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3,004,567

COLLECTING MEASURED QUANTITIES OF LIQUID

Filed April 14, 1958

3 Sheets-Sheet 1

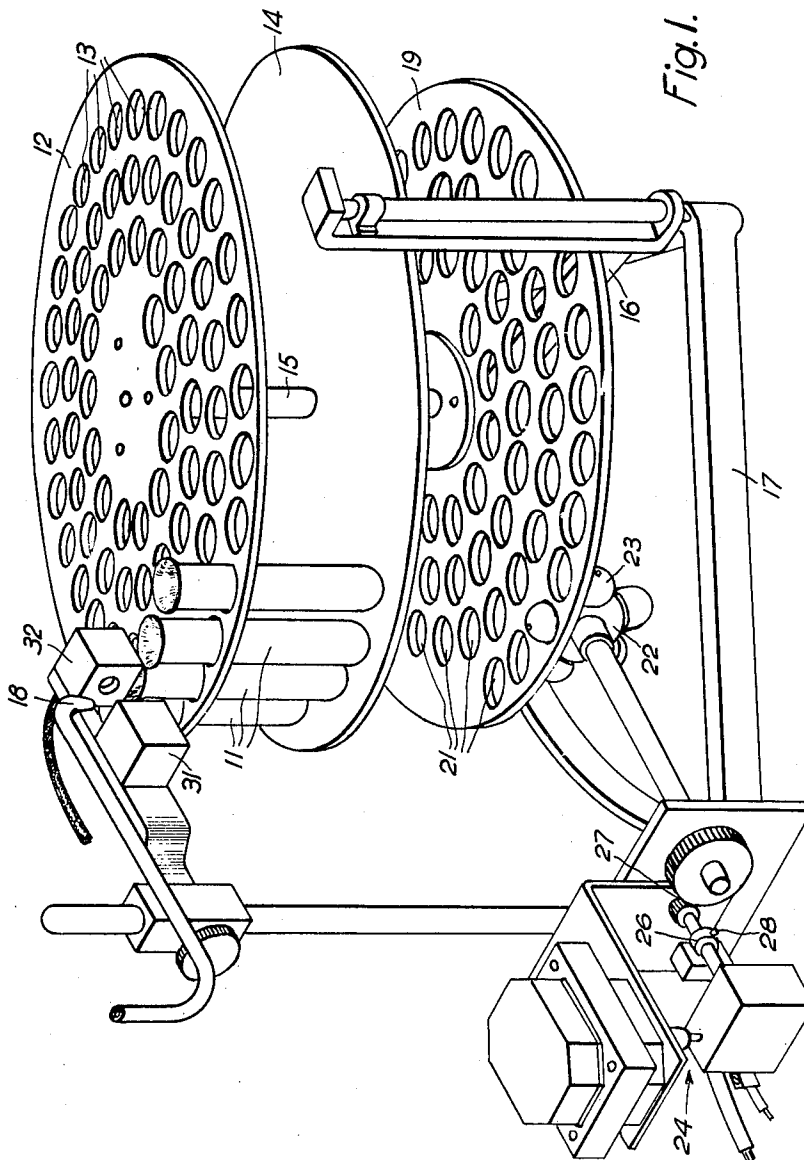


Fig. 1.

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3 Sheets-Sheet 2

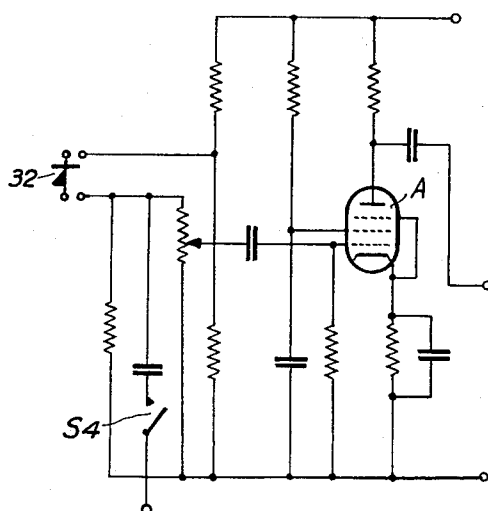


Fig. 2.

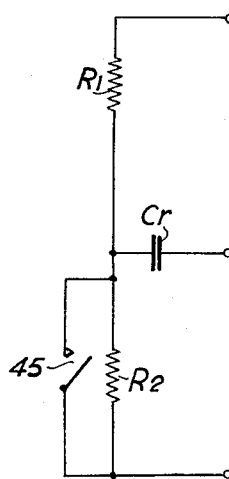


Fig. 4.

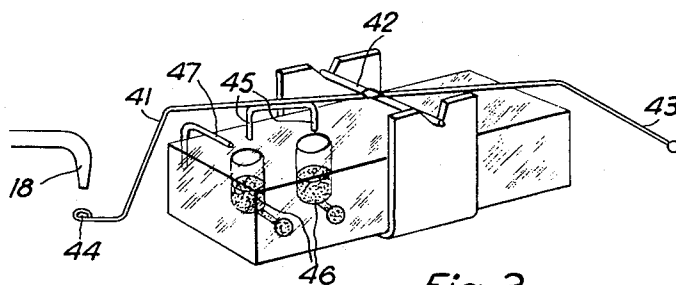


Fig. 3.

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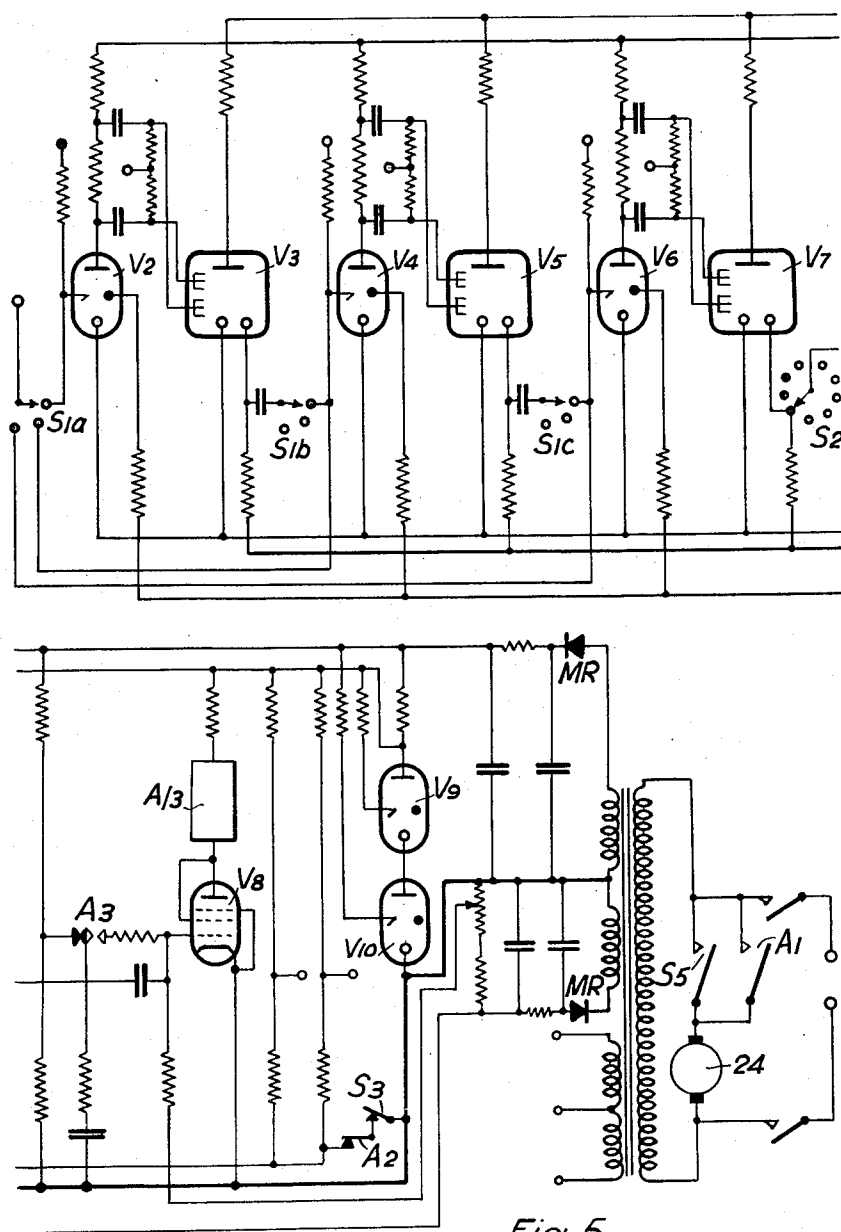


Fig. 5.

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COLLECTING MEASURED QUANTITIES
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12 Claims. (Cl. 141—130)

In many experimental and even industrial fields it is desirable to fractionate a delivery of a liquid into a large number of separate fractions of small volume. For example in the collection of the dropwise discharge of liquid from a chromatographic column it may be desirable to separate the discharged drops into some hundreds of separate samples each of small volume of a few cubic centimetres.

This has been done by counting the drops, the count of the drops being utilized periodically to actuate means to move a turntable carrying the sample collecting vessels (which may conveniently be test tubes) through the angle between adjacent collectors. The drops are counted e.g. by the aid of a beam of light and an electrical counter.

It is known to provide the turntable with a single row or with a plurality of rows, but the angular separation is the same in each row, so that either arrangement wastes a considerable amount of the turntable space and requires the use of a large turntable to carry the large number of sample collectors required. This in turn requires a relatively large and powerful motor to drive the turntable. Thus if two hundred $\frac{3}{8}$ -inch test tubes are carried in a single circle upon a turntable the turntable must be 4 ft. in diameter.

An object of the invention is to provide an arrangement which is more compact and simpler and cheaper to build and to operate than known collectors.

In accordance with the invention the collecting vessels are disposed in a plurality of rows around a common axis with the same linear centre to centre distance between the vessels in every row.

Thus the sample collecting vessels may be on a turntable in a spiral formation about the axis of the turntable at a substantially constant centre to centre distance and the turntable is supported and moved so as to bring each of the collecting vessels in turn to the point at which the liquid is discharged into the vessel. By this means a considerable number of collecting vessels can be carried upon a very much smaller turntable than would be required if the same number of vessels were arranged in a single circle or in a number of concentric circles at constant angular separation.

The actual movements may be controlled from a second disc provided with recesses or holes corresponding accurately with the positions of the collecting vessels and actuated from the equivalent of a gear wheel. In this way, a relatively wide tolerance from the manufacturing point of view can be allowed in the spacing of the collecting vessels.

The invention will be further described with reference to the accompanying drawings in which:

FIGURE 1 is a diagrammatic perspective view showing the essentials of one example of embodiment of the invention.

FIGURE 2 is a circuit diagram of the counting system used in FIGURE 1.

FIGURE 3 is a diagrammatic perspective view of an alternative form of drop counting system to that shown in FIGURE 1.

FIGURE 4 is a circuit diagram of the counting system used in FIGURE 3.

FIGURE 5 is a circuit diagram of the counter itself.

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The embodiment shown in FIGURE 1 is for use with test tubes 11 as sample collecting vessels and it comprises a flat metal turntable 12 in which holes 13 are arranged on a spiral of close pitch with the centre to centre distance of the holes constant. The test tubes may hang from their rims in the holes or may be supported by a further disc indicated at 14. The turntable 12 is carried upon a vertical spindle 15 which is mounted in turn upon an arm 16 swingingly mounted on a base support 17 so that by conjoint movement of the arm and rotation of the turntable all of the tubes 11 may be brought in turn into a position under a liquid outlet shown at 18. A disc 19 having holes 21 arranged identically to the pattern of holes 13 in the turntable is carried upon the lower end of the vertical spindle 15 and serves to control the movement of the turntable 12 to bring each of the test tubes 11 in turn into position to receive the drops of liquid delivered. Since the centre to centre distance of adjacent holes in the disc is exactly equal to the centre to centre distance of adjacent holes in the turntable, the ratio of the distances is 1:1, which ratio is an integer. The disc 19 is moved by a small wheel 22 which carries a number of hemispherical projections 23 equally spaced upon its periphery. These projections 23 enter the holes 21 from below, the wheel 22 and disc 19 thus constituting an elementary form of crown wheel drive which adapts itself to the changing radius at which the holes 21 lie from the axis of the spindle 15 and which also permits wide manufacturing tolerance. The projection might instead be on the disc 19 and the holes in the wheel 22. Also any other tooth form which will give the same result may be used. Rotation of the wheel 22 through one tooth space will step the disc 19 and with it the turntable 12 through the centre to centre distance between the holes 13 throughout the whole range of the spiral holes, and thus if the disc 19 is arranged in the same relationship to the turntable 12 as the wheel 22 is in relation to the outlet 18, the next subsequent test tubes will be brought into the correct collecting position at each such step.

The turntable 12 may be interchangeable with others having holes of different sizes for different sized tubes 11 but of the same centre to centre spacing and arrangement.

A small geared electric motor 24 provides the motive power to drive the wheel 22. This is started by an output relay closed by the drop counter described below and stops itself after bringing an empty tube into position, that is after moving the driving shaft of the wheel through a predetermined angular distance, by means of a switch actuated from the shaft. This stopping may be brought about by the operation of a microswitch S5, by a cam 26 on the shaft of the motor. As shown the wheel 22 has five projecting teeth 23, and gearing 27 between the motor shaft and wheel shaft has a ratio of 2:5 so that the cam 26 has two active projections 28, 180° apart.

Various known drop counting methods may be used. Thus, as shown in FIGURE 1, a photo-electric drop counting system may comprise a light source 31 to produce a beam of light which is focussed upon a germanium junction photo-cell indicated at 32. When a drop interrupts the light beam the photo-cell produces a voltage pulse which is amplified at A FIGURE 2 and is passed to the counter described below with reference to FIGURE 5. The threshold sensitivity of the amplifier may be adjusted so that it responds to normal drops but not to minute secondary drops. The switch S4 enables a connection to be made to a main supply at a suitable voltage for testing purposes.

Another possibility is to arrange for the drop to fall between the electrodes of a condenser, when the change in capacitance may be detected by a suitable circuit which

delivers a voltage pulse which after any necessary amplification can again be counted by the counter of FIGURE 5.

The drops may be detected by the movement of a mechanical system. For example, as shown in FIGURE 3, a short length 41 of wire may be balanced upon a pivot 42 by a counterpoise 43 and located so that a loop 44 upon one end fills with liquid until a drop is formed therein. As the drop comes to a predetermined size, the wire 41 tips about its pivot 42 and brings two platinum contacts 45 into respective pools 46 of mercury, thereby to close an electric circuit and provide a counting pulse by means of the circuit shown in FIGURE 4. When the wire reaches a stop 47 the drop falls off into the sample collecting tube in position below and the wire 41 returns to its original position.

The circuit shown in FIGURE 4 is intended to be energised from a stabilized high tension voltage supply available from the circuit of FIGURE 5 and therefore includes a potentiometer constituted by the resistances R1 and R2 to reduce the voltage across the contacts 45 and it also includes a capacitor Cr to avoid a direct connection to the counter circuit.

The counter may be of the same form as a commercial batching counter but a somewhat simpler circuit may be used. This uses a cold cathode counting tube having separate connections to each cathode so that by selecting the appropriate cathode it is possible to predetermine the number of drops passing before an output relay is energised. The output selector may be preceded by one or more stages each multiplying the selected number by a predetermined factor. Thus by suitable switching arrangements it is possible to count units up to the number provided in the counting tube or in a multiple of any of these numbers.

The circuit shown in FIGURE 5 has an output tube counting in units from 1 to 9 preceded by two switchable multiplying stages, each multiplying by a factor of ten so that it is possible to count in units from 1 to 9, in tens from 10 to 90, or in hundreds from 100 to 900.

The output tube is a "Dekatron" tube with ten cathodes and is shown at V7, and the impulses which actuate it come through a cold cathode trigger valve V6. The several cathodes of the tube V7 are connected to the contacts of a selector switch S2 and when the count reaches the selected value, an impulse passes to a multiplying tube V8, thereby exciting an output relay A3 having three contacts. The contact A1 which is thereby closed is in parallel with the switch S5, so starting the motor 24; the contact A2 which is thereby opened returns the counter circuit to zero; and contact A3 which is thereby changed over causes the tube V8 to cease the excitation of the output relay A3, whereupon the contacts A1, 2, 3 return to the starting position. By now the motor 24 has rotated the cam 26 sufficiently for the switch S5 to close, so that the motor continues to run until the cam 26 reopens the switch, thus stopping the motor when the next collecting vessel 11 is in position beneath the outlet 18. Counting of the drops collected starts afresh and when the selected number is reached the above cycle is repeated.

If the input pulses from the drop counting system pass directly to the trigger valve V6, the tube V7 will count the drops in single units. A similar counting tube V5 with its trigger valve V4 has only its tenth cathode externally connected and if brought into action, for every ten impulses it receives causes an impulse to be given to the tube V7, so that the count will then be in tens of drops. Yet another similar counting tube V3 with its trigger valve V2 has only its tenth cathode externally connected and if brought into action, for every ten impulses it receives causes an impulse to be given to the tube V5 which in turn for every ten impulses it receives causes an impulse to be given to the tube V7, so that

the count will then be in hundreds of drops. Selection of units, tens or hundreds is effected by a switch S1 having three sets of contacts marked S1a, S1b and S1c respectively, and the action of which will be clear without further description. Other usual components of the circuit such as a supply transformer, rectifiers MR, voltage stabilising tubes V9, V10 and the like will also be clear to those skilled in the art without further description. It will further be obvious that any desired number of multiplying stages can be provided. The multiplying stages are readily modified to multiply by factors of 2 or 5 using 10-way tubes or by 2, 3, 4, 6 or 12 using 12-way tubes. In this way the number of drops to be collected in each sample may be varied over a wide range.

It will be understood that the speed of movement of the turntable 12 by the wheel 22 should be such that no liquid is lost during the change from one test tube 11 to the next. If necessary vessels with wide funnel-like openings can be used so that a drop or drops falling during the movement of the turntable are caught in the correct vessel.

If rather larger samples or fractions are required than implied by the use of a drop-counting technique, a device of known kind which incorporates an electrically controlled valve to discharge a measured quantity of liquid into each successive vessel may be used. Other such possibilities include the use of a siphon which discharges a given volume of liquid, so arranged as to make and break electrical contacts, or a weighing mechanism consisting of a bucket suitably counterbalanced which with a given quantity of liquid, tilts and discharges its contents into a collecting vessel which is in position and then returns to the filling position. The tipping movement of the bucket is employed to actuate electrical contacts. The circuit modification necessary will be obvious to those skilled in the art.

If the holes 13 (and therefore also the holes 21) are arranged at a constant centre to centre distance in concentric circles instead of in a spiral formation, the spindle 15 may still be supported on a swinging arm such as 16 and contacts be arranged on the disc 19 (or on the turntable) so as to be closed (as by engagement with a stationary ramp) to divert the current controlled by the switch S5 and the relay contact A1 from the motor 24 to a single cycle mechanism such as a solenoid and ratchet feed which swings the arm, thus effecting a change from the last vessel of one circle to the first vessel of the next circle, the circuit being opened again at the end of the cycle. The corresponding holes 21 in the disc 19 would be joined by a slot to allow the disc to move readily in relation to the engaged tooth 23 of the wheel 22.

An alternative arrangement of switching for the motor 24 avoids the risk of the motor stopping due to release of the relay A/3 before the cam 26 has allowed the switch S5 to reclose. In this alternative the impulse from the drop counter operates a single-pole change-over switch which is stable only in either of its two positions, and the switch S5 is also a change-over switch, while the cam is shaped so that this switch is changed over at the necessary intervals (180° in the present example) and is held for the whole interval. The motor and supply are then connected in series between the arms of the two switches. Then the motor runs for the necessary interval when the change over of the micro-switch stops it until another impulse changes over the single pole switch and so on.

We claim:

1. Apparatus for collecting measured quantities of liquid from a common point of discharge comprising a rotatable spindle, a turn-table on said spindle, a plurality of collecting vessels supported by the turn-table in a spiral formation about the axis of the turn-table at a substantially constant centre to centre distance, a disc fastened to the spindle having openings therein in a

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spiral formation which matches in its form that in which the collecting vessels are disposed, a gear wheel meshing with the openings in the disc thereby to constrain the disc both circumferentially and radially, means for rotating the gear wheel intermittently through an amount which will bring each vessel in turn to a predetermined position in relation to the gear wheel, means for discharging liquid at a predetermined position so that it is collected by the vessel at that point, and means for determining the periods during which the vessel remains stationary at the predetermined position.

2. Apparatus as set forth in claim 1 in which the teeth of the gearwheel are in the form of rounded projections.

3. Apparatus as set forth in claim 1 wherein the axial position of the gear wheel remains fixed and said turntable and disc are supported by means permitting radial movement.

4. Apparatus as set forth in claim 3 in which the rotatable spindle is journaled in a swingably mounted arm, the swinging of said arm effecting the radial movement of the turntable and disc.

5. Apparatus as set forth in claim 3 in which the ratio of the centre to centre distance between adjacent vessels on the turn-table to the centre to centre distance between adjacent openings in the disc is an integer.

6. Apparatus as set forth in claim 5 in which there is a one to one correspondence between the centre to centre distance between adjacent vessels on the turn-table and between adjacent openings in the disc and the gear wheel is rotated through the pitch distance between adjacent teeth between successive intervals.

7. Apparatus as set forth in claim 1 in which said means for pre-determining the periods at which each vessel remains stationary at the pre-determined position is actuated by the passage of liquid into the vessel so that each vessel gives place to the next succeeding vessel when a measured quantity of liquid has been collected.

8. Apparatus for collecting measured quantities of liquid in collecting vessels from a common point of discharge comprising a supporting frame, a horizontal driving shaft journaled in said frame having a gear wheel with rounded teeth, a movable support carried in said frame, a turn-table and a disc fastened together on a

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common vertical axis journaled in said movable support to the driving shaft, the movement of said support being free and in a direction such that the axis of the turntable and disc can move axially in relation to the driving shaft, a series of collecting vessels carried by said turntable disposed in a spiral formation around its axis at a constant centre to centre distance, a matching spiral series of holes in said disc, said gear wheel meshing with said holes thereby constraining the disc both circumferentially and radially so that rotation of said gear wheel causes the collecting vessels to come into succession to a pre-determined position, liquid discharge means located at the pre-determined position, means for rotating said driving shaft, and means for interrupting the drive when a vessel is brought to the pre-determined position and for re-starting it when a measured quantity of liquid has been discharged into the vessel.

9. Apparatus as set forth in claim 8 wherein the means for rotating said driving shaft is an electric motor and the means for interrupting the drive comprises a switch and a cam driven by the motor operating the switch.

10. Apparatus as set forth in claim 9 in which the means for restarting the drive includes an output relay which when actuated closes a supply circuit to the motor, and means for actuating the relay when a measured quantity of liquid has been discharged into the vessel.

11. Apparatus according to claim 10 in which the means for measuring the liquid discharged comprises a photoelectric drop counting system and an electric counting circuit which when a predetermined count is reached excites the output relay.

12. Apparatus according to claim 11 in which the counting circuit includes a multi cathode counting tube and at least one switchable multiplying stage preceding the tube.

References Cited in the file of this patent

UNITED STATES PATENTS

2,672,271 Gorham ----- Mar. 16, 1954

FOREIGN PATENTS

325,376 Great Britain ----- Feb. 20 1930