Sept. 8, 1959 J. BUCHLER 2,903,342

DEVICE FOR FRACTIONATED EXTRACTION USING MORE THAN TWO SOLVENTS

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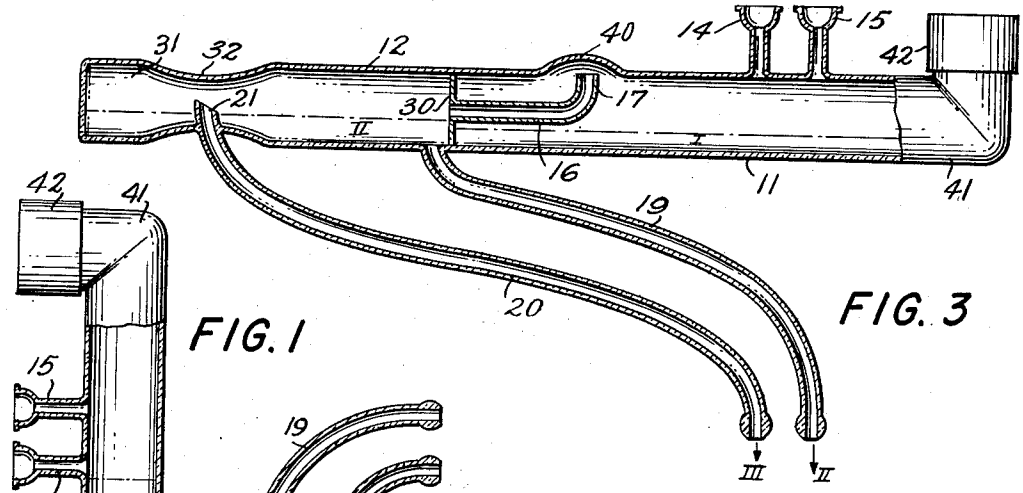


FIG. 3

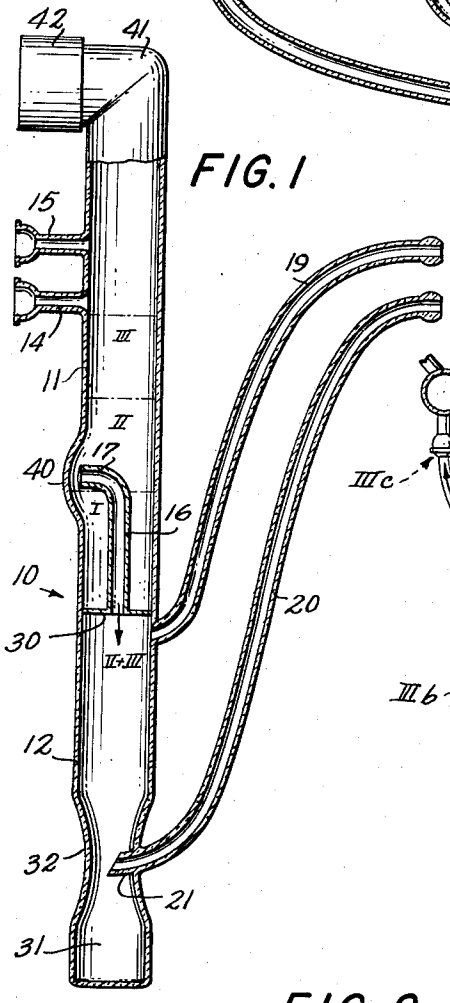


FIG. 1

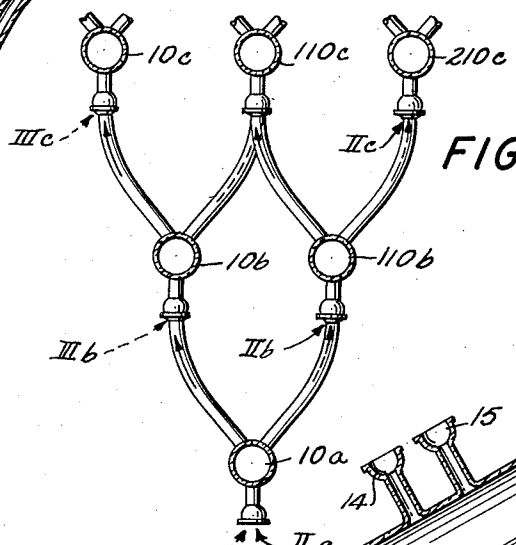


FIG. 4

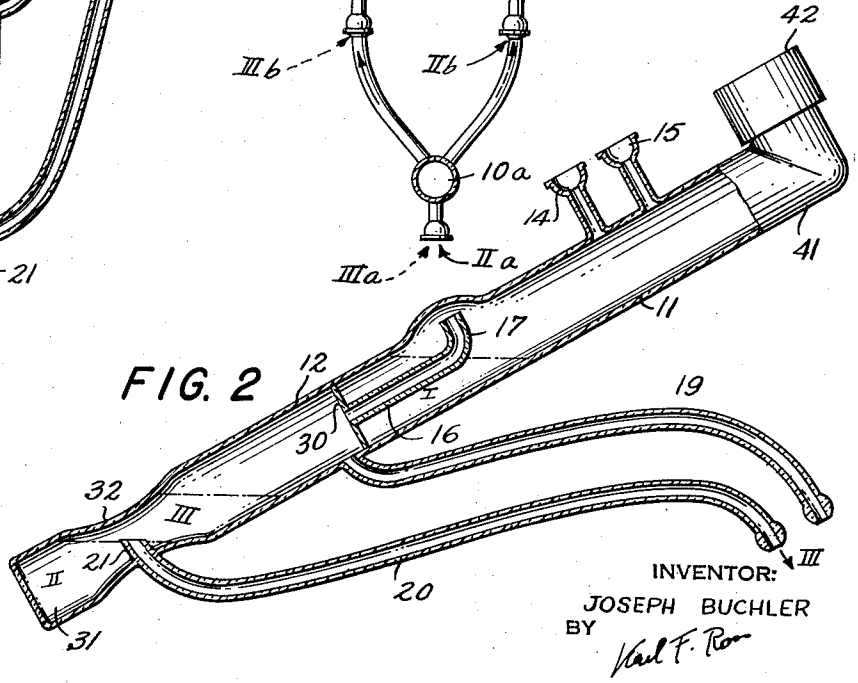


FIG. 2

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DEVICE FOR FRACTIONATED EXTRACTION USING MORE THAN TWO SOLVENTS

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8 Claims. (Cl. 23—270.5)

My present invention relates to an improved device for the recovery of a desired component from a mixture of substances having an affinity for different solvents which are immiscible with one another, a procedure sometimes referred to as "fractionated extraction."

In my co-pending application Ser. No. 580,022, filed on 23 April 1956, now Patent No. 2,892,688, issued 30 June 1959, I have described a device suitable for successively removing two mobile phases from a tube containing a multi-phase mixture. According to this previous application, a unit comprising two rigidly joined, substantially parallel tubes linked by a connecting duct is used, either single or combined to an array, to perform the distribution, transfer and withdrawal of the various liquid phases.

It is an object of the present application to bring forth further improvements and modifications of my previously disclosed device with a view to reducing both the number of parts and the space required for the fractionated-extraction process.

To accomplish this, the two tubes of the previous application have been combined into a single tubular unit, comprising essentially a pair of rigidly interconnected, aligned glass tube portions. The first, preferably longer, vessel portion is here again provided with one or more entrance apertures and one exit aperture while the second vessel portion has two exit openings and one entrance opening, the latter being joined to the exit aperture of the first vessel portion. The openings are so arranged that, upon a tilting of the unit at different angles, one mobile phase can pass through one of these openings while the other mobile phase or phases will leave the vessel portion by the other opening.

The novel arrangement of the two vessel portions also simplifies the mode of interconnecting a plurality of units into a cascade array in such a manner as to obtain, within a restricted space, two mobile phases moving over different paths through the array. Thus, an array of this description may be more conveniently mounted in a rack or holder enabling the simultaneous tilting of all the units in a proper manner.

The invention will be described in greater detail with reference to the accompanying drawing in which:

Fig. 1 is a sectional view of a unit embodying the invention, comprising a main tube portion and a somewhat shorter auxiliary tube portion, with both the fixed and mobile phases introduced into the main tube portion;

Fig. 2 is a sectional view showing the unit of Fig. 1 in an inclined position in which the mobile phases are decanted from the main tube portion to the auxiliary tube portion from which the upper mobile phase can be readily withdrawn;

Fig. 3 is a similar sectional view showing the unit in horizontal position to illustrate the withdrawal of the middle mobile phase from the auxiliary tube portion; and

Fig. 4 is a top plan view of several units as shown in Figs. 1 to 3 coupled together to form several stages of a cascade array.

The unit **10**, Figs. 1 to 3, comprises a main glass tube portion **11** and, coaxial therewith, an auxiliary glass tube portion **12** rigidly interconnected so as to form a single tube subdivided into two compartments by a transverse diaphragm **30**. Tube portion **11**, which functions as a mixing chamber, has two entrance nipples **14**, **15** and a connecting duct **16** leading, as a sole entrance, into an overflow chamber defined by tube portion **12**. Connecting duct **16** terminates, within tube portion **11**, in an elbow **17** terminating within a bulge **40** of this tube portion. The end of tube portion **11** remote from portion **12** has a widened elbow **41** provided with a screw cap **42**. The mixing chamber **11** can be thoroughly rinsed through this elbow and through entrance nipples **14**, **15** when screw cap **42** is removed.

The extremities of nipples **14**, **15** form female coupling halves which are complementary to male coupling halves formed by the extremities of the two exit nipples **19**, **20** of tube portion **12**. The two nipples **19**, **20**, which are laterally bent on opposite sides of a plane bisecting the tube portions **11**, **12** and the nipples **14**, **15**, can thus be coupled to respective units of a second stage in a triangular array, as illustrated in Fig. 4. Nipple **19**, rising from the end of auxiliary tube portion **12** close to the main tube portion **11**, extends at an inclination and terminates at the same level as entrance nipple **15**. Nipple **20**, whose inclination is roughly the same as that of nipple **19**, has an extension **21** entering tube portion **12** at a point whose distance from the closed end of that tube portion is selected to define adjacent that end a storage space **31** whose volume is a fraction (e.g. about one-half) of the volume of the space existing in tube portion **11** between levels of elbow **17** and nipple **14**; exit nipple **20** terminates at the same level as entrance nipple **14**.

In operation, portion **11** is filled in its horizontal position (Fig. 3) with three immiscible solvents constituting a lower phase I, a middle phase II and an upper phase III. These solvents will generally be liquids, although it should be understood that they may also include solids, e.g. in powder form, especially as the lower phase I. The volume of these solvents, which in a practical case may be up to 25 cc. per phase, is so selected that the level of the lower phase I will not reach the orifice of the inclined duct extension **17** in any operative position, as will be apparent from Figs. 1–3, and that in the upright position of the unit the combined level of all three phases will remain below the level of the lower entrance nipple **14**, as clearly seen in Fig. 1.

After the solvents and the sample to be analyzed have been introduced into tube portion **11**, the unit is tilted forwardly so that the elbow **41** thereof will be at its lowest point, with elbow **17** facing upwardly as in Fig. 3. The unit may now be shaken by tilting it from this position back and forth to the horizontal. After the contents of tube portion **11** have thus been thoroughly agitated, the unit is left to rest so as to allow the several phases to settle out and separate; this may be done in the horizontal or in any slightly tilted position in which the solutions do not reach entrance nipples **14**, **15** or the top of elbow **17**.

Next, the unit is carefully tilted back into its vertical position (Fig. 1) whereupon the two mobile phases will flow through elbow **17** into the auxiliary tube portion **12**. If required, the unit may now be left to allow the phases to settle for a short period. For the next step, the decanting of mobile phases III and II, the unit will be inclined forwardly as shown in Fig. 2 until the meniscus at the interface between phases III and II just touches extension **21** of nipple **20**. Upper phase III now flows out through nipple **20**, whereupon a further tilting of the unit to substantially horizontal position, as shown in Fig. 3, enables withdrawal of the middle phase II through

nipple **19**. The bevel of extension **21** is roughly parallel to the direction of the main portions of nipples **19** and **20**. This extension **21** is located at a restricted portion **32** of chamber **12** which reduces the volume of phase II present above this extension and renders less critical the positioning of the unit in the decanting operation of Fig. 2.

Subsequently, fresh middle and upper phases may be added through nipples **14** and/or **15**, followed by a repetition of the same cycle. Eventually, fixed lower phase I may be removed from tube portion **11** through nipples **14**, **15** by placing the unit horizontally on its face with these nipples facing down, or through elbow **41** after removal of screw cap **42**.

Fig. 4 illustrates how, for a serial decantation, an array of units can be built up from a first stage represented by a single unit comprising a tube **10***a*; a second stage formed by two units **10***b* and **110***b*; a third stage composed of three units **10***c*, **110***c* and **210***c*; and so forth. The exit nipples **19** and **20**, extending laterally on opposite sides from the central phase of each unit, engage with the entrance nipples **14** and **15** of the left-hand and right-hand units, respectively, of the next stage. For connecting the entrance and exit nipples with each other, suitable clamps may be used, e.g. as shown in Fig. 1 of my above-mentioned Patent No. 2,892,688.

The array of units may be fastened to a common holder or mounting rack of suitable type, e.g. as illustrated in Fig. 7 of my co-pending application.

Fig. 4 also represents a flow chart illustrating the paths over which the mobile phases II (solid arrow) and III (broken arrow) move through the array. It will be assumed that all main tube portions originally contain their allotted amount of fixed phase I which remains therein throughout the extraction process; tube **10***a* also contains initially the mixture to be fractionated. Subsequently, middle and upper phases are introduced from without into tube **10***a* as indicated by arrows II*a* and III*a*, respectively.

Following the agitation, resting and decanting steps previously described, middle phase from tube **10***a* is transferred to the next tube **10***b* which also receives fresh upper phase from without, through its unconnected entrance nipple, e.g. by means of a syringe (not shown), as indicated by arrow III*b*; at the same time, upper phase from tube **10***a* is transferred to the tube **110***b* to which fresh middle phase is added from the outside in the previously described manner, as indicated by arrow II*b*. Agitation and decanting again take place, whereupon middle phase from tubes **10***b* and **110***b* is transferred to tubes **10***c* and **110***c*, respectively, upper phase from these same tubes being simultaneously transferred to tubes **110***c* and **210***c*, respectively.

It will thus be seen that the central unit **110***c* of the third stage receives both middle mobile phases from the preceding stage but that the end tubes **10***c* and **210***c* receive only one phase each, the remaining phase being again added from outside, through their respective unconnected entrance nipples, as indicated by arrows II*c* and III*c*. The same operational method is valid for any subsequent stage.

The number of outlets available in an array with a number n of stages equals $2n$; half of these outlets carry the mobile middle phase II, the other half carrying the mobile upper phase III. The total number of units in an n-stage array is $n(n+1)/2$. In practice, it has been found convenient to use assemblies of eleven stages or more, resulting in an extraction ratio of almost unity.

My invention is, of course, not limited to the specific embodiment described and illustrated but may be realized in various modifications and adaptations without departing from the spirit and scope of the appended claims. It will be appreciated, for example, that fractionation with four solvents will be possible by decanting the three uppermost (mobile) phases from a vessel similar to tube portion **11** into a unit **10** and proceeding with the separation of these phases within the latter unit in precisely the manner hereinabove described, and that in analogous manner the number of phases may be further increased, subject only to the availability of a sufficient variety of suitable solvents.

I claim:

1. A device for the fractionation of a mixture, comprising an elongated tube, a diaphragm in said tube subdividing the interior thereof into a lower and an upper chamber, and a duct rising from said diaphragm into said upper chamber and forming a passage interconnecting said chambers, said tube being provided with two entrance apertures opening into said upper chamber at different elevations above the upper end of said duct, said tube being further provided with a lower and an upper exit aperture opening into said lower chamber at different levels above the bottom of said lower chamber, the portion of said lower chamber extending between said bottom and the level of the lower exit aperture being of substantially smaller volume than the portion of said upper chamber extending between the levels of said upper end of said duct and of the lower one of said entrance apertures, both of said entrance apertures being located on the same side of said upper chamber, said exit apertures being provided on a side of said tube opposite the side of said entrance apertures.

2. A device according to claim 1 wherein said upper end of said duct forms an elbow turned toward the side of said entrance apertures.

3. A device according to claim 1 wherein said tube is formed at the side of said entrance apertures with an outward bulge at the level of said elbow, said duct terminating at said bulge substantially flush with the inner wall of said upper chamber.

4. A device according to claim 1, comprising a pair of entrance nipples extending outwardly from said entrance apertures in radial direction of said upper chamber, and a first and second exit nipple extending upwardly from said upper and said lower exit aperture, respectively, on opposite sides of an axial plane bisecting said entrance nipples, said exit nipples respectively terminating at the levels of said entrance nipples and having free extremities complementary to those of said entrance nipples.

5. A device according to claim 4 wherein said second exit nipple is provided with an inward extension entering said lower chamber.

6. A device according to claim 5 wherein said inward extension is beveled in a plane substantially parallel to the general direction of said exit nipples.

7. A device according to claim 5 wherein said lower chamber is provided with a restricted portion, said inward extension being located at said restricted portion.

8. A device according to claim 1 wherein said upper exit aperture is located adjacent said diaphragm.

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