

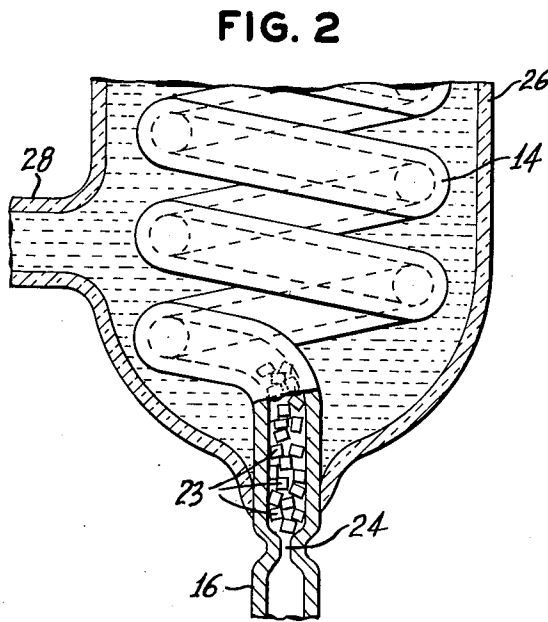
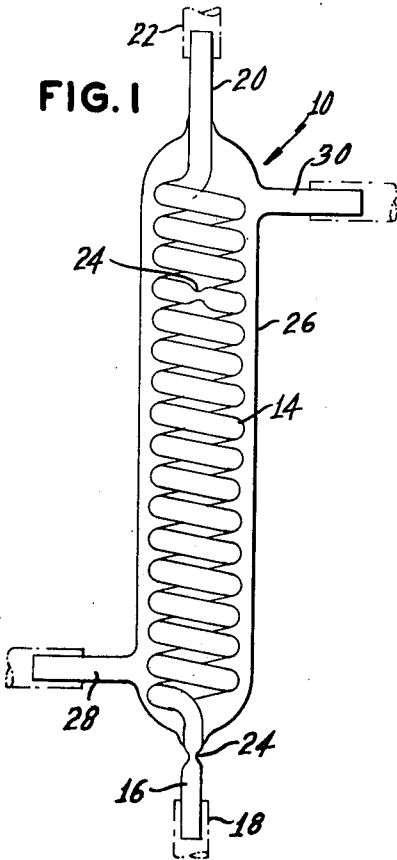
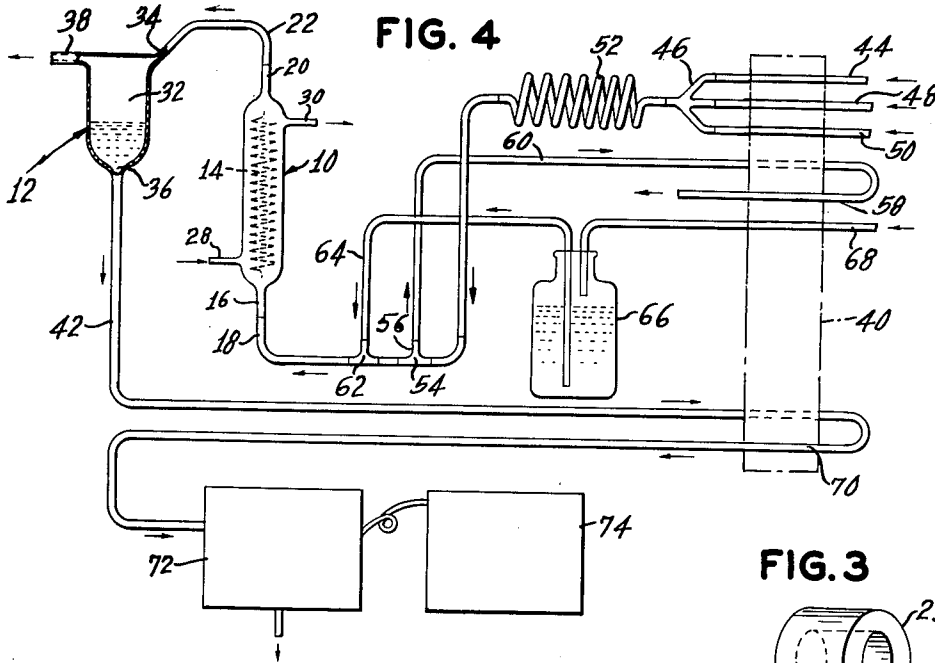
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COCURRENT PACKED HELICAL COIL EXTRACTOR

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## 3,199,956 COCURRENT PACKED HELICAL COIL EXTRACTOR

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This invention relates to apparatus for the separation of a constituent of a liquid by the use of solvent extraction techniques.

One object of the invention is to provide solvent extraction apparatus having improved means for bringing portions of the liquid solvent and the liquid sample into intimate contact with each other for treatment of the sample by the solvent to extract a constituent of the sample by the action of the solvent.

Another object is to provide solvent extraction apparatus of the type indicated with means for separating the resulting constituent-containing solvent from the other liquid of the resulting mixture.

A further object is generally to provide apparatus of improved construction for continuously extracting and separating immiscible liquid phases of different specific gravities, respectively, from a mixture of the phases in a stream.

The above and other objects, features and advantages of the invention will be more fully understood from the following description of the presently preferred embodiment of the invention considered in connection with the accompanying illustrative drawings.

In the drawings:

FIG. 1 is a vertical view of apparatus according to the present invention;

FIG. 2 is a vertical sectional view, on a larger scale, of the lower end of the apparatus of FIG. 1;

FIG. 3 is a perspective view of a part of the apparatus; and

FIG. 4 is a more-or-less diagrammatic illustration of the apparatus in conjunction with apparatus for automatically determining the quantity of a constituent of a liquid.

Referring now to the drawings in detail, the solvent extraction apparatus comprises a liquid contact device 10 for bringing portions of a liquid solvent and a liquid sample into intimate contact with each other for treatment of the sample by the solvent to extract a constituent of the sample by the action of the solvent. A liquid separating device 12 is in fluid flow communication with the contact device 10 and is operable to receive the resulting stream from the latter and separate the resulting immiscible liquids of different specific gravities, respectively, of the stream from each other, as will be more clearly understood hereinafter.

The liquid contact device 10 comprises a helical coil of tubing 14, preferably made of glass sold under the trademark "Pyrex," and it is to be noted that the convolutions of the coil are relatively close to each other so that a relatively long length of tubing is provided but which only occupies a relatively short length. The coil provides a liquid passage for the flow therethrough of the liquid sample and an extracting liquid solvent wherein, during the passage of the stream through the coil, the solvent is effective to extract a constituent of the sample. For introducing the stream containing the sample and solvent liquids, the bottom of coil 14 is provided with a tubular inlet part 16 to which an inlet tube 18 is connected, and the upper end of the coil is provided with a tubular outlet part 20 to which an outlet tube 22 is connected.

The flow passage formed by coil 14 is filled with a plurality of beads 23, preferably made of glass sold under the trademark "Pyrex," which provide liquid contact surfaces that are wetted by the sample and solvent liquids of the stream, whereby all portions of the solvent and sample are brought into intimate contact with each other for the extracting action of the solvent on the sample. Preferably the beads are in the form of hollow cylinders, as illustrated by FIG. 3, so that relatively large areas of contacting surfaces are provided with beads of relatively small dimensions. For example, each bead can be in the order of 1.0 to 1.5 mm. outside diameter, 0.5 mm. inside diameter, and 0.75 to 1.0 mm. long; and with beads of the indicated size, the inside diameter of coil 14 can be in the order of 3.0 mm. To retain the beads in their position in the coil and to permit the flow of liquid into and out of the coil, constricted openings 24 are provided in the coil and are longitudinally spaced from each other with the beads positioned between the constrictions. No beads are provided downstream of constriction 24 at the outlet end of the coil so that the resulting immiscible and separable phases have an opportunity to begin to separate before they flow from the contact device 10.

Coil 14 can be mounted in a jacket 26, also preferably made of glass sold under the trademark "Pyrex," and temperature-controlled fluid is transmitted through the jacket via inlet and outlet tubes 28 and 30, respectively, so that the extracting and separating operations can be conducted under controlled temperatures. The flow of the fluid through the jacket can be controlled by suitable pump means.

The separating device 12 comprises a vertical chamber 32, preferably formed from glass sold under the trademark "Pyrex," and the chamber has an upper inlet 34 which is in liquid flow communication with the outlet part 20 of coil 14 via tube 22, for the introduction of the liquid stream containing the immiscible and separable liquid phases into the separating chamber. It will be understood that the solvent liquid containing the extracted ingredient of the sample constitutes one liquid phase of the mixture of the stream and is separable from the other liquid phase of the stream because it is immiscible with and has a different specific gravity than the other liquid phase of the stream and these physical characteristics are utilized by the separating device 12 for separating the phases from each other. Chamber 32 is also provided with an outlet opening 36 at its bottom for the resulting separated heavier liquid phase of the stream, and is provided with another outlet opening 38 at the upper part of the chamber for the resulting separated lighter liquid phase of the stream.

A proportioning pump 40, preferably of the type shown and described in U.S. Patent No. 2,935,028 issued May 3, 1960, is provided for controlling the relative rate of flow of the resulting separated heavier liquid phase through its outlet 36 and the heavier liquid portion of the incoming stream through inlet 34. More particularly, in the case where the solvent and the extracted ingredient contained therein is heavier than the other liquid of the stream, then pump 40 is operable to pump the separated heavier liquid from chamber 32 through outlet opening 36 at a rate of flow which is less than the rate of flow of the heavier portion of the incoming stream through inlet 34, so that during operation of the apparatus, the heavier liquid phase of the stream settles toward the bottom of chamber 32 while the lighter liquid of the stream moves toward the top of the chamber and flows out through outlet 36 with a small portion of the heavier liquid phase. In this manner, all of the heavier phase is separated from the lighter phase and nearly all of the

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heavier phase is transmitted from the separating device, in the form of a stream, through conduit 42.

In the event that the ingredient-containing solvent is lighter than the other liquid of the stream, then pump 40 is operated so that the rate of flow of the resulting separated heavier phase of the liquid through outlet 36 is greater than the rate of flow of the heavier liquid portion of the stream through inlet 34. This results in the flow of all of the separated heavier liquid and a small portion of the lighter liquid from the chamber through outlet 36 while only the separated lighter liquid flows through outlet 38, whereby nearly all of said lighter liquid can be transmitted, in the form of a stream, separate from the other liquid. As will be understood hereinafter, the separated ingredient-containing solvent stream is analyzed to determine the quantity of the ingredient present in the stream.

Liquid contact device 10 and separating device 12 are especially useful in a continuous treatment and analysis system of the type shown and described in U.S. Patent No. 2,797,149 issued June 25, 1957, for determining the quantity of a substance which is present in a liquid and which can be extracted from the liquid by a solvent. For example, the apparatus is especially useful in monitoring the tetraethyl lead content of the atmosphere at or near manufactories which produce tetraethyl lead. For such use, air from the atmosphere is continuously bubbled through a liquid, for example water, and the resulting tetraethyl lead content of the liquid is determined by transmitting the liquid, in the form of a stream, to the apparatus illustrated by FIG. 4. The liquid is transmitted to pump tube 44 of pump 40 and is transmitted, by the action of the pump, to a fitting 46 where it joins a stream of segmentizing air or other inert and separable fluid simultaneously transmitted through pump tube 48 and a diluent liquid, for example water, simultaneously transmitted through pump tube 50. It is to be understood that the separable fluid introduced through pump tube 48 is free of any tetraethyl lead. At fitting 46, the different streams join each other to form a segmented stream comprising a series of longitudinally spaced liquid segments, separated from each other by intervening fluid segments which help maintain the passages of the apparatus clean, as explained in said U.S. Patent No. 2,797,149.

The constituents of each liquid segment are mixed together in their respective liquid segments as the segmented stream flows through the horizontal helical mixing coil 52. From the mixing coil, the segmented stream passes through a separating device 54 for removing the separable fluid segments from the stream. More particularly, the device comprises an offtake tube 56 which is connected to suction pump tube 58 via conduit 60. Due to the aspirating action of the suction, the segmentizing fluid segments are removed from the segmented stream with a minor portion of the liquid and a consolidated liquid stream is transmitted to fitting 62 where it joins a stream of liquid containing a solvent and a color producing reagent which is transmitted through a conduit 64. In the illustrative example of tetraethyl lead determinations, the solvent for the tetraethyl lead is chloroform and the color producing reagent is dithizone reagent. Since the chloroform might have adverse effects on the material of the pump tubes, it is preferably pumped to fitting 62 through the means of a displacement bottle 66 which contains the solvent and color producing reagent, and a suitable liquid, for example water, is transmitted by pump tube 68, and introduced into the bottle for displacing an equal quantity of the color producing reagent and solvent.

The resulting liquid stream is transmitted through conduit 18 and through the liquid contact device 10 whereby the solvent and reagent are brought into intimate contact with the tetraethyl lead constituent of the stream. The solvent extracts the tetraethyl lead from the liquid and, due to the action of the color producing reagent, the

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liquid phase containing the solution of chloroform and tetraethyl lead, has a color which indicates the quantity of the tetraethyl lead in the solution. This colored liquid is separated from the remaining liquid, by the action of the separating device 12, and is transmitted from the latter through conduit 42 by the action of pump tube 70, to a colorimeter 72 which measures the intensity of the color to provide an indication of the quantity of tetraethyl lead present. The results of the colorimetric analysis are recorded on the recorder 74.

While I have shown and described the preferred embodiment of the invention, it will be understood that the invention may be embodied otherwise than as herein specifically illustrated or described, and that certain changes in the form and arrangement of parts and in the specific manner of practicing the invention may be made without departing from the underlying idea or principles of this invention within the scope of the appended claim.

What is claimed is:

1. Solvent extracting apparatus, comprising a helical coil of tubing providing a liquid passage and having an inlet and an outlet for the flow of liquid through said passage, and a plurality of separate members positioned in said passage in the path of flow of the liquid and providing liquid impingement contact surfaces for bringing the various portions of the liquid into intimate contact with each other, said coil of tubing having a constricted opening providing an inlet for the liquid and having another constricted opening, longitudinally spaced from said first mentioned constricted opening, and providing an outlet for the liquid, said members being disposed in said passage between said constricted openings, and said coil of tubing having convolutions downstream of said constricted opening providing said outlet for the liquid, said convolutions having no members therein.

2. Solvent extracting apparatus, comprising a helical coil of tubing providing a liquid passage and having an inlet and an outlet for the flow of liquid through said passage, a plurality of separate members positioned in said passage in the path of flow of the liquid and providing liquid impingement contact surfaces for bringing the various portions of the liquid into intimate contact with each other, said members being in the form of hollow cylinders, and means in liquid flow communication with said outlet for separating one liquid phase from another liquid phase of the liquid from said passage due to the immiscibility and different specific gravities of the phases, said separating means comprising a vertical chamber having an inlet opening in liquid flow communication with said outlet through which the liquid from said passage is introduced into said chamber, an outlet opening below said inlet opening for flow from the chamber of the resulting separated heavier liquid phase, another outlet opening above said first mentioned outlet opening for the flow from the chamber of the resulting separated lighter liquid phase, and pump means in liquid flow communication with said inlet opening and said first mentioned outlet opening of said chamber for controlling the relative rates of flow of the separated heavier liquid through said first mentioned outlet and the heavier liquid portion of the incoming liquid through said inlet opening.

3. Solvent extracting apparatus, comprising a helical coil of tubing providing a liquid passage and having an inlet and an outlet for the flow of liquid through said passage, a plurality of separate members positioned in said passage in the path of flow of the liquid and providing liquid impingement contact surfaces for bringing the various portions of the liquid into intimate contact with each other, said coil of tubing having constricted openings longitudinally spaced from each other for retaining said members in said passage while permitting the flow of the liquid through said passage, a jacket enclosing said coil and having an inlet and an outlet for the flow of temperature controlled fluid through the jacket and around said coil of tubing, said members being in the form of hollow

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cylinders, and means in liquid flow communication with said outlet for separating one liquid phase from another liquid phase of the liquid from said passage due to the immiscibility and different specific gravities of the phases, said separating means comprising a vertical chamber having an inlet opening in liquid flow communication with said outlet through which the liquid from said passage is introduced into said chamber, an outlet opening below said inlet opening for flow from the chamber of the resulting separated heavier liquid phase, another outlet opening above said first mentioned outlet opening for the flow from the chamber of the resulting separated lighter liquid phase, and pump means in liquid flow communication with said inlet opening and said first mentioned outlet opening of said chamber for controlling the relative rates of flow of the separated heavier liquid through said first mentioned outlet and the heavier liquid portion of the incoming liquid through said inlet opening.

4. Immiscible liquids interaction apparatus comprising:

a helical coil of a plurality of turns of tubing providing a liquid passageway and having an inlet and an outlet for the flow of liquids through said passageway;

a plurality of individual tubular members disposed in the path of flow of the liquids and providing impingement contact surfaces for bringing the various constituents of the liquids into intimate contact with each other;

a jacket enclosing said coil and having an inlet and an outlet for the flow of temperature controlled fluid through said jacket and around said coil;

said coil having a constriction in one of its turns, for dividing said coil into an upstream portion and a downstream portion;

said members being disposed solely in said passageway in said upstream portion, permitting free flow of liquids through said passageway in said downstream portion.

5. Immiscible liquids interaction apparatus comprising:

a helical coil of a plurality of turns of tubing providing a liquid passageway and having an inlet and an outlet;

said coil having a constriction in one of its turns, for dividing said coil into an upstream portion and a downstream portion;

a plurality of individual tubular members randomly disposed solely in said passageway in said upstream portion in the path of flow of the liquids and providing impingement contact surfaces for bringing the various constituents of the liquids into intimate contact with each other, while permitting free flow of liquids through said passageway in said downstream portion;

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a jacket enclosing said coil and having an inlet and an outlet for the flow of temperature controlled fluid through said jacket and around both said upstream and said downstream portions of said coil; and source means of a plurality of immiscible liquids coupled to said inlet of said coil for passing the liquids into said coil under pressure.

6. Immiscible liquids interaction apparatus comprising: a helical coil of a plurality of turns of tubing providing a liquid passageway and having an inlet and an outlet;

said coil having a constriction in one of its turns, for dividing said coil into an upstream portion and downstream portion;

a plurality of individual tubular members randomly disposed solely in said passageway in said upstream portion in the path of flow of the liquids and providing impingement contact surfaces for bringing the various constituents of the liquids into intimate contact with each other, while permitting the free flow of liquids through said passageway in said downstream portion;

a jacket enclosing said coil and having an inlet and an outlet for the flow of temperature controlled fluid through said jacket and around both said upstream and said downstream portions of said coil;

separating means coupled to said outlet of said coil for separating one immiscible liquid from another including a vertical chamber having an inlet coupled to said outlet of said coil, a first outlet below said inlet for the flow from said chamber of the separated denser liquid, a second outlet above said first outlet and below said inlet for the flow from said chamber of the separated less dense liquid; and

pump means coupled to said inlet of said coil for supplying thereto at a given rate the two immiscible liquids, and to said first and second outlets of said chamber for removing therefrom at given rates the two separated immiscible liquids.

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