

[54] **MIXER-SEPARATOR FOR AUTOMATED ANALYTICAL CHEMISTRY SYSTEM**

[75] Inventor: **Ian E. Bush**, Plainview, L. I., N.Y.

[73] Assignee: **The United States of America as represented by the Secretary, Department of Health, Education and Welfare, Washington, D.C.**

[22] Filed: **Jan. 5, 1972**

[21] Appl. No.: **215,482**

[52] U.S. Cl. **233/20 R, 233/22, 233/46**

[51] Int. Cl. **B04b 1/14, B04b 11/06**

[58] Field of Search **233/19 R, 19 A, 20 R, 233/20 A, 47 R, 21, 22, 46**

[56] **References Cited**
UNITED STATES PATENTS

2,615,320	10/1952	Belaieff	233/46 R
2,578,468	12/1951	Fleischer	233/20 R

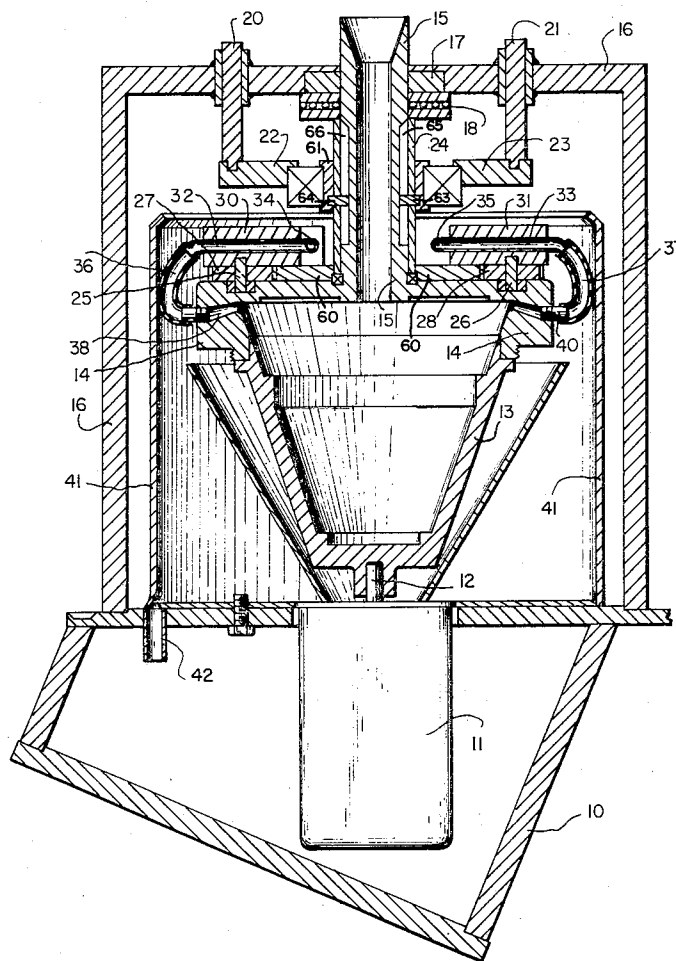
2,538,529	1/1951	Komline	233/20 R
2,200,202	5/1940	Harvey	233/20 R
972,030	10/1910	Smith	233/20 R
3,333,765	8/1967	Maurer	233/22

Primary Examiner—George H. Krizmanich
Attorney—Alvin Browdy

[57] **ABSTRACT**

A mixer-separator unit for use in automated analytical chemistry systems wherein two liquid phases are brought into intimate contact and then separated. The two liquids are put into a vessel where slow rotation stops them around in the vessel to secure intimate mixing. Fast rotation is begun after a preset interval which results in fast and efficient centrifugal separation of the phases. After another interval tap jets are opened to withdraw the denser phase and then the lighter phase.

13 Claims, 5 Drawing Figures



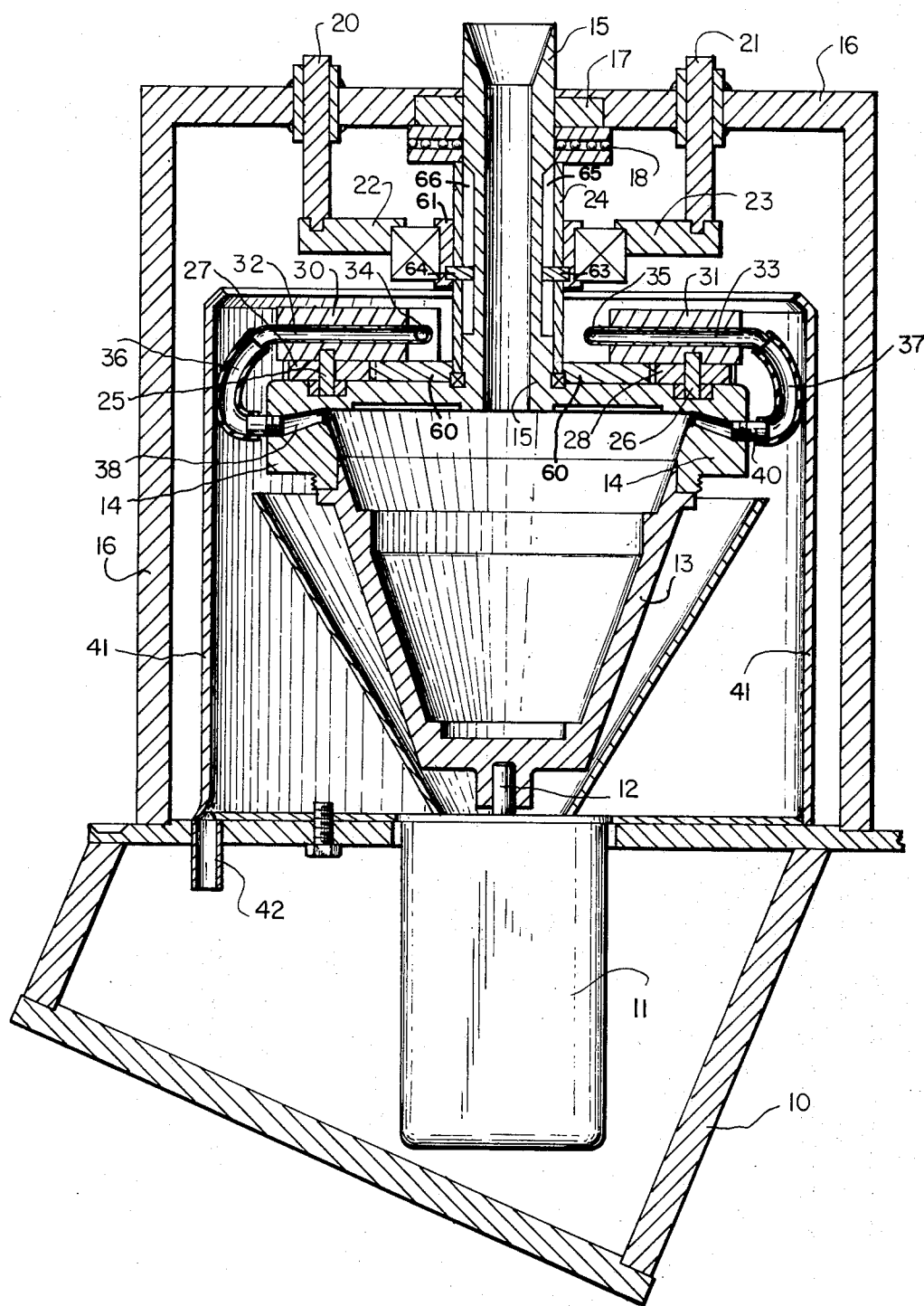


FIG. 1

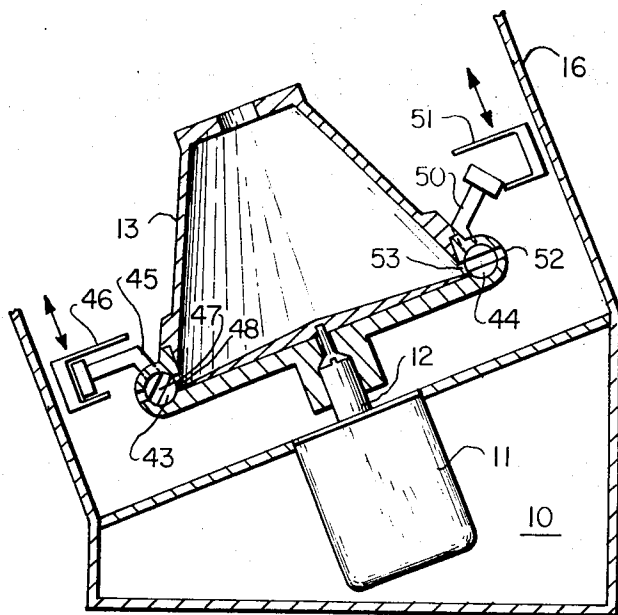


FIG. 2

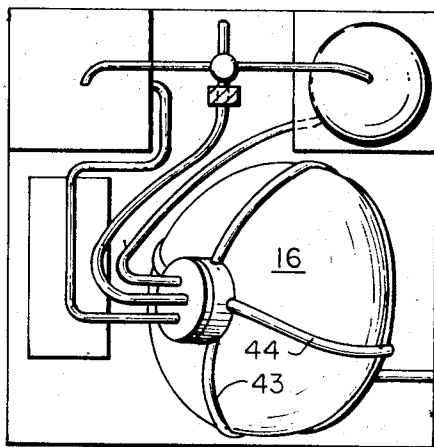


FIG. 4

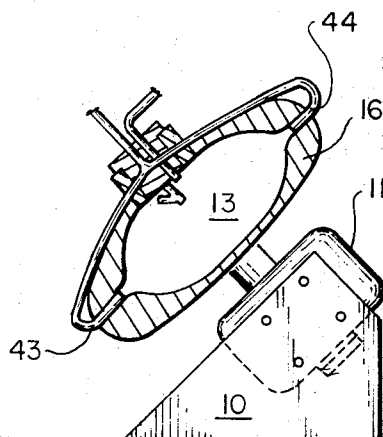


FIG. 3

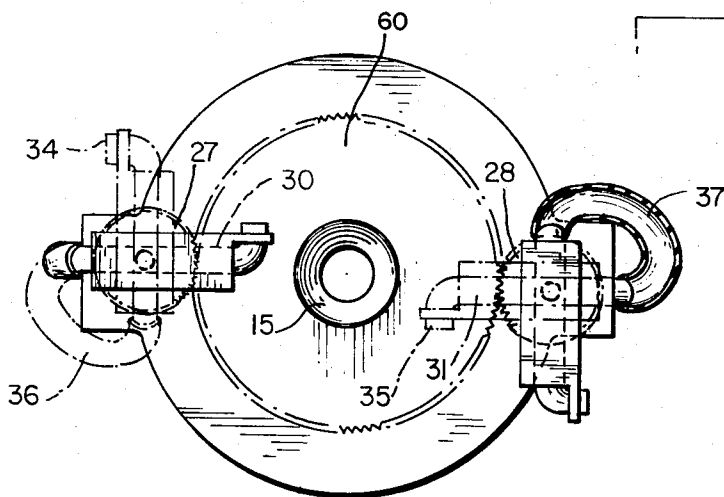


FIG. 5

MIXER-SEPARATOR FOR AUTOMATED ANALYTICAL CHEMISTRY SYSTEM

The present invention relates to liquid mixer-separators and, more particularly, to liquid mixer-separators which bring two liquid phases into intimate contact and then separates them.

An analytical procedure in chemistry consists of a series of operations carried out in a fixed sequence which may be considered as steps or stages. At first sight the variety of such procedures suggests that their generalization would be difficult. Several existing systems are based on a series of units arranged in a linear series connected by tubing for transport of samples and reagents, and activated by time delay programming switches of the type common in domestic washing machines. They depend on the fact that many simple analytical procedures, especially in clinical chemistry, involve a simple and standardized sequence of operations. Different procedures can be obtained merely by varying the reagents mixed with the sample; the time sequence of operations remaining the same.

The disadvantages of such types of apparatus are numerous, the main ones being as follows: First, each set (or channel) of the equipment has to be set aside for one procedure per batch of samples. In order to carry out several procedures in parallel it is necessary to purchase and run many channels, involving large capital outlays and much waste of investment when channels are out of use or being converted to a new procedure. Second, they are unable to carry out efficiently certain operations which are common in analytical procedures for trace-substances: e.g. liquid-liquid extraction and washing, evaporation of bulky extracts, filtering, and chromatographic extraction or fractionation.

The basic idea of the new systems, of which the present invention is one of the components, is that all analytical procedures can be boiled down to sets of stages whose logical structure is uniform. This uniformity of each stage immediately suggests two features of a generalized apparatus for automated analytical chemistry;

A. The information content of an analytical procedure is relatively small and amenable to a very simple type of computer programming.

B. The means by which samples and reagents are transported through the system is the most important feature of the system. Thus, all stages of an analytical procedure consist effectively of two standard sub-stages.

1. Bring sample and reagents into a "vessel" or "operant unit" designed to carry out a certain operation;

2. Carry out unit operation.

The sample may be the original sample in the usual sense or a derived fraction of it resulting from previous stages of the procedure. To emphasize this it will be called the "reactant" or "reactant phase" in the following description. The important thing is that most of the information content of sub-stage (2) is contained in the mechanical properties of the apparatus or unit used for each stage. The transportation sub-stage (1) can be defined simply by the *types of units* involved—namely the next operant unit in the sequence and the types of reagent supply units to be marshalled into the operation.

The number of different types of units is not too large and can be worked out in terms of the phases involved in each operation. At the boundaries of the system a large number of physical measuring instruments can be

attached but usually only one or a few of each type are needed.

The system arising from this analysis is essentially a *matrix* of about 10 types of operant units connected by a transportation system capable of passing reactants and reagents rapidly between any and all pairs of units in the matrix.

With the above background information relative to the problems and proposals of automated analytical chemistry systems in mind, it is obvious that there exists a great need for efficient and reliable components to carry out these systems. One such essential component, and perhaps the most important, is a mixer-separator unit which forms the subject matter of the present invention; this mixer-separator automatically functions to bring together two liquids, to mix them together in intimate contact, and then to automatically separate them.

The device of the present invention can be controlled by computer, and offers a great improvement over comparable units which are expensive, clumsy, and very slow in performance. The present invention through the expedient of slow rotation slops the two liquids around in a vessel until they are intimately mixed, then a fast rotation is begun which results in separation of the liquids by centrifugal action. At a proper interval, suitable taps or valves are opened to permit the liquids to flow out of the vessel.

It is accordingly, an object of the present invention to overcome the deficiencies of the prior art, such as indicated above.

Another object of the present invention is the provision of an efficient mixer-separator for liquids.

Another object of the present invention is the provision of a mixer-separator which may be controlled by a computer.

Yet another object of the invention is the provision of a mixer-separator which may be used in an automated analytical chemistry system.

Still another object of the invention is the provision of a mixer-separator which permits individual withdrawal of the liquids after they have been separated.

Other objects and many of the attendant advantages of the present invention will be readily appreciated as the same becomes better understood by reference to the following detailed description when considered in connection with the accompanying drawings in which like reference numerals designate like parts throughout the figures thereof and wherein:

FIG. 1 is a sectional view of a preferred embodiment of the invention;

FIG. 2 shows a modification of the invention;

FIG. 3 shows another modification of the invention;

FIG. 4 shows another view of the modification of FIG. 3; and

FIG. 5 shows a method of opening and closing the mixer-separator.

Referring now to the drawings there is shown in FIG. 1, which represents a preferred embodiment of the invention, a cross-sectional view of the mixer-separator. There is provided a base 10 of substantially triangular configuration, one leg of which serves as a foundation and the hypotenuse of which forms a plane about 30°-45° with the horizontal. Securely fastened to the hypotenuse side of the base is an electric motor 11 having a drive shaft 12 extending outwardly from the base. A mixing vessel substantially in the shape of a trun-

cated cone is attached to motor drive shaft 12 at it's smaller end so that the entire vessel is rotated by the motor 11.

Positioned across the larger outer end of vessel 13 is a lid 14 which is securely fastened to the vessel 13, the center of the lid having a tubular shaped portion 15 connecting with the inner area of the vessel and comprising an input channel for the admittance of fluids to such vessel 13. Over the entire mixing vessel 13 and its connections is a cover 16 which joins the base 10, the cover 16 being of relatively heavy gage material as a safety feature for protection of personnel when the device is being spun particularly at high speed by motor 11. The outer end of the tubular channel 15 extends through the cover 16 and has its edges beveled to assist in the admittance of fluid to the vessel. A collar 17 forms a part of lid 16, and with its associated ball bearings 18 provides a rotating support for the outer end of the tubular channel 15.

Also extending through the lid 16 are two dowel pins 20 and 21, having L-shaped arms 22 and 23 which contact a sleeve 24 enclosing the upper part of channel 15. The free ends of arms 22 and 23 work in two vertical grooves and a partial spiral slot (not shown) in sleeve 24 as will be more fully described hereinafter. Pivotaly mounted on either side of the channel 15, by means of pins 25 and 26, are two small gears 27 and 28, these gears 27 and 28 serving as means for turning two movable arms 30 and 31. Through the center of each of the movable arms 30 and 31 there is provided, respectively, a channel 32 and 33 ending in tap-jets 34 and 35, while connected at the other ends of such channels are two short pieces of flexible tubing 36 and 37. Tubing 36 is fastened to a connection 38 through the lid 14 and into vessel 13 while tubing 37 is fastened to a connection 40. A circular splash board 41 is located near vessel 13 and a drain plug 42 connects the top half of the mixer-separator with the hollow area of the base 10.

In FIG. 2, there is shown another-embodiment of the invention. Here, as in FIG. 1, there is a triangular shaped base 10, with the hypotenuse at a slight angle to the horizontal, and a motor 11 integrally fastened to this hypotenuse. It is obvious, however, that in this embodiment the truncated, cone-shaped, mixing vessel 13 has been reversed so that now the larger end is downward and fastened to the drive motor 11. The smaller end does not require a lid, but there is provided a port for the admission of fluids to be mixed by the unit. The tap-jets 43 and 44 have a slightly different configuration in that they take the form of two-position rotary valves. In FIG. 2 tap-jet 43 is shown in the off position while tap-jet 44 is in the on, or open, position to permit withdrawal of fluid from the vessel. To operate tap-jet 43 there is provided a valve arm 45 which is activated by a U-shaped bracket 46. When the bracket 46 is moved up and down (by means either manual or automatic, not shown) the outer end of valve arm 45 moves in the bracket to rotate the circular valve part of the tap-jet and thereby align a channel 47 through the valve with a drain hole 48 in the side of vessel 13, or to block the drain hole as the case may be. By like analysis, tap-jet 44 has a valve arm 50 which moves within a bracket 51 to align or block channel 52 with drain hole 53.

In the embodiment shown in FIGS. 3 and 4, the mixer-separator has it's mixing vessel 13 in the shape of a

flattened oval rather than a truncated cone, and the tap-jets for withdrawing fluid are connected to the sides of the oval, and extend around the sides and over the back to meet with drain pipes external to the mixer itself.

Turning now to FIG. 5 there is shown a top view of the mechanism for withdrawing fluid from the embodiment of FIG. 1., with the tap-jets in both the open and closed positions. In place of the taps 43 and 44 of FIG. 2, flexible chemically inert tubes 36 and 37 are connected to a plain outlet at each end of one of the diameters of the rotor and these pass upward and then inwardly through small movable arms 30 and 31, ending in a bend which provides short outlet jets 34 and 35. The position of arms 30 and 31 is controlled either by gearing, such as the gears 27 and 28 in the illustrated embodiment, or by a variety of conventional cams or connecting rods. The central gear 60, cam, or connecting rod rotor is mounted on a sleeve 24 enclosing the upper shaft of the main centrifugal rotor. Rotary movement of this gear 60, etc. is provided by a further sleeve 61 containing two diametrically opposed dowel pins 20, 63 and 64 which work in two vertical grooves 65 and 66 on the main shaft of the rotating tubular channel 15 and a partial spiral slot [not shown] in the sleeve 24. These dowel pins 63 and 64 are mounted in sleeve 6 in contact with a thrust bearing so that movements of this bearing in the direction of the axis of the main rotor can be achieved during high speed rotation of the latter. The vertical movement of dowel pins 20 and 21 causes the corresponding vertical movement of sleeve 61 and pins 64 and 65 which work in the vertical grooves 65 and 66 of the tubular channel 15 and the spiral slot in sleeve 24. The vertical movement of pins 64 and 65 in the spiral slot, therefore, rotate the sleeve 24 and hence the central gear 60 with respect to the rotor and in turn moving rocker arms 30 and 31 from the closed position to the open position. In the closed position, the outlets of the tubes are within the circumference of the inner cylindrical surface of the body of liquid in the position it takes under the centrifugal force of high speed rotations. Under these circumstances, no ejection of fluid takes place. In the open position, the outlets are at the circumference of the main rotor so that ejection takes place into a collector ring similar to the previous modifications positioned at the appropriate height.

As to the operation of the device the two liquids which are to be mixed are inserted in the mixing vessel 13 through the tubular opening 15, and the motor 11 is turned on at slow speed. This slow rotation slops the two liquids around in the vessel to secure intimate mixing. After a preset interval fast rotation of motor 11 is begun which results in rapid and efficient centrifugal separation of the phases. After a safe interval the two tap-jets 34 and 35 are deflected to the open position by means of gearing 27 and 28 (or valves 43 and 44 are opened by the movement of brackets 46 and 51 in FIG. 2). Since the heavier liquid making up the denser phase will be ejected first, when the tap-jets are opened this denser phase is ejected through jets 34 and 35 and against splash board 41 to be withdrawn by drain plug 42. The interface with the lighter phase is sensed by an electronic transducer (not shown) which, via appropriate circuitry, again opens the tap-jets, and this time the lighter liquid is ejected. The version shown in FIG. 3 has a conventional take-off head using tubes and a spe-

cially machined grooved head as in a continuous flow centrifuge or cream-separator.

From the above description of the structure and operation of the present invention it is obvious that the device offers many improvements over the weaknesses and shortcomings of prior mixer-separators. The invention provides for the thorough and intimate mixing of two liquids and then the fast and efficient centrifugal separation of the dense and lighter phases. The device may be readily used in an automated analytical chemistry system, and is adaptable for control by a computer.

Obviously many modifications and variations of the present invention are possible in the light of the above teachings. It is therefore to be understood, that within the scope of the appended claims, the invention may be practiced otherwise than as specifically described.

I claim:

1. A mixer-separator including
 - a base, said base having a side at some angle with the horizontal;
 - rotation means mounted on the side, said rotation means having an axis of rotation;
 - a mixing vessel attached to the rotation means and rotatable about the axis of rotation thereby, said mixing vessel including fluid holding means;
 - valveless ejection means for separating one liquid from another liquid, said means being carried by said mixing vessel and communicating with the fluid holding means for controlling the retention of liquid therein, when said ejection means is in a first position, and the withdrawal of liquid therefrom, when said ejection means is in a second position, said ejection means including an outlet means, the outlet means being a first axial distance from the axis of rotation when said ejection means is in the first position, the first axial distance being less than the axial distance from the axis of rotation to the circumference of the inner cylindrical surface of the liquid in the position it assumes during centrifugal separation, and a second axial distance from the axis of rotation when said ejection means is in the second position, the second axial distance being

greater than the axial distance from the axis of rotation to the circumferences of the inner and outer cylindrical surface of the liquid in the position it takes during centrifugal separation; and

collection means for collecting the ejected liquid.

2. The device of claim 1 wherein the rotating means is a motor which runs at more than one speed.

3. The device of claim 2 wherein the mixing vessel is essentially in the shape of a truncated cone with its small end attached to the rotating means.

4. The device of claim 3 wherein said fluid holding means is an interior chamber of said mixing vessel, said interior chamber being of a shape substantially corresponding to the shape of said mixing vessel.

5. The device of claim 2 wherein the mixing vessel is essentially in the shape of a truncated cone with its larger end attached to the rotating means.

6. The device of claim 5 wherein said fluid holding means is an interior chamber of said mixing vessel, said interior chamber being of a shape substantially corresponding to the shape of said mixing vessel.

7. The device of claim 1 wherein said valveless ejection means includes a flexible tube connecting the fluid holding means with the outlet means.

8. The device of claim 1 further including means for moving said valveless ejection means between the first and second positions.

9. The device of claim 1 wherein said valveless ejection means is rotatably connected to said mixing vessel.

10. The device of claim 9 further including means for rotating said valveless ejection means between the first and second positions.

11. The device of claim 10 wherein said means for rotating said valveless ejection means includes gears.

12. The device of claim 1 wherein said outlet means is a tap-jet.

13. The device of claim 1 wherein said collection means includes a splash board, said splash board substantially enclosing said mixing vessel and said valveless ejection means.

* * * * *

45

50

55

60

65