMBER 38

Frame 1

An index to documents for bag No. 3450 C.I.O.S. Target No. 30/501. Ruhrchemie A.G. at Sterkrade and Holten. This frame lists general titles for the documents in reel No. 38. The order is not the same as the items appear in the reel.

Frames 2, 3, 4, 5, and 6

Show charts of operating conditions for a reactor converting CO hydrogen mixtures with an iron catalyst. Of no particular interest.

Frames 7 to 9

A patent application from Ruhrchemie dated April 4, 1944 regarding an apparatus for the automatic supply of accurately measured amounts of liquid, particularly at highly reduced pressures. By means of automatic manameters and electrical regulation of magnetic valves. A constant differential is maintained across a capillary feeding the liquid. A sketch of the apparatus is shown in Frame 10.

Frames 11 to 15

A patent application on an apparatus for automatic sampling at reduced pressures. By means of magnetic valves samples are withdrawn under reduced pressure from a low pressure system. A sketch of the apparatus is included.

Frames 16 and 17

A patent application dated April 20, 1944 on a process for obtaining high molecular weight compounds from low molecular weight olefins by catalytic hydration and subsequent fusion with alkali. The process is characterized in that the amount of alkali used in the fusion is calculated to satisfy the carbonyl number of the aldehyde which is the dehydrogenation product of the intermediate alcohol.

Frames 18 and 19

Patent application from Ruhrchemie dated May 15, 1944 on a process for making resins. In the process cyclopentadienes are subjected to the action of carbon menoxide and hydrogen, more particularly water gas,

at elevated temperatures and pressures of the order of 125°C. and 150 atmospheres with, for example, a Fisher Tropsch catalyst.

Frames 20 and 21

Illegible notes.

Frame 22

A chart on operation of equipment converting CO and hydrogen. Probably goes with the first few frames of this reel.

Frames 23 to 51

Iaboratory notes applying to the preparation and use of methanization catalysts. Most of record illegible but believed to apply to hydrogenation of gasolines and of toluene. Contains several charts and tables of data.

Frames 52 and 53

Instruction sheet from Ruhrohemie dated June 15, 1942 on the

Frames 63 to 126

Longhand report including many charts and tables, most of the frames are illegible. Discusses hydrogenation of polymer naphtha with nickel catalyst. Some of the experiments were carried out with cracked gasoline. Part of the notes refer to preparation of the nickel catalyst described above.

Frame 127

A memo from Oberhausen-Holten dated Cotober 5, 1943 to the Reichsministry for Munitions on the subject of the excessing the expansion of facilities for production of excellences.

Frames 128 to 133

A report dated June 12, 1943 on the recovery and use of sulfur from coal. Gives status of the production of coal, the sulfur content of coal and methods of recovering sulfur from the coal, particularly through treatment of gas obtained in treatment. Gives figures on possible production of sulfuric acid from the sulfur content of the coal. Mentions also the conversion to useful sulfates (by Dr. Martin).

Frames 134 to 143

Reports on the capacity of power plants of the Ruhrchemie, Discusses existing plants, plants under construction, the capacity of old steam plants, enlargement of older plants, and the status of the building program.

Frame 143

A memo dated May 9, 1943 regarding power plant expansion.

Frame 144

A memo from Dortmund dated March 2, 1943. Of no interest.

Frame 145 and 146

A report on an experiment with diluted cobalt catalyst showing results of a life test. The reactor was on stream for 195 days.

Frames 147 to 150

A report to Dr. Altpeter on iron catalyst synthesis comparing products and operation with similar charges of cobalt catalyst.

Frames 151 to 152

Longhand notes of no interest.

Frame 153

Letter dated June 19, 1943 to the Director of German Mitsui Company regarding scaps and detergents.

Frames 154 and 155

Letter to Dr. Altpeter dated June 7, 1943 on the production of fatty acids. The letter relates to the recovery of fatty acids from fractions of the products of fuel synthesis.

Frames 156 and 157

Similar letter dated February 6, 1943 on synthetic fatty acids from the Ruhrchemie Fischer paraffin showing the possible production of fatty acids by oxidation of the wax with and without prior cracking.

Frame 158

Longhand notes on the above subject showing the same statistics.

Frames 159 to 162

Report dated June 3, 1943 giving a review of the production of the coke oven and hydrogenation plants of DAVV.

Frames 163 and 164

Letter dated February 12, 1943 from Franz Fischer of the Kaiser Wilhelm Institute to Professor Martin discussing the interesting possibilities of the use of carbon dioxide hydrogen mixtures for gasoline synthesis.

Although the use of CO2 gives lower yields of hydrocarbons Professor Fischer

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Frame 173

Blank letter form from Rhurchemie.

Frame 174 to 176

Report from Dr. Apfelbeck to Dr. Martin relating to exploratory work on the high voltage electrolytic hydrogenation of coal with lead electrodes in contact with the coal

Frames 177 and 178

Another letter dated April 7, 1943 from Dr. Apfelbeck to Dr. Martin on the same subject.

Frame 179

Memo concerning correspondence with Dr. Apfelbeck.

Frames 180 and 181

Another letter dated February 10, 1943 from Apfelbeck to Martin on the same subject.

Frame 182

Brief note dated February 9, 1943.

Frames 183 to 187

Patent application on the process for hydrogenating coal. The coal to be hydrogenated is brought into-contact with the hydrogen which is in a nascent state, the coal is used directly as the cathode in the electrolysis production of the hydrogen or is immediately over the cathode of the electrolytic cell.

Frames 188 to 191

Detailed construction drawing of a reactor vessel weighing about 5500 Kilograms and having a diemeter of about 3.1 meters. The vessel was designed for low pressure.

Frame 192

Drawing showing insulation of reactor.

Frame 193

A hand sketch

Frames 194 to 210

Detailed construction drawings of foundations and reactor supports.

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Frames 211 to 214
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Frames 302 to 309

Analysis of sulfuric acid.

Frames 310 to 324

Determination of the heating value of coal.

Frames 325 to 329

Determination of small amounts of oxygen in gases. First method is a colorimetric method with emmoniacal cuprous chloride. The second method described is a titrimetric method with manganous chloride. The manganous chloride solution, after contact with oxygen-bearing gas, liberates iodine from potassium iodide solution and the liberated iodine is titrated with thiosulfate solution.

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Determination of hydrogen sulfide in coke oven gas. Methods using cadmium acetate, zinc acetate and iodine solution.

Frames 337 to 350

Analysis of spent gas-purification contact agents. Gives methods for determination of water, soluble sulfur, determination of total sulfur by combustion, and determination of cyanide.

Frames 351 to 354

Examination of fresh gas-purification contact agent with respect to its ability to absorb hydrogen sulfide.

Frame 355

Tital sheet - Analytical instructions. Apparently misplaced.

Frames 356 to 359

Continuation of discussion of gas-purification agent tests.

Frames 360 to 362

Analysis of bog iron ore. Gives methods of determination of water ignition loss, insoluble residue and iron.

Frames 363 and 364

Analysis of Luxmasse. The Luxmasse analysis is carried out in a similar manner to that for the bog iron ore. In addition, its alkali content must be determined because of the method of manufacture. Luxmasse is the insoluble residue, mostly iron oxide, which remains after solution of the alumina constituents of bauxite with caustic.

Frames 365 to 384

Determination of the yield from catalytic oxidation of ammonia on platinum catalyst. The ammonia is burned with air over the catalyst, and the NO yield determined.

Frames 385 to 392

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Frames 393 to 407

Examination of the vapors from the crude nitrate saturators and determination of nitrogen loss.

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Frames 435 to 439

Gas analysis. Discusses methods of taking gas samples.

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Analysis of Girbotol solution (ethanolamine-potassium phosphate solutions).

Frames 462 to 473

Analysis of pure Girbotol solution (ethanolamine).

Frames 474 to 479

Examination of the alkaline wash liquor from carbon dioxide scrubbing of coke oven gas and other alkaline liquors.

Frames 480 to 486

Determination of carbon monoxide in hydrogen-nitrogen mixtures. The method uses the iodine pentoxide reaction.

Frames 487 to 496

Determination of small quantities of acetylene in gases.

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Frames 497 and 498

Determination of the chlorine content of the residual gases from the Holten Chemical Plant. Gases are those which come from the ethylene oxide manufacture.

Frames 499 to 503

Determination of small amounts of ammonia in gas. Titrimetric and colorimetric methods are given.

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Determination of total and of organic sulfur in coke oven gas. A combustion method.

Frames 510 to 520

Determination of small quantities of carbon monoxide in the synthesis gas for synthetic ammonia. The iodine pentoxide method.

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Frames 562 to 571

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Frames 572 to 574

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Frames 584 to 588

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Frames 598 and 599

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Frames 600 to 602

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Frames 603 and 604

Determination of solid foreign matter and asphalt in oil.

Frames 605 to 612

Analysis of kieselguhr. Analysis for water, ignition loss, acid-soluble iron content, aluminum content, calcium, water-soluble sulfate, chloride and pH.

Frames 613 to 616

Determination of very small quantities of water in hydrocarbons, especially in motor benzol and in gasoline. Method according to Broche and Scheer, Brennstoffchemie, Volume 13 (1932) page 281. Method depends on the liberation of hydrogen from calcium hydride and measurement of the gas volume.

Frames 617 to 630

Analysis of sodium nitrate. Similar to the analysis of ammonium nitrate given earlier.

Frames 631 to 636

Determination of small quantities of chlorides. A titrimetric procedure with mercuric ion using diphenyl carbazone as the indicator.

Frames 637 to 640

Determination of water in mineral oils. Depends on the titration of HCl formed by the reaction of acetylchloride with the water in the oil in the presence of pyridine or aniline.

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Determination of water in alcohols. Depends on the reaction of acetylchloride and water in the presence of aniline.

Frames 646 and 647

Determination of peroxides in mineral oils by the potassium iodide method.

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Determination of the aging tendency of oils. Determination of ter number, exidation number, and coke number.

Frames 652 to 656

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Frames 668 to 672

Determination of isobutylene in gases. The determination is made by selective absorption in sulfuric acid. Reference given is Industrial and Engineering Chemistry, Analytical Edition, Volume 30 (1938) page 360.

Frames 673 to 693

Heating value of gases with the Junkers calorimeter.

Frames 694 to 709

Aging of lubricating oils. Behavior with oxygen. Gives the Indiana oxidation test and the Ruhrchemie method.

Frames 710 to 718

Determination of hydrocyanic acid in coke oven gas. Two methods are given, one in which the HCN is converted to ammonium thiocyanate with ammonium polysulfide solution and another in which the HCN is absorbed in alkaline ferrous sulfate solution and is converted to sodium ferrocyanide. The ferrocyanide is decomposed and the HCN is determined with silver ion.

Frames 719 to 756

Tables for calculation of density and heating value of gases.

Frames 757 to 776

Test of the activity of catalysts for the Casale ammonia synthesis. Detailed description of method and sketch of apparatus used.

Frames 777 to 785

Determination of isobutylene in gases by addition of hydrogen chloride. The method described in Industrial Engineering Chemistry, Analytical Edition, Volume 9 (1937) page 511 and in the Canadian Journal of Research, Volume 2 (1930) page 267.

Frames 786 to 810

Determination of hydrocarbon with one to five carbon atoms and of the isomers of butane and butylene by fractional precision distillation, typical low temperature gas fractionation.

Frames 811 to 818

Determination of the hydroxyl number of alcohols. The free hydroxyl groups are determined by reaction with acetic anhydride and determination of the liberated acetic acid.

Frame 819

Title page - Collected Experimental Work on Lubricating Oil Synthesis.

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Frames 820 to 826

Dated January 9, 1945. Report on the conversion of olefins in cracked gasoline in a step-wise synthesis into residual oil containing from 5 to 50% bright stock. Oils having a viscosity at 50°C. from 20° to 22° Engler with 50 - 55% bright stock having viscosity of 50° Engler at 50°C. were produced from cracked gasoline by taking 100 parts of catalyst oil with 4 parts of aluminum chloride per 100 parts of clefin heating at 20 - 25°C. for 12 hours followed by 2 hours at 50°C. In order to activate the catalyst oil the temperature is periodically raised to 50° to 70°C.

Frames 827 to 834

Dated December 19, 1944. Division of the synthesis of oil from cracked gasoline into seven consecutive steps. Similar to polymerization of the olefins in cracked gasoline given above but in which aluminum chloride was added to the gasoline in seven separate additions of small amounts. The experiment showed that the values for density, viscosity and pole height, Conradson carbon, icdine number and tar content decreased steadily in the seven steps. The pour point improved correspondingly.

Frames 835 to 839

Dated December 16, 1944. Effect of iron content of the aluminum chloride on the polymierization of cracked gasoline. Aluminum chloride can be used with a content of iron up to about 20% iron chloride without harm. Pure iron chloride does not act as a normal polymerization catalyst.

Frames 840 to 847

Dated November 25, 1944. Division of the gasoline polymerization into a primary step and a main synthesis step. Iron chloride or preferably aluminum chloride was added to cracked gasoline in a small amount prior to the main polymerization step. The pretreatment gave a very unsaturated, highly viscous tarry material with a pole height of 2 to 4. The value of the separation of this material from the synthetic oil can be determined only by motor test. The results were believed superior to that obtained by pretreatment of the cracked gasoline with clay.

Frames 848 to 856

Dated October 24, 1944. Division of the C7 cracked gasoline fraction into several cuts by distillation and polymerization of the resulting narrow fractions, especially the 1-heptene. Cracked gasoline was separated by distillation into 1°C. boiling range fractions. Polymerization of these fractions gave oils whose pole heights varied from 2.21 to

Frame 857

Dated June 19, 1944. Dechlorination experiments with zinc turnings and clay and with aluminum turnings and clay were made. The results indicated that the zinc was the better dechlorination agent.

Frame 858

Dated March 21, 1944. A brief memorandum regarding the patent application on an apparatus for testing oxygen stability of lubricating oil.

Frames 859 to 870

Dotal March The Tolele Donardanan in the autificated anim of

synthetic oil in the Rubrchemie test equipment. Numerous experiments showed that synthetic oils which had not been inhibited gave, in the oxidation test, results which showed that the oxygen consumption went through a maximum in the beginning and that stability increases on the average with increasing viscosity. Onls obtained from the higher olefins were more stable than those obtained from lower molecular weight olefins. The low-boiling, low-viscosity fractions of the oil were much less stable than bright stock. Synthetic oils are oxidized and changed noticeably at 100°. With increasing temperatures the oxidation process increases steadily and at 160° a good measurement was not possible because of the rapidity of the reaction.

Frames 871 to 873

Dated March 3, 1944. Apparatus for the oxidation resistance of lubricating oil. Description of the oxidation equipment, with drawings.

Frames 874 to 876. -

Duplicates of frames 871 to 873.

Frames 877 and 878

Dated March 1, 1944 and February 29, 1944. Letters of transmittal of report on summary of work done on oil synthesis, etc.

Frames 879 to 887

185° fraction containing 61 - 64% olefins yielded 72% oil from olefins and the pole height was 1.83 to 1.93. An intermediate fraction had a pole height of 2.5.

Frames 888 to 911

Dated January 20, 1944. Comparison between cracked gasoline and primary recycle gasoline with respect to analysis and synthetic oil manufacture. The comparison of cracked gasoline and primary recycle gasoline for production of oil showed that the recycle gasoline was not suitable for oil synthesis.

Frame 912

Dated January 11, 1944. On possible aviation oils. Proposal for blending synthetic oils and aviation oils.

Frames 913 to 924

Dated December 20,1943. For oil synthesis the optimum conditions are: one hundred parts of cracked gasoline boiling from 50 to 220°C., four parts of aluminum chloride, and one hundred parts of aluminum chloride tar are treated for two hours at 15° and two hours at 50°. The product has an average viscosity at 50°C. of 38° Engler.

Frames 925 to 941

Dated December 16, 1943. Model experiment on the production of cil with addition of recycle gasoline to the cracked gasoline (second part). Polymerization of the mixed cracked gasoline charge shows that the recycle gasoline from cobalt synthesis gives poorer results with an old oven charge than with fresh catalyst. The catalyst oil must be heated to at least 60° in order to reactivate it for further use. The value of the cil must be determined through large scale tests.

Frames 942 to 952

Dated October 12, 1943. Regarding stabilization of synthetic cils by addition of inhibitors before synthesis. Additional active inhibitors found included trithioformaldehyde, alpha nitroso beta naphthol, alpha naphthoquinone and thionalid (?). The addition of sulfur and diphenylamine to form phenthiazene is not necessary because this outstanding inhibitor can be prepared easily by other methods.

Frame 953

Dated October 27, 1943. Letter of transmittal for above report.

Frames 954 to 967

Dated September 16, 1943. On preparation of high viscosity residual oils from pretreated cracked gasoline in the single step synthesis. Single

step oil synthesis can be carried out with undistilled cracked gasoline boiling from 51 to 220° to give an oil with a viscosity at 50°C. of 37 to 38° Engler. This oil contains about 90% bright stock having a viscosity of 50° Engler. About 9% of aluminum chloride based on olefin charge must be used at low temperature. Short chain olefins give higher viscosity oils with lower pole height.

Frames 968 to 970

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viscosity residual oil.

Frame 971

June 29, 1943. Memo on production of aviation bright stock in the place of normal aviation motor oil.

Frames 972 to 973

June 23, 1943. Production of high viscosity oils. Repetition of discussion given earlier.

Frame 974

Dated May 4, 1943. Letter of transmittal for report that follows.

Frames 975 to 984

Report on oil synthesis. Addition of phenthiazene in the form of its components to the cracked gasoline used in oil synthesis. Concludes that addition of phenthiazene in the form of its components gives less stabilization than does addition to final oil.

Frames 985 and 986

March 18, 1943. Conditions for production of low viscosity

of special alloy. The analytical information obtained included oxygen consumption, viscosity increase, neutralization number, saponification number, Conradson carbon and flash point.

Frames 1010 to 1017

single-step oil synthesis including olefin balance and investigation of the catalyst oil. Under the optimum polymerization conditions the oil yield from olefins in cracked gasoline amounted to 74.3%. However, 14.2% of the olefins were lost into the catalyst oil.

Frames 1018 to 1027

Dated January 27, 1943. Report on model experiments on the tentative production of oil from a mixture of cracked gasoline and recycle naphtha. Model experiments were outlined for production of synthetic oil from three charging stocks, cracked gasoline from a Diesel oil fraction, cracked gasoline from wax, and recycle parhths from the cobalt catalyst.

step oil synthesis. Concludes that too much aluminum chloride should not be used because the oil yield is decreased.

Frames 1058 to 1061

October 31, 1942. Formation of lubricating oil from the residual clefins from the lubricating oil synthesis. Gives quantities of clefins remaining in the gasoline after oil synthesis.

Frames 1062 to 1079

October 19, 1942. Preliminary experiments on technical production of aviation oil stabilized with inhibitors. By preparing phenthiazene from sulfur and diphenylamine an inhibitor of satisfactory properties was obtained.

Frames 1080 to 1081

October 7, 1942. Experiments on the elimination of the emulsion forming tendency of Tycol. A procedure for removing the undesirable emulsion tendencies could not be found.

Frames 1082 to 1087

September 30, 1942. Some experiments on the conversion of Dubbs gas oil to lubricating oil. 39% yield of a synthetic oil with a pole height of 2.7 to 2.9 was obtained.

Frames 1088 to 1098

August 26, 1942. The stabilization of oils by addition of sulfur or sulfur chloride to cracked gasoline before polymerization. By the addition of sulfur or sulfur chloride to cracked gasoline before the oil synthesis polymerization an oxygen-stable oil was obtained.

Frames 1099 to 1113

August 20, 1942. Division of a normal upper layer from the oil synthesis into fractions by distillation and analytical investigation of the fractions.

Frame 1114

June 25, 1942. Note concerning two oxidation tests.

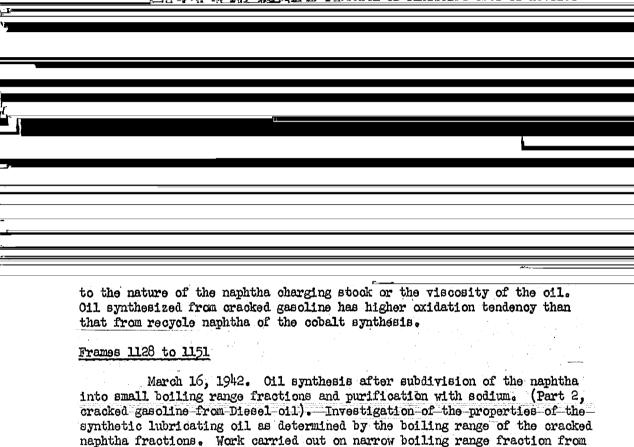
Frames 1115 to 1122

A report entitled with the question "Does Naphtha Undergo a Change Through Cracking in Polymerization to Lubricating Oil"? Data are presented to show that the olefin content of the cracked naphtha does not undergo any cracking to lower boiling hydrocarbons in the oil synthesis.

Frame 1123

April 17, 1942. A-memo-on-four-oxidation-tests.

Frames 1124 to 1127"



Frame 1152

March 4, 1942. Memo on three oxidation tests.

Frames 1153 and 1154

March 4, 1942. Suitability of cetene mixtures for polymerization to lubricating oil.

Frames 1155 and 1156

January 29, 1942. Suitability of Swedish shale oil distillates for preparation of synthetic oils. The oil yield from Swedish shale oil

Frames 1167 to 1178

October 10, 1941. Experiments on the preparation of lubricating oil II within the scope of the agreement with Japan. Gives results on experiments to determine if yields and quality of oil will fall within the guarantees of the Japanese agreement.

Frames 1179 to 1181

September 24, 1941. Concerns the dependence of inhibitor action on the boiling range of the cracked naphtha used for oil synthesis. The higher the boiling range of the original naphtha, that is, the longer are the carbon chains the more effective a given amount of inhibitor in stabilizing the oil.

Frames 1182 to 1184

August 1, 1941. Preparation of oil from recycle benzene from Hoesch (not complete). The naphthas from Hoesch were not as satisfactory.

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