

[54] **REAGENT TUBE AND CENTRIFUGALLY OPERATED SOLID-LIQUID SEPARATING DEVICE**

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[51] Int. Cl. **B01d 33/00**

[58] Field of Search 210/83, 84, 97, 117, 136, 210/339, 359, 361, 362, 371, 419, 429, DIG. 23; 233/26

[56] **References Cited**

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[57]

ABSTRACT

A reagent receptacle adapted for mounting in a centrifuge and having a discharge opening positioned for centrifugal discharge of matter therefrom; a filter mounted in the receptacle for filtering the matter discharged; and a normally closed centrifugal force responsive valve controlling flow through the opening.

13 Claims, 5 Drawing Figures

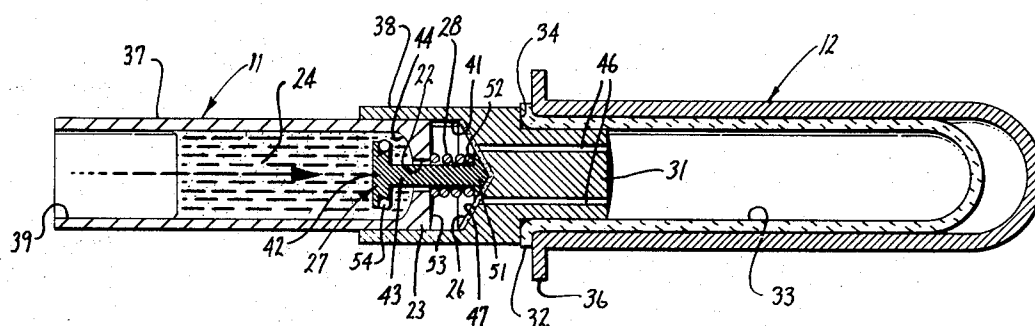


Fig. 1

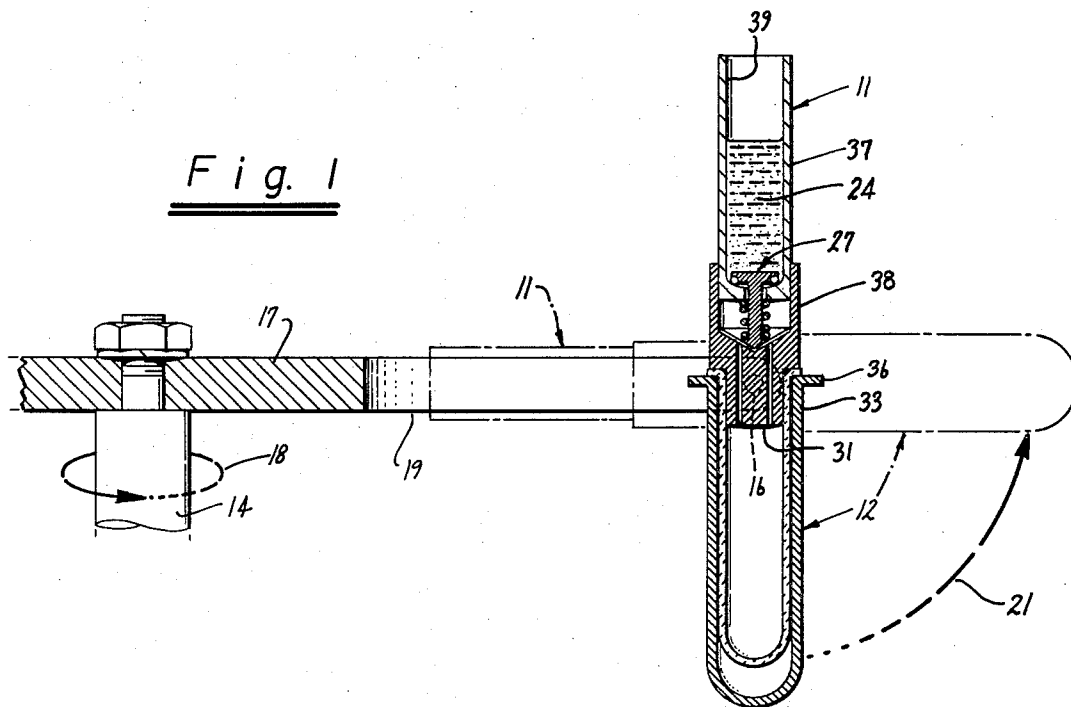


Fig. 2

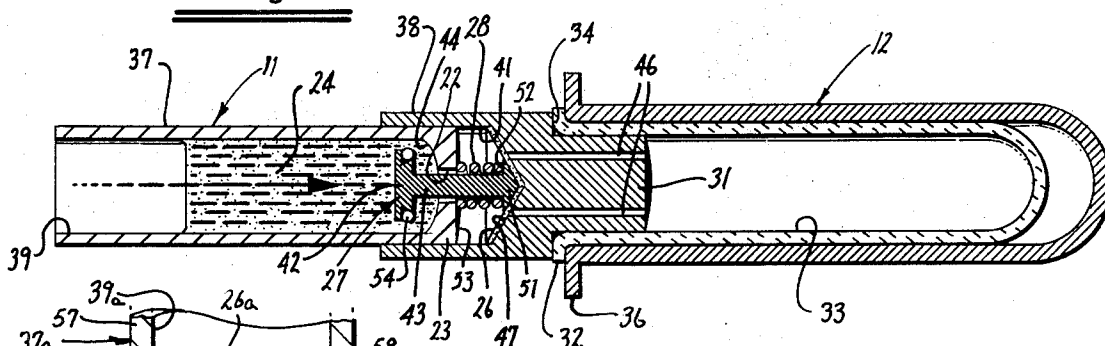
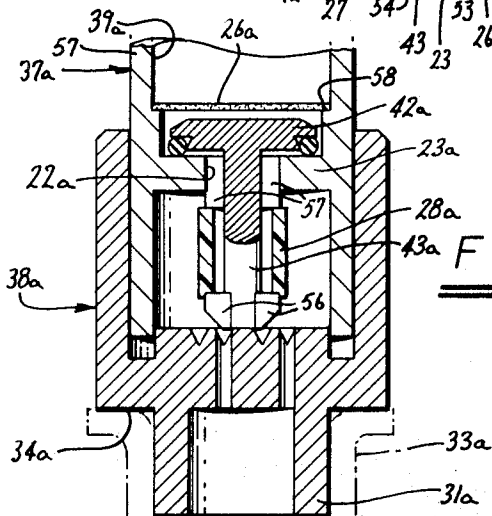


Fig. 3



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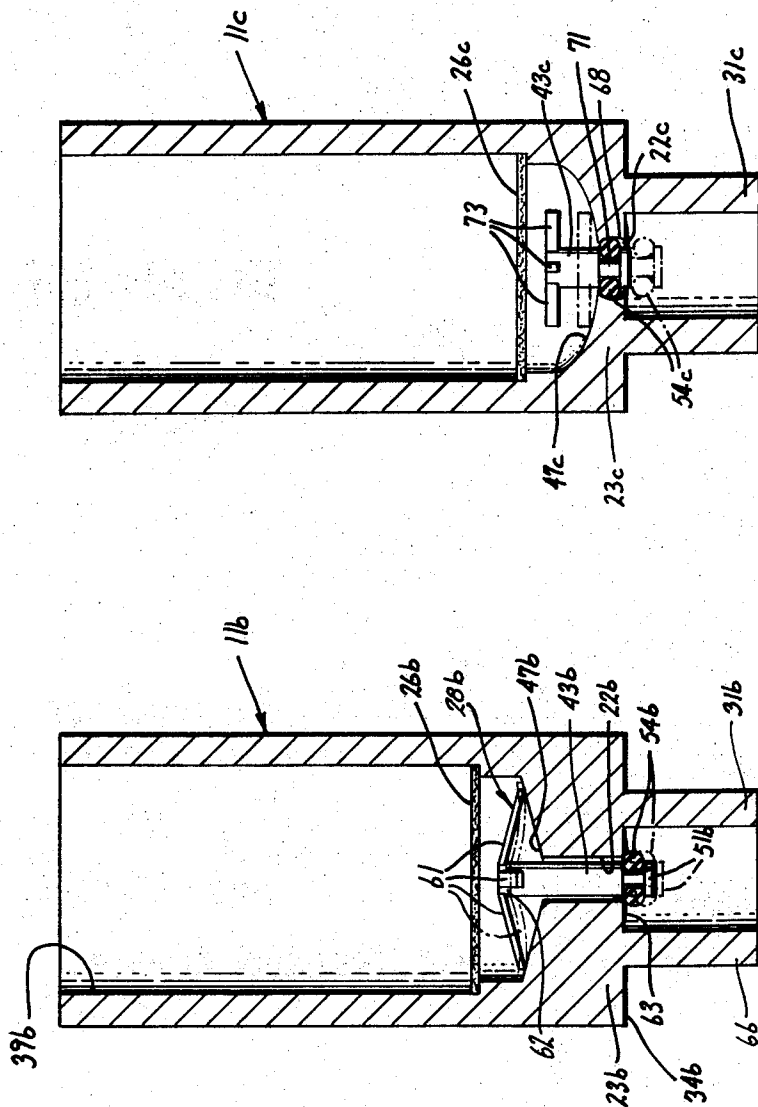


Fig. 5

Fig. 4

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REAGENT TUBE AND CENTRIFUGALLY OPERATED SOLID-LIQUID SEPARATING DEVICE

The invention relates to devices used for separation of solids and liquids by centrifugation, see U.S. Pat. Nos. 2,665,008, 2,854,143 and 3,300,051.

In many procedures used in chemical and medical laboratories, various ingredients and/or reagents are required to be mixed together or reacted and subsequently separated, as for example by passing through a filter. Centrifugal filtration is greatly to be desired in those instances where rapid filtration is needed to shorten the length of the procedure and expedite the return of information to a doctor or other person waiting thereon. Transfer of material from a reagent receptacle to a centrifugal extractor is not always possible or feasible, may be awkward or messy, and may introduce contamination.

An object of the present invention is to provide a combined reagent tube and centrifugally operated solid-liquid separating device in which the various ingredients and reagents may be placed in a reagent receptacle for interaction or reaction for as long a period as desired, and then without opening the container or requiring transfer of the contents thereof, the container itself is placed in a centrifuge for rapid and efficient separation of the solid and liquid matter, thus enabling completion of the procedure and return of the results thereof in the shortest possible time.

Another object of the present invention is to provide a low-cost, disposable device of the character described which may be manufactured by low-cost, mass production techniques and furnished in sterile condition to laboratories for their convenient, one-time use.

The invention possesses other objects and features of advantage, some of which of the foregoing will be set forth in the following description of the preferred form of the invention which is illustrated in the drawings accompanying and forming part of this specification. It is to be understood, however, that variations in the showing made by the said drawings and description may be adopted within the scope of the invention as set forth in the Claims.

REFERRING TO SAID DRAWINGS:

FIG. 1 is a cross sectional view of a reagent tube and centrifugally operated solid-liquid separating device constructed in accordance with the present invention;

FIG. 2 is a cross sectional view of an enlarged scale of the device illustrated in FIG. 1 and shown in its centrifugally oriented position;

FIG. 3 is a cross sectional view of a modified form of the device;

FIG. 4 is a cross sectional view of a further modified form of the device; and

FIG. 5 is a cross sectional view of another modified form of the device.

The combination reagent tube and centrifugally operated solid-liquid separating device of the present invention comprises briefly a reagent tube or receptacle 11 for receipt of the various ingredients to be operated upon and which is adapted for mounting in the hinged receiving cup 12 of a centrifuge for whirling in an orbit around the central drive shaft 14 of the centrifuge and for swinging with cup 12 around a hinge pin 16 from a

normally upright position, as shown in full line in FIG. 1, to a horizontal position in the plane of rotation of the centrifuge, as shown in phantom line in FIG. 1 and in full line in FIG. 2. Standard laboratory centrifuges will comprise a central hub mounted for rotation with shaft 14, as depicted by arrow 18; a plurality of arms 19 connected to hub 17 in circumferentially spaced relation and radiating therefrom; and a plurality of test tube receiving cups 12 usually demountably pivoted to arms 19 along horizontal axes provided by pins 16. Pivot pins 16 are located adjacent the normally upper ends of cups 12 so that the latter will depend vertically from pins 16 in the stationary condition of the centrifuge. Upon rotation of the centrifuge, cups 12 will swing outwardly due to centrifugal force as suggested by arrow 21. The balance of the operating structure of the solid-liquid separating device includes a discharge opening 22 in the normally lower end 23 of receptacle 11 in position for centrifugal discharge of matter 24 in the receptacle when the latter is horizontally oriented, as illustrated in FIG. 2; a filter 26 mounted in the receptacle for filtering of matter discharged through opening 22; and a normally closed centrifugal force responsive valve 27 controlling flow through opening 22 and having a minimum required centrifugal opening force. The latter function is here provided by spring means 28 for retaining the valve in closed position and permitting opening of the valve above a predetermined value of centrifugal force. As will be observed, the centrifugal force not only functions to open valve 27 but also to force the liquid component in matter 24 through filter 26. The normally lower end 31 of the receptacle is formed for receiving the open top 32 of a test tube 33 which is in turn dimensioned for fitting within the mounting cup 12 of the centrifuge for supporting the apparatus. As here shown, the lower end portion 31 of the receptacle is of reduced cylindrical form so as to fit within the open top 32 of test tube 33 and to provide an annular shoulder 34 for supporting test tube end 32 and also forming a support on the upper end 36 of cup 12.

In the forms of the invention illustrated in FIGS. 1 and 2, and FIG. 3, receptacle 11 is made up of a pair of telescopically mounted, relatively movable sections 37 and 38. Section 37 is formed with a reagent chamber 39 for matter 24 and with end wall 23 and discharge opening 22 therein. Section 38 is formed with an end chamber 41 telescopically receiving the lower end 23 of section 37 and is formed with the reduced cylindrical end 31 for mounting in test tube 33 and supporting cup 12. Valve 27 here includes a head 42 mounted interiorly of chamber 39 and is connected to a valve stem 43 mounted for reciprocation through opening 22 for movement of head 42 to and from a valve seat 44 provided by the interior surface of end wall 23 surrounding opening 22. Section 38 is formed with a plurality of longitudinally extending passages 46 extending from the bottom wall 47 of chamber 31 to the lower end 31 of the section for conducting liquid into test tube 33. The lower end 51 of valve stem 43 is here supported on wall 47 and is enlarged to provide a shoulder or spring rest 52 for one end of helical spring 28, the opposite end of the spring being mounted in compression against the exterior surface 53 of end wall 23. A light metal spring is all that is required to retain the valve in normally closed position. Preferably, and

as here shown, valve head 42 is fitted with an O-ring 54 for effecting its sealed engagement against seat 44.

In the form of the invention illustrated in FIGS. 1 and 2, filter 26 is mounted directly against the bottom wall 47 of section 38 and may consist of a disk of filter paper or the like. Wall 47 is preferably beveled to a cone shape, as illustrated in FIGS. 1 and 2, to provide a central support for stem end 51 which will bear directly upon filter 26, and passages 46 are spaced radially from stem end 51 to prevent blocking of these passages by the stem.

A description of the operation of the device of FIGS. 1 and 2 follows. The ingredients to be mixed and/or reacted are placed in chamber 39 and stirred, agitated, shaken, heated or otherwise treated as required and held in the chamber for the prescribed period of time. With valve 27 closed, the receptacle 11 may be treated and handled as any other type of reagent tube. Of course, the present device has its particular advantage in those instances where solid and liquid materials placed or formed in chamber 39 are to be separated with either or both of the solid-liquid materials recovered as a sought end product or for further processing.

Upon completion of reaction of the materials in chamber 39, the device is mounted in the centrifuge as above described and the latter energized to place the device in rotating orbit. The initial centrifugal force acting upon the contents of the container and the mass of section 37 will be supported by spring 28 maintaining the valve 27 in closed position. At a predetermined minimum centrifugal force which will correspond with a desired speed of rotation of the centrifuge, generally from about 400 RPM to 1000 RPM, the operating centrifugal force will overcome the resistance of spring 28 causing section 37 to move into section 38 and opening of valve 27. The contents of chamber 37 will then move due to centrifugal force through opening 22 and onto filter 26. The liquid phase will be centrifugally compressed through filter 26 and proceed via passages 46 into test tube 33. The solid matter will be retained on the filter and may, where desired, be recovered therefrom.

A modified form of the invention is illustrated in FIG. 3 wherein the helical metal spring is replaced by a length 28a of lightweight, flexibly resilient, plastic material such as polyvinyl chloride. Such material can be used safely with most laboratory chemicals, acids, alkalis, alcohols, oils and solvents, and may be readily chemically sterilized. A relatively lightweight, thin wall, resiliently flexible tubing is preferred and is available from several commercial sources, as for example Van Waters and Rogers, and Gradko Glass Laboratories, Inc. Tube 28a is here mounted around valve stem 43a with its opposite ends supported on ribs 56 on the lower end of the stem and ribs 57 on end wall 23a surrounding discharge opening 22a, ribs 56 and 57 providing spaced rests for holding tube 28a in compression thereby maintaining valve head 42a on its seat. Under centrifugal force, tube 28a will bow out to foreshorten and permit opening of the valve.

Another feature of the form of the invention illustrated in FIG. 3 is the disposition of filter 26a for easy removal from the device. As here shown, the filter is mounted in chamber 39a in spaced opposed relation to head 42a and for engagement by the head and dislodgment of the filter upon axial displacement of valve stem

43a. Preferably, the wall 57 of section 37a is formed with an annular shoulder 58 providing a mounting for filter 26a. The latter may comprise a plastic or metal screen with a sheet of filter paper or the like mounted thereon. Upon dislodgment of the filter 26a by valve head 42a, the filter may be readily removed with forceps for transfer to a slide, test tube or other procedure. The filter should be well fitted about its periphery in chamber 39a so as to prevent leakage of liquid around the edges of the filter and, accordingly, the filter may become jammed in the chamber during the course of centrifuging the material through the filter. The spacing between filter 26a and valve head 42a is such that the latter will remain spaced from the filter in its normal opening action during centrifuging of the material.

The lower end 31a of section 38a is formed as a depending circular skirt for telescopic insertion into test tube 33a and for providing shoulder 34a for supporting the device in the centrifuge.

A further modified form of the invention is illustrated in FIG. 4 wherein the valve spring 28b comprises a plurality of resilient arms 61 connected to the upper end 62 of valve stem 43b and extend radially from the stem for support on lands provided by the bottom wall 47b of reagent chamber 39b of receptacle 11b. The lower end 51b is here fitted with an O-ring 54b for engagement with the exterior surface 63 of end wall 23b surrounding discharge opening 22b. Normally, arms 61 will hold valve stem 43b upwardly, as seen in FIG. 4, thereby holding valve 54b sealed against seat 63. Under centrifugal force, however, arms 61 will bow downwardly, as seen in phantom line in FIG. 4, thereby displacing valve 54b from its seat to open position.

In this form of the invention, FIG. 4, receptacle 11b is of a one-piece construction, as contrasted to the two-section construction of the forms in FIGS. 1-3, and accordingly the operating mass subject to centrifugal force is that of the valve itself. This will suffice for most applications and provides a simplified, lower-cost construction. Where stronger sealing force is required, the greater relatively moving mass provided in the two-sectional construction may be used. In this form of the invention, FIG. 4, the lower end 31b of the one-piece receptacle 11b is formed for insertion into the open end of a test tube and here comprises an annular depending skirt 66 dimensioned for telescopic insertion into the test tube and defining the supporting shoulder 34b functioning to support the device in a centrifuge as the corresponding shoulder in the first described embodiment. Filter 26b is preferably mounted over the interior end 62 of the valve stem in a manner similar to that shown in FIG. 3 and may be dislodged by removing O-ring 54b and pushing valve stem 43b upwardly through opening 22b.

Another form of the invention is illustrated in FIG. 5 wherein the resilient means for retaining the valve in closed position is incorporated in the valve and its seat by forming these parts for resilient retention in closed position and for centrifugal displacement to open position. As here shown, the wall 68 of discharge opening 22c in end wall 23c of receptacle 11c is formed with an internal concave surface not only forming the discharge opening but also a seat for an O-ring 54c mounted on valve stem 43c. O-ring 54c is formed of resiliently compressible material, such as rubber, and with an external convex surface compressively engag-

ing the interior concave wall 68 in the closed position of the valve. The application of a predetermined amount of centrifugal force applied longitudinally of stem 43c will cause valve 54c to pop out of its seat to an open position, as indicated in phantom line in FIG. 5. The mass of the valve provides such a force on rotating the device at or above a predetermined speed in the centrifuge. Stem 43c is here provided with an annular socket 71 at its lower end for retaining of O-ring 54c in position on the stem for sealing the clearance provided between stem 43c and opening 22c. This same construction is used in the embodiment in FIG. 4. One or more laterally extending arms 73 is connected to the upper end of stem 43c for engagement with the bottom wall 47c of the chamber for retaining the valve assembly in the discharge opening when the valve is in open position. Filter 26c is mounted over the top of the valve stem for dislodgment by removal of O-ring 54c and upward displacement of the valve stem, as in the case of the embodiment shown in FIG. 4. Also, the lower end 31c of the receptacle is formed with the depending skirt and shoulder for mounting in a test tube and in the centrifuge, as also shown in the forms of the invention of FIGS. 3 and 4.

I claim:

1. A centrifugal solid-liquid separation apparatus comprising:
 - a receptacle having a chamber for receipt of reagents to be mixed therein and being adapted for mounting in a centrifuge for orbiting therewith and having said receptacle being formed with a discharge opening for said chamber positioned at the radially outer portion of said receptacle in said orbiting position;
 - a filter carried by said receptacle and mounted for filtering the discharge through said opening;
 - a valve carried by said receptacle and having a normally seated position sealing said chamber from said opening and an unseated position communicating said chamber and opening; and
 - said valve being formed and mounted for responding to a predetermined magnitude of centrifugal force developed by said orbiting for displacement to an unseated position, thereby centrifugally driving the contents of said chamber through said filter and discharge opening.
2. An apparatus as defined in claim 1 said receptacle providing a seat for said valve, said valve and seat being formed for resilient retention of said valve in seated position and for centrifugal displacement of said valve from said seat to unseated position.
3. An apparatus as defined in claim 2, said receptacle having an annular wall with an internal concave surface forming said discharge opening and said seat; and said valve comprising a ring of resiliently compressible material having an external convex surface compressibly engaging said concave surface in the seated position of said valve.
4. An apparatus as defined in claim 3, said valve comprising a stem mounted for axial reciprocation in said

opening and for centrifugal displacement therethrough, said stem being formed with an annular socket demountably receiving said ring.

5. An apparatus as defined in claim 1 and means for supporting said receptacle when placed in said centrifuge for relative displacement of said receptacle and means as a function of centrifugal force and being connected to said valve for movement thereof to unseated position by and upon said relative displacement.
6. An apparatus as defined in claim 5 said receptacle being of elongated form and having said discharge opening in one end thereof; said means comprising a section telescopically receiving said receptacle for radial outward displacement therein in said orbiting position; spring means mounted to resist said outward displacement of said receptacle; and said valve having a stem engaging said section upon said outward displacement to displace said valve to unseated position.
7. An apparatus as defined in claim 6, a liquid receiver demountably secured to said section; and said section being formed with a passage conducting liquid from said discharge opening to said receiver.
8. An apparatus as defined in claim 7 said valve having a head mounted in said chamber and said stem extending through said discharge opening to engage said section; said stem and receptacle providing spaced spring rests; and said spring means being mounted in compression between said rests for retaining said valve in normally seated position.
9. An apparatus as defined in claim 8, said spring means being of helical form surrounding said stem portion.
10. An apparatus as defined in claim 8, said spring means comprising a tube of flexibly resilient plastic material surrounding said stem portion.
11. An apparatus as defined in claim 1 said filter having a mounting in said receptacle in spaced opposed relation to said discharge opening; and said valve being mounted for manual displacement and for engagement with said filter upon said manual displacement for dislodging said filter from said mounting.
12. An apparatus as defined in claim 1 spring means biasing said valve to seated position and comprising a plurality of resilient arms connected to said valve and supported by said receptacle.
13. An apparatus as defined in claim 12 said valve having a stem mounted through said discharge opening; said arms extending radially from said stem; and said receptacle being formed with lands supporting the outer ends of said arms.

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