Oct. 31, 1961 J. VAN POOL 3,006,739

APPARATUS FOR CONTACTING IMMISCIBLE FLUIDS

Filed Dec. 3, 1958 2 Sheets-Sheet 1

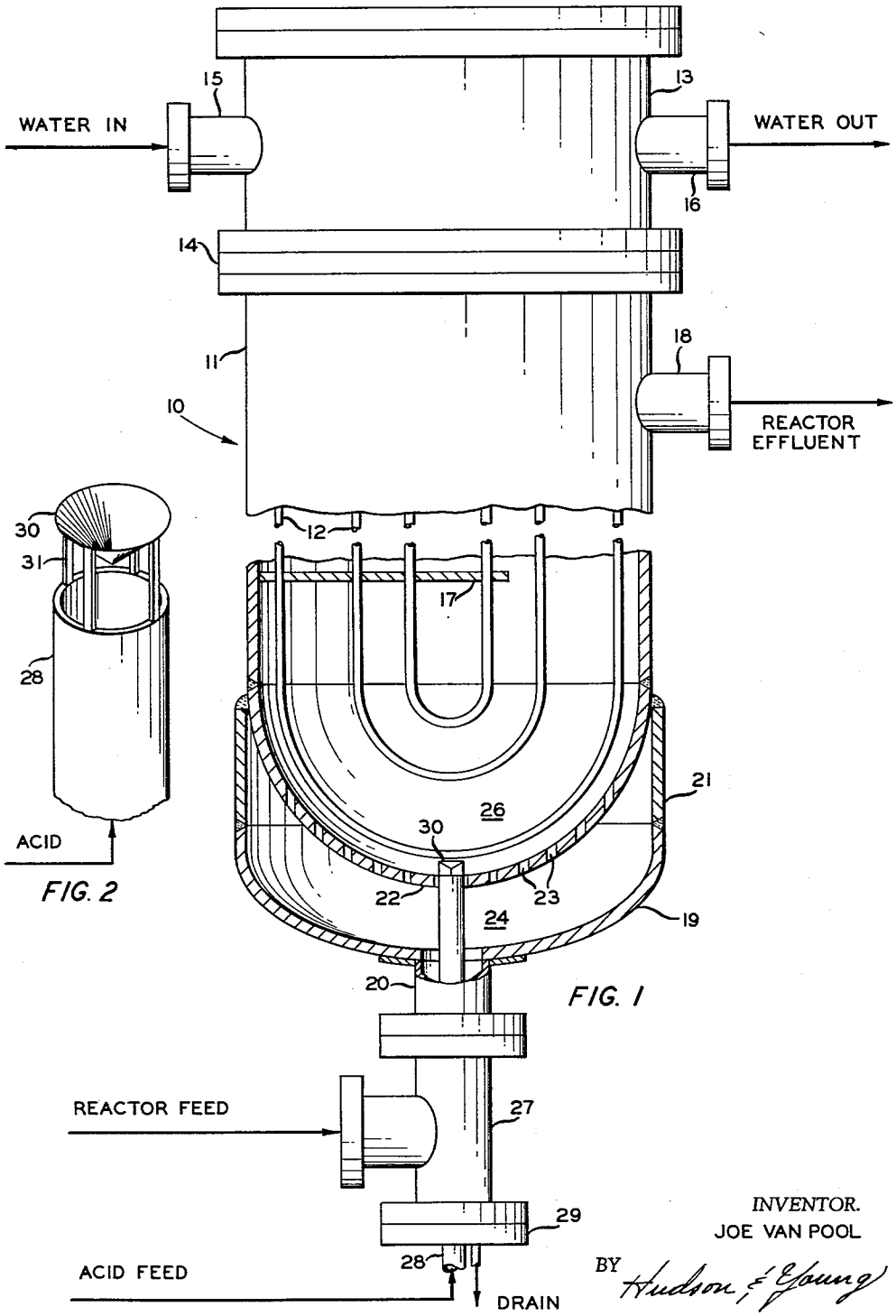


FIG. 2

FIG. 1

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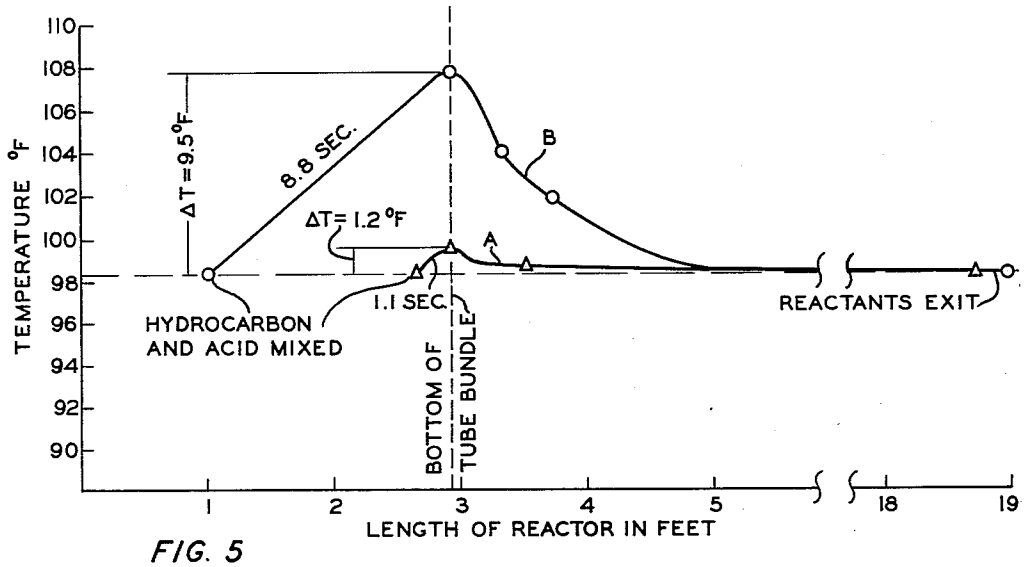


FIG. 5

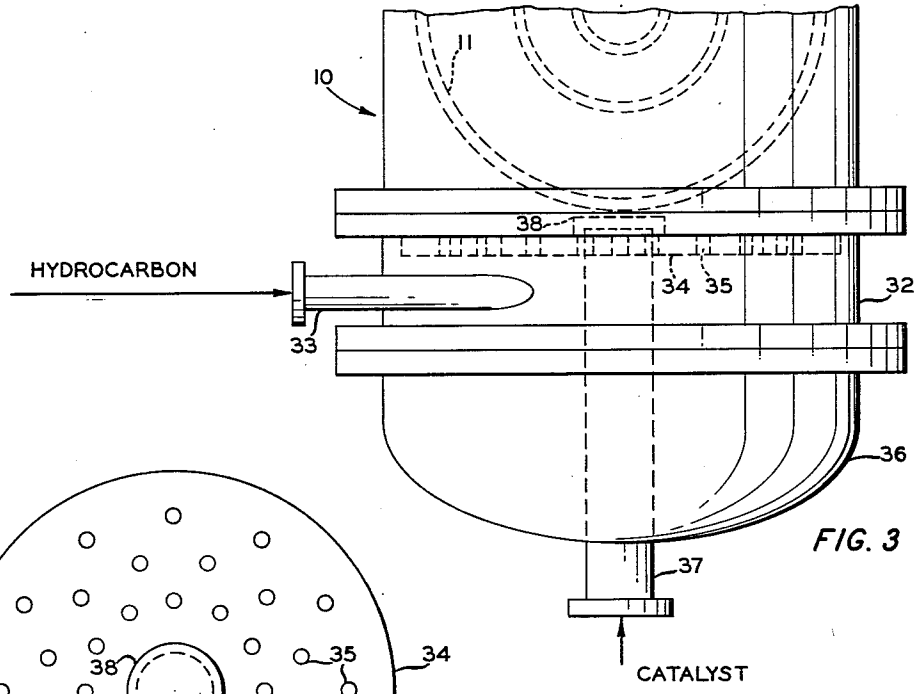


FIG. 3

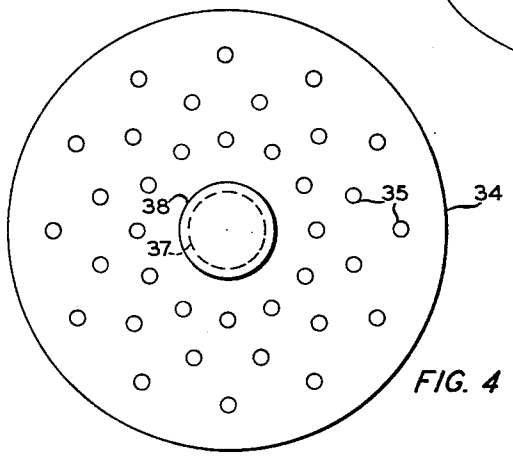


FIG. 4

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United States Patent Office 3,006,739 Patented Oct. 31, 1961

3,006,739

APPARATUS FOR CONTACTING IMMISCIBLE FLUIDS

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Filed Dec. 3, 1958, Ser. No. 777,891
8 Claims. (Cl. 23—285)

This invention relates to a method and apparatus for intimately contacting at least two immiscible fluids. In another aspect this invention relates to a method and apparatus for converting a standard U-tube heat exchanger into an efficient alkylation reactor.

Many processes and apparatus have been devised for contacting immiscible fluids under conditions of strict temperature control. Such processes and apparatus are employed in physically contacting operations, such as the washing of hydrocarbons with various liquid treating agents, and in various chemical reactions wherein hydrocarbons are reacted in the presence of an immiscible liquid catalyst, such as in the alkylation of an isoparaffin with an olefin in the presence of liquid, anhydrous, hydrofluoric acid. Many types of prior art apparatus employ mechanically driven agitators to insure intimate contacting between immiscible fluids while other rely upon ducted internal recirculation. In many reactions, and particularly in alkylation, such as above-mentioned, the effect of temperature is quite critical and removal from or addition of heat to the reaction as may be required is an essential feature of the process, often dictating and limiting the design of the apparatus to be employed. Most prior art apparatus which is used for alkylation processes has been specifically designed for this purpose and for this very reason such apparatus is generally quite expensive.

I have by my invention provided apparatus and a method by which a standard U-tube heat exchanger can easily be converted into an efficient alkylation reactor. This conversion is effected by removing from the heat exchanger the head adjacent the tube bends and replacing this head with a concave closure member containing a confined zone formed by a transverse perforated partition spaced from the closed end thereof. Preferably this closure structure comprises a short extension of the heat exchanger shell, normally cylindrical in shape, a head member closing one end of the extension and a perforated partition closing the other end of the extension to form a confined zone between the head and the partition. This assembly is fastened to the heat exchanger shell in the position from which the original head was removed so that the perforated partition is adjacent the tube bends. An inlet is provided for introducing one fluid into said confined zone and a separate conduit is installed for introducing a second fluid across the face of said partition nearest the tube bends. Preferably this conduit passes through said zone and through the partition terminating in an opening adjacent the partition outside said zone with a baffle disposed in front of the opening to deflect fluid leaving this conduit transversely across the face of the partition. Improved mixing is obtained when one liquid is forced into the confined zone of this adapter thus causing this liquid to jet through the perforations in the partition into the volume of the heat exchanger served by the U-tube bundle. The second liquid is added through said conduit and, upon striking the baffle positioned at the end of this conduit, disperses radially across the surface of the partition intercepting the liquid streams jetting through the perforations in this partition. Preferably the reactor is operated in a vertical position so that gravitational forces will not tend to stratify the liquids within the reactor. Turbulent mixing occurs immediately upon entering the volume of the heat exchanger served by the tube bundle and the cooling or heating, as the case may be, is immediately supplied by the heat exchange means. Advantages of my invention lie in the efficiency of contacting between the two liquids passed through the reactor with the provision for immediate cooling or heating as soon as the liquids are mixed. The simplicity of the arrangement and the construction which enables employing a standard heat exchanger greatly reduces the cost of the reactor thereby placing many processes economically within the reach of small operators. Since U-tube heat exchangers have many applications they enjoy the cost advantages of mass production. Therefore, the use of this equipment in my invention is of considerable importance even in large refineries.

It is an object of my invention to provide an improved contacting apparatus for immiscible fluids. Another object is to provide a reactor which employs as a basic unit of its structure a standard U-tube heat exchanger. Another object is to provide a method of converting such a heat exchanger into an efficient reactor. Another object of my invention is to provide apparatus which is useful in adapting a U-tube heat exchanger to use as an alkylation reactor. Still another object is to provide a method of mixing immiscible fluids, at least one of which undergoes a thermally sensitive chemical transition in the presence of the other, with adequate temperature control. Another object is to provide a method for improving the octane rating of an isoparaffin-olefin alkylate. Other objects, advantages and features of my invention will be apparent to those skilled in the art from the following discussion and drawings in which:

FIGURE 1 is a view of the reactor of my invention showing the lower portion in section;

FIGURE 2 is an isometric detail of the preferred baffle to be used in dispersing one of the fluids across the face of the perforated partition;

FIGURE 3 depicts an alternate arrangement for converting a U-tube heat exchanger to an alkylation reactor;

FIGURE 4 shows the arrangement of orifices in the perforated plate of FIGURE 3; and

FIGURE 5 is a graph showing the effect of the relative position of points of initial mixing and initial cooling upon the reaction temperature in the acid-catalyzed alkylation of an isoparaffin with an olefin.

To more fully explain my invention reference is made to FIGURE 1. A standard U-tube heat exchanger **10** is shown adapted to serve as an alkylation reactor. This heat exchanger has a shell **11** housing a U-tube bundle indicated by tubes **12**. These tubes open into baffled header **13** which with plate **14** forms the closure for the upper end of the heat exchanger shell. Partitions are positioned in header **13** so that water entering through inlet **15** passes downwardly through tubes **12** and returns to a separate chamber in header **13**, leaving through outlet **16**. Such an arrangement is known as a two-pass exchanger. Of course the number of tubes is generally considerably greater than is shown in the drawing which is simplified purposely for the sake of clarity. A number of baffled arrangements in header **12** are possible, as is well known, so that 4- and 6-pass U-tube heat exchangers are available.

It is common practice to employ within such heat exchangers a number of baffles, such as **17**, spaced alternately on opposite sides of the heat exchanger shell so that fluid passing through the open volume of the exchanger shell describes a tortuous path. This increases mixing and heat exchange. The exchanger is provided with an outlet conduit **18** near its upper end and in the reactor of my invention this outlet is employed to remove the mixture of paraffin, alkylate and catalyst. The portion of the reactor described thus far is standard apparatus.

The reactor can be operated in either a horizontal or vertical position but for an alkylation process a vertical position is much to be preferred so that maximum contact with the cooling tubes is assured and separation of immiscible liquids by gravity tends to be prevented by the upward flow of material.

The bottom of the exchanger shell is closed by eliptical head **19** which is equipped with a nozzle **20**. Generally it is desirable to extend the length of the exchanger shell by attaching cylindrical member **21** to the bottom edge of the shell and then fixing the eliptical head **19** to this extension. As shown in FIGURE 1, welding is a convenient method for securing this assembly. Disposed above head **19** and slightly below the bottom of the U-tube bundle is a perforated partition **22**. It is desirable that this partition be positioned quite close to the bottom of the U-tube bundle; for example, within 12 inches of the bottom of the bundle, and for good temperature control the distance from the partition to the heat exchange tubes should be about 2 to 4 inches. As shown in FIGURE 1, this partition is hemispherical in shape, following the contour of the bends in the U-tubes thereby keeping the distance between the partition and the tubes uniform throughout the cross section of the reactor. A plurality of orifices **23** in partition **22** provide communication between zone **24**, which is referred to as the feed zone, and volume **26** of the heat exchanger which is referred to as the contacting zone. As will be apparent to those skilled in the art from my description, it is one of my objects to serve as much of the contacting zone **26** as possible with the heat exchange tubes **12**. This is done in my reactor by keeping the volume between perforated plate **22** and the bottom of the U-tube bundle quite low.

T **27** is attached to nozzle **20** so that feed material enters through T **27** and passes through nozzle **20** into feed zone **24**. This feed material is maintained under pressure so that it jets through orifices **23** in a plurality of streams distributed throughout the cross section of the reactor. In alkylation reactions this material is preferably the hydrocarbon mixture of isoparaffin and olefin. The second liquid, for example, the acid catalyst, enters the reactor through conduit **28**. Conduit **28** passes through seal **29**, closing the bottom of T **27**, upwardly through T **27** and nozzle **20** and completely through feed zone **24**, terminating immediately above or at the upper face of partition **22**. Preferably this conduit is disposed centrally along the axis of the cylindrical reactor. Positioned over the opening of conduit **28** is a deflector plate or baffle **30**. Baffle **30** is designed to deflect liquid issuing from conduit **28** along a radial path so that it travels transversely across the upper face of the partition **22** and intercepts streams of fluid jetting through orifices **23**. A preferred baffle is one having the shape of an inverted cone as shown in FIGURE 2. This baffle can be fixed to the end of conduit **28** by supporting members **31** or the baffle can be fixed to partition **22**. In the latter case it is frequently desirable to allow conduit **28** to be adjustable in height so that the distance between the opening in the end of this conduit and baffle **30** can be adjusted for best operation. Conduit **28** can have a threaded end fitted into a threaded hole in plate **22** so that an adjustment in height can be made by rotating the conduit. Alternatively conduit **28** can be permitted to fit loosely in an opening in plate **22** and be adjustably supported by seal member **29**.

The arrangement shown in FIGURE 1 provides efficient contacting with considerable turbulence as the two fluids are mixed at the bottom of the reactor. The partition **22** prevents any mixing of the liquids in feed zone **24** and by using the arrangement shown the mixing action can be made to take place uniformly throughout the cross section of the reactor. For reasons of simplicity, the arrangement as shown in FIGURE 1 is to be preferred but advantages of my invention in regard to efficient mixing and immediate cooling of the liquids can be enjoyed by employing more than one acid inlet or a branched catalyst inlet with outlets above the perforated partition and a deflector or baffle over each outlet.

One method of readily converting a U-tube heat exchanger into the apparatus shown in FIGURE 1 comprises drilling a plurality of holes in the head of the heat exchanger adjacent the tube bends. In this case the existing head becomes perforated partition **22**. Extension **21** is then fitted around the shell of the heat exchanger, welded into place and eliptical head **19** with nozzle **20** is attached as shown in FIGURE 1. The acid feed conduit is provided with its deflector plate assembled and is positioned within a central opening cut or drilled in the end of the original heat exchanger head. The conversion from heat exchanger to alkylation reactor is then complete.

For reasons pointed out above, the assembly of FIGURE 1 is preferred but my invention can also be practiced with apparatus as shown in FIGURE 3 employing a flat plate for the perforated partition. In this figure a flanged extension and eliptical head are used. To convert a conventional heat exchanger of the U-tube design to an alkylation reactor as shown in FIGURE 3, the head of the exchanger adjacent the tube bends is removed and replaced with a flanged extension **32** having an inlet conduit **33** affixed thereto. Plate **34** having perforations **35** is positioned within this extension at its upper end and the lower end of the extension is closed by head member **36** which can be the original head removed from the heat exchanger. Catalyst conduit **37** passes upwardly through this head member and through the perforated plate **34** terminating slightly above or flush with its upper surface as previously described. A deflecting plate **38** is disposed over the opening of conduit **37** so that flow of material from conduit **38** will intercept the streams issuing from the holes in plate **34**.

A desirable arrangement of the apertures **35** in plate **34** is shown in FIGURE 4. Since the velocity of the discharge stream from conduit **37** is greatest near the conduit, it is desirable to have a greater number of orifices per unit of area near this central conduit. This is the case in the pattern of FIGURE 4 in which the percent of free area in the drill plate is higher near the center of the plate than toward its periphery. The same desirability of design would apply to the hemispherical drilled head of FIGURE 1.

To more fully describe my invention the following examples are presented with conditions for an alkylation reaction between an olefin and isoparaffin in the presence of a hydrogen fluoride catalyst. These conditions are presented as being typical and should not be construed to limit my invention unduly.

Example I

A hydrocarbon feed stream containing a mixture of butylenes, propylene, isobutane and normal paraffins is fed continuously into the feed zone of the reactor of FIGURE 1 through T **27** and nozzle **20** at a rate of 465 barrels per hour. The composition of this stream is as follows:

	Barrels per hour
Olefins	25
Fresh isobutane	30
Recycle isobutane	330
Normal paraffins	80

The ratio of isobutane to olefin on a volume basis is 14.4:1. Hydrofluoric acid is fed continuously through conduit **28** to maintain an acid to hydrocarbon volume ratio of 0.5:1. Pressure in the reaction zone is sufficient to maintain the reactants in the liquid phase and the temperature is maintained as nearly as possible at 98° F. Temperature ranges of such reactions are generally from 50 to 120° F. and preferably the temperature should not exceed 100° F. to obtain best results in octane rating of the finished alkylate.

The reactor mixture passes upwardly through the reactor leaving through outlet **18**. The effluent passes to a separate settling zone, not shown, where the acid and hydrocarbon separate into lower and upper phases respectively. Acid can be recycled. The hydrocarbon is fractionated in well-known steps to recover alkylate and unreacted isobutane which is recycled to the reactor. Alkylate is produced at a rate of 44 barrels per hour, this alkylate having a Research Octane rating with 3 cc. of tetraethyllead added of 104.9. The recycle isobutane stream is 85 percent pure (15 percent n-butane and propane) and is fed back to the reactor at a rate of 338 barrels per hour.

The reactor is a converted U-tube heat exchanger 19 feet long and 5 feet in diameter. The U-tube bundle has 1 inch O.D., 10 gauge tubes providing 7000 square feet of surface area. Partition **22** contains 75 holes ⅜ inch in diameter and is positioned so that the acid and hydrocarbon mixes 3 inches from the bottom of the tube bundle. Cooling capacity of the reactor is sufficient to remove the heat of reaction. The cooling water temperature at the inlet is 86° F. and at the outlet 94° F. The temperature of the reacting mixture along the length of the reactor is shown in the graph of FIGURE 5, line A. It is seen that the temperature rise between initial mixing and initial cooling is about 1.2° F. Flow through the reactor is such that cooling begins about 1.1 seconds after the initial mixing of hydrocarbon and acid.

Example II

By comparison the same reaction is carried out in a reactor in which hydrocarbon and acid catalyst are fed into a common line and enter the bottom of the reactor. Feed rates, stream compositions, cooling water temperature and reaction pressure are the same as in Example I. The reactor of this example is similar to that of Example I except that it is not equipped with perforated partition **22** and the mixing of hydrocarbon and acid occurs in T **27**, 23 inches from the bottom of the tube bundle. The temperature of this reaction mixture as it passes through the reactor is shown by line B on the graph of FIGURE 5. A temperature rise of 9.5° F. is indicated and a period of about 8.8 seconds elapses between the time of initial mixing and initial cooling. Alkylate at 44 barrels per hour is produced but the research octane rating of this alkylate is 103.5 (3 cc. of tetraethyl lead added). It can be appreciated by those skilled in the art that a difference in octane rating of 1.4 points in the range above 100 is appreciable. That such improvement is obtained by the simple arrangement of my invention is believed significant.

It is not believed necessary to give further details regarding the operation of the alkylation of isoparaffins with olefins. Such operating conditions are well known to those skilled in the art. Further details regarding such operating conditions can be found in the copending application of A. B. Leonard and G. R. Hettick, Serial No. 257,522, filed November 21, 1951, now U.S. Patent No. 2,764,623, issued September 25, 1956, and also in Patents 2,393,857, issued to F. E. Frey on January 29, 1946, and 2,410,498, issued to H. J. Hepp on November 5, 1946.

It is believed clear that the method of the invention is not limited to the alkylation of isoparaffins with olefins using HF acid. The invention can also be employed using other liquid catalysts such as aluminum chloride-hydrocarbon complex, sulfuric acid, etc. Likewise, the method of the invention can be employed in carrying out processes wherein immiscible liquids are contacted and/or reacted such as in polymerization and various sweetening operations. The apparatus of the invention can be employed in any process wherein immiscible liquids are contacted and then separated. For example, the apparatus can be used in copper sweetening, amine treating, caustic washing, etc., and in other liquid-liquid operations.

While the invention has been described as employing the heat exchange means of the apparatus for cooling purposes said heat exchange means can be employed for heating purposes if desired.

Various other modifications of the invention will be apparent to those skilled in the art upon reading this disclosure. Such modifications are believed to be within the spirit and scope of the invention.

I claim:

1. A contacting apparatus comprising a U-tube heat exchanger having an elongated shell, a U-tube bundle connected to an inlet and outlet header at one end of said shell, an outlet in said shell near said header and a head member closing the opposite end of said shell, a perforated partition disposed transversely in said shell forming a feed zone between said head member and said tube bundle, means for admitting fluid into said feed zone, a conduit passing into said shell through said partition and terminating near the face of said partition toward said tube bundle, and means for dispersing fluid from said conduit across said face of said partition.

2. A contacting apparatus comprising an elongated vertical shell, a U-tube bundle disposed within said shell through substantially its entire length and opening into discrete inlet and outlet zones disposed at and closing the top of said shell, a head member closing the bottom of said shell, a perforated partition disposed transversely within said shell above said head member and slightly below the lower end of said U-tube bundle, thereby forming a reactant inlet zone between said head member and said partition, a first reactant feed conduit communicating with said inlet zone, a second reactant feed conduit passing through said inlet zone and said partition, terminating centrally within said shell immediately above said partition, baffle means positioned above the outlet of said second conduit for dispersing effluent material transversely above said partition, and upper shell outlet means.

3. A contacting apparatus comprising a cylindrical vertical shell, a U-tube bundle disposed longitudinally within said shell and extending substantially the full length of said shell, a baffled header closing the upper end of said shell and communicating with the tubes in said bundle, a head member closing the lower end of said shell, a perforated partition positioned transversely in said shell above said head member and slightly below said tube bundle thereby forming an inlet zone above said head member communicating with the upper volume of said shell through the perforations in said partition, a first feed conduit communicating with said inlet zone, a second feed conduit passing upwardly through said head member and partition and terminating in a centered opening immediately above said partition, an inverted conical baffle positioned above said opening, said baffle serving to disperse fluid leaving said opening transversely above said partition, and an outlet conduit communicating with the volume within said shell at the upper end thereof.

4. Apparatus of claim 3 wherein said partition is hemispherical in shape following the contour of the lower end of said U-tube bundle.

5. Apparatus of claim 3 wherein said partition is a disk.

6. Apparatus of claim 3 wherein said partition is positioned less than 12 inches from said tube bundle.

7. Apparatus of claim 3 wherein said partition is perforated so that a greater proportion of free area lies toward the center of the partition than towards its periphery.

8. An adapter for use in converting a conventional U-tube heat exchanger having a cylindrical shell into an alkylation reactor by replacing the shell head adjacent the tube bends with said adapter comprising a cylindrical shell extension member having a diameter approximately equal to the heat exchanger shell diameter, a head member closing one end of said extension member, a perforated wall closing the other end of said extension member forming a confined zone therein, first conduit means

communicating with said zone, second conduit means passing through said zone and centrally through said wall and terminating in an opening adjacent the outer face of said wall, and a baffle spaced in front of said opening to deflect material issuing from said opening outwardly across the outer face of said wall.

References Cited in the file of this patent

UNITED STATES PATENTS

2,386,681	Hadden	Oct. 9, 1945
2,720,447	Jones et al.	Oct. 11, 1955
2,855,449	Owen	Oct. 7, 1958