PATENT SPECIFICATION

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COMPLETE SPECIFICATION.

An Improved Production of Gaseous and Liquid Fuels from Carbonaceous Materials.

I, John Conrad Arnold, a British subject, of 29, Southampton Buildings, Chancery Lane, London, W.C.2, do hereby declare the nature of this invention (a communication from Standard Oil Development Company, a corporation duly organised and existing under the laws of the State of Delaware, United States of America, having an office at Linden, New Jersey, United States of America) and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

The present invention relates to the production of fuels and more specifically to the production of gaseous and liquid fuels including fuel gases and motor fuels from solid and heavy liquid carbonaceous materials, such as coal, coke, peat, tar sands, oil shale, heavy oil residues and the like.

Various methods of converting carbonizable materials, such as coal, coke, oil shale, and the like into fuel gases and liquid fuel products are known to the art. The conventional procedure most widely practiced in the manufactured gas industry involves a carbonizing or coking treatment of carbonizable solids, such as coal, to produce coal gas together with valuable normally liquid hydrocarbons and other valuable byproducts followed by a gasification treatment of the coke with steam and oxidizing gases to produce water gas or producer gas. The carbonization stage yields normally gaseous and liquid fuel products mainly consisting of hydrogen and hydrocarbons of high calorific value in amounts varying between 10 and 40% of the charge, depending on the quality of the carbonizable material. In all practical cases the major portion of the carbonaceous charge is converted into gas mixtures containing varying amounts of CO and H_2, the calorific value of which runs about 300 B.Th.U. per cu. ft. of gas. In order to produce liquid of a high calorific value, it has been proposed to subject the gas mixtures containing CO and H_2 to a catalytic synthesis reaction to produce liquid hydrocarbons therefrom. The tail gas from the hydrocarbon synthesis which consists of normally gaseous hydrocarbons having 1 to 4 carbon atoms per molecule, unconverted CO and H_2 and inert constituents, such as CO_2, may be used as a high B.Th.U. fuel gas, while the liquid synthesis products represent an additional yield of motor fuels. This combined carbonization, gasification and synthesis process affords a highly efficient and substantially complete conversion of solid carbonaceous material into fuel gas and motor fuels of high B.Th.U. value, particularly since it has become possible to overcome the numerous drawbacks of periodic fixed bed operation by the application of the well known fluid solids technique which permits continuous operation of any one or all of the conversion stages.

However, even this greatly improved conversion procedure still leaves a problem of considerable magnitude unsolved. This is the adjustment, with the necessary speed and economy, of the rate of fuels production to the strongly fluctuating rate of fuels consumption. This problem is greatly complicated by the fact that the fluctuations of the rates of gas consumption and motor fuel consumption are by no means parallel. The gas consumption rate goes through a maximum and a minimum at different hours of the day; it reaches a peak in winter and a definite low in summer. Motor fuel consumption, on the other hand, does not fluctuate during a day in a manner appreciable for the manufacturer but has a definite peak in summer and reaches its minimum during the winter months. Heretofore, the manufactured gas industry has coped with the fluctuations of gas consumption by the use of stand-by equipment which can be put into operation and taken out of operation within a relatively short time. Such method of production control, if consistent quality specifications of the fuel gas have to be met, requires stand-by equipment for all conversion stages, which is in use only part of the time and thus constitutes a heavy load on plant investment and process economy. Moreover, the production of more or less fuel gas entails a parallel rise and drop in the motor fuel production in a manner which is not desirable since motor fuel demand will normally vary inversely as fuel gas demand. This situation calls for the provision of extensive gas storage space to act as a buffer for the fluctuations in consumption, which is so expensive as to be impractical.

The present invention overcomes the aforementioned difficulties and affords various additional advantages. These advantages, the nature of the invention and the manner in which it is performed will be fully understood from the following description thereof read with reference to the drawing which shows semi-diagrammatic views of apparatus particularly adapted to carry out the invention.

It is the main object of this invention to provide a process of improved flexibility for the conversion of solid and heavy liquid carbonaceous materials of the type specified into normally gaseous and liquid fuel products.

Another object of the present invention is the conversion of solid and heavy liquid carbonaceous material into fuel gases and motor fuels in a continuous process which may be operated at a constant optimum process efficiency and readily adapted to fluctuations in fuel gas and motor fuel consumption rates without a change of operating equipment capacity.

Another object of this invention is an improved process of the type specified which permits variations of fuel gas production and motor fuel production in opposite directions without adversely affecting process efficiency, operating equipment capacity or product quality.

A still further object of the invention is the conversion of solid and heavy liquid carbonaceous materials into fuel gas and motor fuels by an improved continuous combined gasification and hydrocarbon synthesis process applying the fluid solids technique and permitting full and instant adaptation of rates of fuel production to rates of consumption of either fuel without adversely affecting process efficiency, operating equipment capacity or product quality.

Other and further objects and advantages of the present invention will appear from the following more detailed description and claims.

It has been found that these objects and advantages may be accomplished quite generally by converting the solid and heavy liquid carbonaceous starting materials in a first conversion process into gas mixtures comprising CO and H_2, subjecting the CO and H_2 produced to a catalytic hydrocarbon synthesis reaction to produce liquid fuels and fuel tail gas to be used as city gas and providing means permitting a recycle of a portion of the synthesis tail gas to the first conversion process in such a manner as will improve the efficiency of the combined process with respect to the conversion of the carbonaceous fuel charge into liquid and gaseous fuel products in the desired proportions.

In accordance with one embodiment of the invention, the recycled synthesis tail gas is used as fuel in the first conversion process to supply at least a portion of the heat required for carbonization and/or gas generation, in place of a corresponding amount of carbonaceous fuel charge which otherwise would have to be burnt for this purpose. Another embodiment of the invention comprises a recycle of synthesis tail gas to the water- or producer-gas generation stage of the first conversion process in order to modify, by reforming the hydrocarbons, the CO and H_2 content of the gas generated and/or to enrich the synthesis feed gas and thus the synthesis tail gas used as city gas with gaseous hydrocarbons.

It will be readily appreciated that the amount of synthesis tail gas recycled to and used in the conversion process may be adjusted in accordance with the fuel gas requirements of the market. When the fuel gas demand is high the proportion of synthesis tail gas recycled will be small and vice versa. Simultaneously, the liquid fuel production may either be maintained at a constant absolute rate or adjusted to demand fluctuations by an appropriate control of the synthesis conditions in a manner known per se, for example, by a change of the synthesis temperature from optimum for liquid production, if it is desired to reduce the output of liquid synthesis products, or vice versa. During the periods when maximum liquid yield is not desired, plant operation may be adjusted to reduce cost, as, for instance, using catalyst longer without reactivation or replacement, decreasing the number of stages in operation, if more than one is normally used, etc. If it is desired to control the synthesis conditions so as to keep the conversion of CO and H_2 low, say below 90%, for instance in order to assist in adjustment of heating value and specific gravity of the tail gas, or to control distribution of synthesized hydrocarbons, or ratio of elimination of oxygen as H_2O and CO_2, the synthesis tail gas contains CO and H_2 and is of a relatively low B.Th.U. value; in this case, it is of particular advantage to recycle part of the synthesis tail gas through the gas generation stage of the first conversion process whereby the hydrocarbon gas content of the synthesis feed gas may be increased and the synthesis tail gas marketed as fuel gas may be enriched with hydrocarbon gases. It is evident that these embodiments and modifications of the present invention may be combined in any desired manner to adapt the yields of liquid and gaseous fuels to any conceivable fluctuations in the quantitative and qualitative demands of the market without any need for stand-by equipment or excessive storage space.

While the utilization of synthesis tail gas in accordance with this invention may be carried out within certain limits in synthesis gas manufacture by conventional fixed bed techniques using ordinary water gas or producer gas sets charged with coke, it has been found that practically unlimited flexibility of the process may be accomplished and any desired amount of the synthesis tail gas may be used in the synthesis feed gas plant when the latter is operated by the fluid solids technique. Such a fluid technique synthesis gas plant may, for instance, comprise a gas generation stage in which fluidized finely-divided solid carbonaceous material is reacted with steam to produce water gas, while the heat required for the water gas reaction is supplied in the form of sensible heat of hot solid residue from a separate fluidized combustion stage or heater wherein a portion of the carbonaceous solids is subjected to combustion with air. The arrangement of a separate heater and gas generator affords the additional advantage that synthesis tail gas may be continuously recycled in any desired proportions simultaneously or alternately as substitute fuel to the heater and as a gas modifier to the gas generator, thus enhancing the flexibility of this process. It should be understood, however, that this invention is applicable to any other type of fluid fixed or moving bed synthesis gas plant.

Having set forth the general nature and objects, the present invention will be best understood from the more detailed description hereinafter, in which reference will be made to the accompanying drawing wherein:—

Fig. 1 is a schematic flow plan illustrating in a general way a system suitable for carrying out the process of this invention; and

Fig. 2 is a partly schematic and partly diagrammatic illustration of a system suitable for carrying out a preferred embodiment of this invention.

Referring now in detail to Fig. 1, the system schematically illustrated therein essentially comprises a synthesis gas plant 2 for manufacturing synthesis gas containing CO and H_2 from carbonaceous fuels, such as coal, oil shale, heavy oil residues, coke or the like, and a hydrocarbon synthesis plant 6 for the catalytic conversion of CO and H_2 to synthetic liquid and gaseous fuels. Both the synthesis gas plant 2 and the hydrocarbon synthesis plant 6, may be of any design known to the art. For example, the synthesis gas plant 2 may consist of a single gas generation stage for the conversion of coal or coke with steam and oxidizing gases to water gas, or it may comprise a carbonization stage for the production of coal gas, liquid carbonization products and coke, in addition to the water gas producing stage. The heat required for water gas production and/or carbonization may be supplied by partial combustion of carbonaceous material in a make and blow or continuous manner, within the carbonization and/or water gas producing zones or in a separate heater. Temperatures may vary between about 1000° and 2000°F. for the carbonization, 1400°-2200°F. for the water gas generation, and 1800°-2500°F. for separate heaters, depending on the character of the starting material and the products desired. Fixed bed, moving bed or fluid solids technique may be employed in a manner known per se. Means for separating and refining gaseous and liquid carbonization products may be provided. The hydrocarbon synthesis plant 6 may comprise conventional means for purifying, particularly desulfurizing, the synthesis gas, one or more fixed bed, moving bed or, fluid catalytic reactors and a product recovery system for separating liquid from gaseous synthesis products, as well as conventional oil-refining installations. The usual synthesis catalysts such as metals of the iron group, particularly iron or cobalt, if desired, supported on siliceous carriers may be used at temperatures ranging from about 350° to about 800°F. and pressures ranging from about 1 to about 50 atm. absolute, depending on the composition of the synthetic products desired. These conditions are well known in the art and do not as such form a part of the present invention. However, this invention includes the proper correlation of all such conditions as affect the relative proportions of liquid and gaseous final products to the recycle of synthesis tail gas to the synthesis gas plant, as will appear more clearly hereinafter.

In operation, the synthesis gas plant 2, which may be kept under the same pressure as the hydrocarbon synthesis plant, converts the carbonaceous charge supplied, as indicated at 1, to synthesis gas taken off through 3 and, if desired, to coal gas and liquid fuels from carbonization, which may be refined in any conventional manner, recovered through lines 4 and 5, and marketed as fuel gas and liquid motor fuels, respectively. The synthesis gas is passed to the hydrocarbon synthesis plant and converted after suitable purification to liquid hydrocarbon products and tail gas containing unconverted CO and H_2, hydrocarbon gases having 1 to 4 carbon atoms per molecule and inerts such as CO_2 and N_2.

The relative amount and composition of the synthetic liquid products and the synthesis tail gas may be readily adjusted to market demands by a proper choice of the synthesis conditions. For example, the following tabulation compares typical processing conditions and results, using an iron catalyst, involved in controlling liquid/gaseous hydrocarbon selectivity ratio, and unsaturation of the lower boiling hydrocarbons synthesized.

Type Operation	*High Liq. Yld.*	*High Tail Gas Make Low B.Th.U.*	*High Tail Gas Make High B.Th.U.*	*High Cunsats.*
Synthesis Temp. °F.	600	550—575	625	600—625
Vol. fresh syn. gas/vol. cat. space/hr. ...	500	600	300	1000
Effective cat. age	Moderate	Moderately old	Old	Moderate
Pressure, atm....	20	10	10	20
Ratio B.Th.U. in liq./B.Th.U. in Gas	1.2—3.0	0.6—1.2	0.4—1.2	1.2—2.5
% unsats. in C_3—C_5 ...	—	—	—	75—90
Tail gas composition				
% H_2 ...	2—15	10—40	1—10	5—20
% CO	1—5	5—15	1—5	1—5
% CH_4+C_2's	10—30	5—25	25—50	10—30
% CO_2+N_2	50—75	30—60	35—60	50—75

The desired shift may sometimes be obtained by changing only some of the variables indicated for change above. Exact composition of catalyst may also affect product distribution and properties, especially as influenced by promoters. Characteristics of the catalyst may be modified without suspending operation of the plant, if desired, by adding continuously or periodically to the reaction zone individual catalyst components as desired.

As mentioned above, the liquid synthetic products may be refined by any conventional method, such as thermal and/or catalytic cracking, hydrogenation, reforming, isomerization, alkylation, polymerization, etc. When the synthesis reaction is carried out under conditions conducive to the formation of large proportions of unsaturates, the present process permits the production of highly valuable antiknock motor and aviation fuels. For this purpose, the liquid carbonization products recovered in the synthesis gas plant may be treated to separate the fraction containing benzene and other suitable aromatics boiling within the motor fuel range and these aromatics may then be alkylated, using unsaturates from the synthesis reaction, in the presence of a catalyst, such as aluminum chloride, phosphoric acid or the like in a manner known per se. C_3, C_4, and C_5 unsaturates will normally be desired for this purpose, and the alkylation may be carried out free from or in the presence of other synthesis products in the same boiling range as the alkylating unsaturates. The finished liquid fuel is passed to storage through line 7.

Synthesis tail gas of proper composition and B.Th.U. content to comply with any desired market specifications for fuel gas is withdrawn through line 8. A portion corresponding to the market demand is branched off through line 9 and piped to places of consumption. CO_2 may be removed using known scrubbing processes as necessary to assist in meeting specifications. Alternatively, partial removal of CO_2 may be accomplished by washing the gas with a basic solvent under pressure and flash expelling the CO_2 from the solution at atmospheric pressure without heating. It will also be understood that composition, heating value, and specific gravity of the tail gas may be further adjusted by blending with any desired amount of synthesis gas bypassed around the synthesis reactor. Other known means of adjusting gas composition to assist in meeting specifications, in such combination and order as may be desired, may also be employed. Any excess fuel gas produced is passed through recycle line 10 and returned to the synthesis gas plant either to serve as a substitute fuel to to supply heat therein or to modify the composition of the synthesis gas produced in the gas generation stage or both, as indicated above and described in more detail in connection with Fig. 2 of the drawing.

Referring now to Fig 2, the system shown therein illustrates a preferred embodiment of the present invention involving the application of the fluid solids technique in the synthesis gas manufacturing plant which, by way of example, is shown to comprise essentially a fluid-solids gas generator 202 and a fluid-solids heater 210. Some optional and some necessary conventional auxiliary equipment, such as coking or carbonizing equipment, gas-solids separators, preheaters, means for fluidized solids feed and withdrawal, valves, etc., have been omitted from the drawing in the interest of brevity and simplification because this auxiliary equipment and its operation do not form a part of the present invention.

In operation, finely-divided solid carbonaceous material, such as coal, oil shale, coke or the like, of a roughly uniform fluidizable particle size which may fall between the approximate limits of 400 mesh per linear inch and 1/4 inch diameter is fed in any manner known per se through line 201 to gas generator 202. If coke is used it may be derived from a previous carbonization stage of the same system. The finely-divided solids are maintained in generator 202 in the form of a dense, turbulent, ebullient mass fluidized by steam supplied through line 203 to the lower portion of generator 202. The amount of steam should be sufficient to convert a substantial portion of the carbonaceous constituents of the solids into water gas at temperatures of between about 1400° and 2000°F. At least a substantial portion of the heat required for the gasification reaction is supplied as sensible heat of solid combustion residue recirculated from heater 210, as will appear hereinafter. The superficial velocity of the steam-gas mixture in generator 202 is such as will cause the desired fluidization in generator 202 and may generally vary between 0.5 and 10 ft. per second, depending on the size and density of the solids charged.

Solid fluidized gasification residue, which may still contain about 0.5 to 5% of carbonaceous constituents, is withdrawn downwardly from generator 202 through line 204 which may have the form of a fluidized standpipe, and suspended in air or other oxidizing gas flowing through line 205. The suspension enters heater 210 through line 206 which may receive synthesis tail gas recycled through line 208 from the hydrocarbon synthesis plant, as will appear hereinafter. The solids form in heater 210 a dense, turbulent, ebullient bed fluidized by the air, the synthesis tail gas and the flue gas produced. Combustion of the carbonaceous constituents of the solids and the CO, H_2 and gaseous hydrocarbons of the synthesis tail gas added through line 208 takes place at temperatures which may vary between about 1800° and 2500°F., depending on the oxygen supply which may be adjusted to the heat requirements in gas generator 202. Flue gas is taken overhead through line 212 to be used for any desired purpose including heat recovery for the system. Fluidized solid combustion residue is withdrawn downwardly and passed substantially at the heater temperature through line 213 to gas generator 202 in amounts sufficient to supply the heat required therein. Excess solid combustion residue may be withdrawn through line 214 and discarded after heat recovery for the system. The rate of circulation of hot solids from heater 210 to generator 202, of course, depends on the temperature difference between heater 210 and generator 202 but may, in general, vary between 25 lbs. and 200 lbs. of solids recirculated per lb. of fresh feed to generator 202. If desired, additional heat may be generated by partial combustion within generator 202, oxygen or air enriched with oxygen being supplied together with the steam.

Raw synthesis gas is taken overhead from generator 202 and passed through line 215 to hydrocarbon synthesis plant 218 to be purified and converted into synthetic liquid fuel and fuel tail gas recovered through lines 219 and 220 respectively, in a manner similar to that outlined in connection with synthesis plant 6 of Fig. 1. The gas required by the market is sent through line 221 to the pipe lines, while any excess tail gas is recycled through lines 222 and 208 to the synthesis gas plant. As indicated in the drawing, recycle tail gas may be returned to heater 210 through lines 208 and 206 or to generator 202 through lines 224 and 203 or to both. In the first case, the tail gas serves as additional fuel in heater 210 with the result that the amount of solid carbonaceous feed to generator 202 may be reduced by a corresponding proportion. In the second case, the tail gas may be used to modify the composition of the synthesis gas with respect to its content of CO, H_2 and/or gaseous hydrocarbons. If desired, the conditions of generator 202 may be so controlled, for instance, by raising the generator temperature above about 1800°F. that a substantial proportion of the hydrocarbons present in the tail gas is converted to additional amounts of CO and H_2.

It will be readily understood from the foregoing that systems operated in accordance with this invention are of practically unlimited flexibility with respect to possible adjustments to quantitative or qualitative fluctuations of the requirements of the fuel gas market. These changes may be made by operating at all times at a constant maximum process efficiency by mere recycle of excess fuel gas without affecting the output of liquid motor fuels which may be kept at any desired constant value. If desired, however, the liquid fuel production may be likewise varied by a proper control of the conditions of the hydrocarbon synthesis without affecting the amount of fuel gas available for the market. Also, certain fractions, particularly those boiling above the motor fuel range, may be recycled to the synthesis gas manufacturing stage in a similar manner as the tail gas. Preferably, the system will be so designed that there is a safe margin of excess fuel gas to be recycled at even the highest rate of fuel gas demand. Operation in this manner does not involve a loss in efficiency since the gas recycled may be utilized to generate heat required for the process. Thus, this process may be operated during the summer to produce minimum quantities of fuel gas and a maximum quantity of liquid fuel. During the winter, however, when gas demand increases and gasoline demand decreases, the gas recovery may be raised accordingly with a simultaneous decrease of the liquid fuel production and this without deviating from optimum plant operating conditions throughout the year.

The present invention will be further illustrated by comparison of the following specific examples.

EXAMPLE 1.

A plant with a synthesis gas production capacity of 100 million cu. ft./day of $CO + H_2$ is available. It is desired for winter operation to supply to consumers 37 million cu. ft./day of manufactured gas of 540 B.Th.U./cu. ft. heating value and 0.6 specific gravity. By operating at a temperature of 550°-575°F. with a catalyst of moderately great average age, 80% of the $CO + H_2$ is converted to gaseous and liquid hydrocarbons and slightly oxygenated hydrocarbons. The tail gas from the operation, including the 20% unconverted $H_2 + CO$, is scrubbed of about 10 million cu. ft./day of CO_2, thereby making available a fuel gas meeting the above specifications, and having a composition of 25% CO, 31% H_2, 15% N_2, 20% CH_4, and 9% higher hydrocarbon gases. No gas is returned to the synthesis gas manufacturing section. 2400 bbls/day of liquid or liquefiable product, all of which is or can easily be converted into high octane gasoline, and 250 bbls/day of alcohols, etc. are also recovered. Total coal consumption of the plant is 1800 tons per day.

EXAMPLE 2.

The plant of Example 1 is required to supply during the summer only 15 million cu. ft./day of manufactured gas to consumers. The operating temperature of the synthesis reaction is increased to 600°F., and the catalyst replacement rate is raised until the average age of the catalyst in use is significantly lower than that of Example 1. 95% of the $CO + H_2$ is converted to gaseous hydrocarbons, liquid hydrocarbons, and oxygenated compounds. 3800 bbls/day of liquid or liquefiable hydrocarbons and 400 bbls/day of alcohols, etc., similar to the liquid production of Example 1, are recovered. 13,000 million B.Th.U./day of tail gas are available, but only 8,000 million B.Th.U./day are required for consumer supply after treatment to meet specifications. Tail gas equivalent to 5,000 million B.Th.U./day is therefore returned to the synthesis gas manufacturing section, and total coal consumption of the plant is reduced to 1600 tons per day.

While the foregoing description and exemplary operations have served to illustrate specific applications and results of the present invention, other modifications obvious to those skilled in the art are within the scope of this invention.

Having now particularly described and ascertained the nature of the said invention, and in what manner the same is to be performed, as communicated to me by my foreign correspondents, I declare that what I claim is:—

1. A process for controlling the liquid/gas product distribution in the production of liquid and gaseous fuels by the conversion of carbonaceous materials into a gaseous mixture containing carbon monoxide and hydrogen, and the subsequent catalytic treatment of said mixture to produce normally liquid hydrocarbons and a fuel tail gas wherein the said fuel tail gas is recovered and a portion thereof in excess of the desired requirement of fuel gas is recycled to the reaction for converting the carbonaceous material into the carbon monoxide-hydrogen mixture.

2. A process according to Claim 1, wherein the reaction for converting the carbonaceous material to carbon monoxide and hydrogen is carried out by a process comprising a gasification stage, preferably preceded by a carbonization stage, and a combustion stage, wherein the portion of the gaseous products from the subsequent synthesis reaction recycled to this reaction is supplied to one or more of said stages.

3. A process as claimed in Claim 1 or 2, wherein the fuel gas fraction obtained from the synthesis reaction is used as an industrial and/or domestic fuel gas, and the amount recycled to the synthesis gas producing reaction is decreased as the demand for such fuel gas increases, and is increased as the demands for such fuel gas decreases or the demand for liquid fuels increases.

4. A process as claimed in Claim 2, wherein the process for producing the mixture of carbon monoxide and hydrogen from the carbonaceous material comprises a gasification stage and a combustion stage wherein the solid residue from the gasification stage is subjected to a combustion reaction with an oxidizing gas, the heated solid residue therefrom being transferred to the gasification stage to supply at least part of the heat required therein.

5. A process as claimed in Claim 4, wherein the carbonization stage, if used, the gasification stage and the combustion stage are carried out in separate zones, with the solid material in each zone in the form of dense, ebullient masses of fluidized solid, and the said solids are transferred from zone to zone in fluidized form.

6. A process as claimed in any of the preceding claims, wherein the catalytic conversion of the carbon monoxide and hydrogen mixture is maintained below about 95% of the total theoretical conversion.

7. A process as claimed in any of the preceding claims, wherein at least a part of the heat required for the conversion of the carbonaceous material into a mixture containing carbon monoxide and hydrogen is supplied by the combustion of part of the fuel gas recycled to the said reaction from the catalytic synthesis reaction.

Dated this 6th day of August, 1946.

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[This Drawing is a reproduction of the Original on a reduced scale]

Fig. 1.

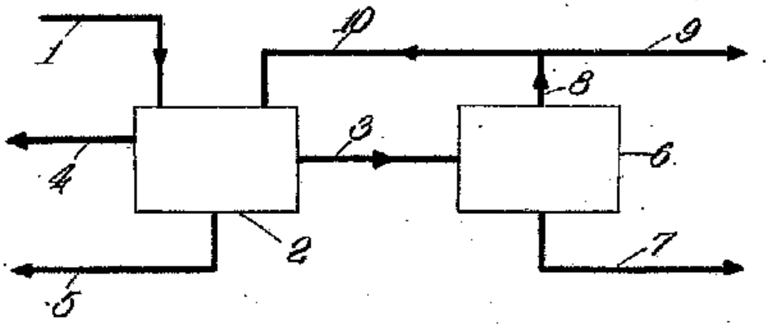
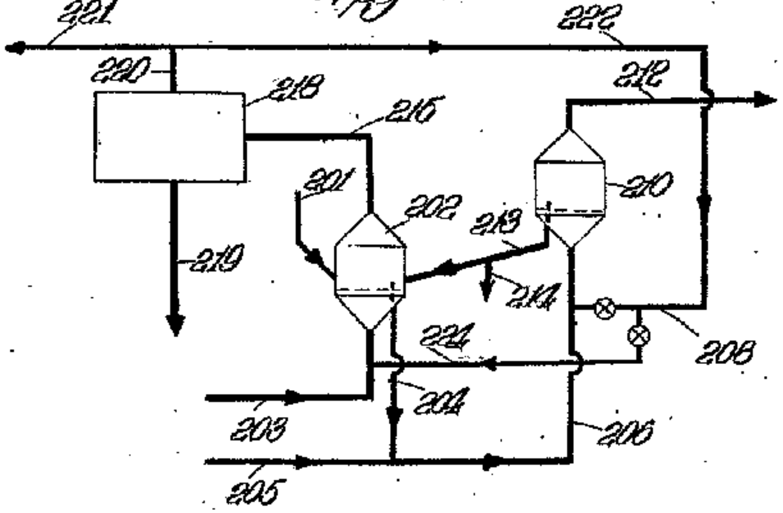


Fig. 2.



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