

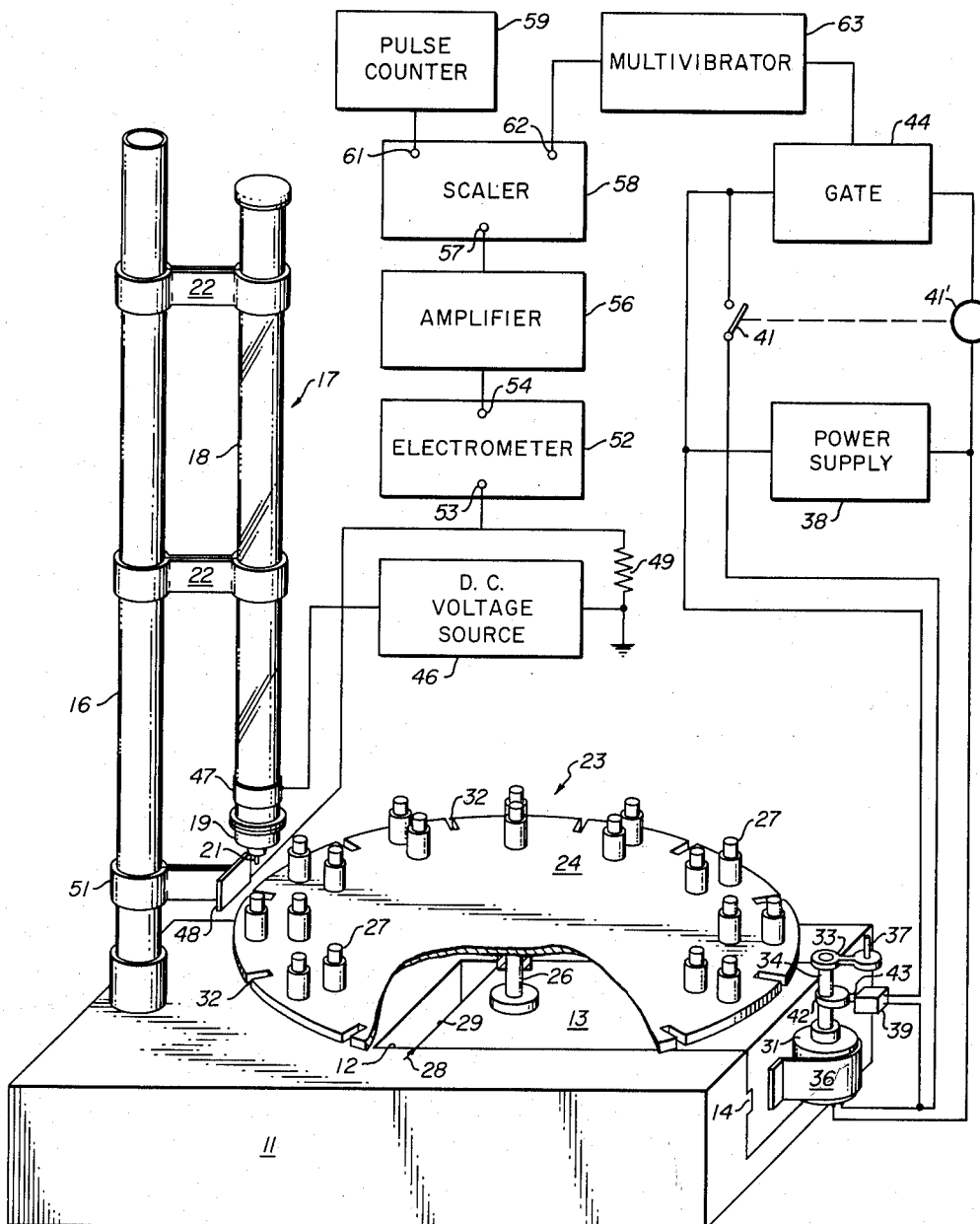
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FRACTIONATING COLUMN PRODUCT COLLECTOR CONTROL

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FRACTIONATING COLUMN PRODUCT COLLECTOR CONTROL

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The present invention relates to chemical fractionat-
ing columns and more particularly to the automatic col-
lection of eluted products therefrom. The invention uti-
lizes electronic means for detecting minute globules of
eluate from the column orifice whereby the rotation of
a turntable is controlled to position new collecting vessels
under the column and thus separately collect successive
portions of the eluate.

In a column fractionating process such as the well
known ion exchange processes, the components of the
mixture in solution are separated by virtue of the dif-
ferent rates of adsorption inherent to the particular com-
ponents. A small orifice at the base of the column allows
the absorbed and subsequently eluted components to
leave the column a drop at a time to be separately col-
lected in a sequence of collecting tubes. Successive tubes
of a group will then typically contain a concentration of
a single component in a gaussian distribution of concen-
tration within the group, and successive groups of tubes
within the sequence will contain the separated compo-
nents in the order of decreasing rates of adsorption. In
various other separation processes, use is made of es-
sentially similar collection apparatus.

Generally the collecting tubes are arranged in a circle
on a rotatable circular rack or turntable with the tubes
being equally spaced around the turntable. The frac-
tionating column is disposed above the rack, over a point
on the circle of tubes, whereupon rotation of the turntable
successively passes each of the tubes under the orifice of
the column.

A problem is present in providing a means to syn-
chronize the rotation of the turntable with the drop flow
rate of the eluate in order that a new collecting tube will
automatically be positioned under the orifice of the
column at the necessary intervals. Since the drop flow
rate of a liquid is tedious to establish and may not be
constant, it is not practical to pre-set the turntable rota-
tion to a corresponding speed for it can be seen that any
phase discrepancies may be compounded and the opera-
tion can easily become completely out of phase. For
continuous synchronization it is necessary to have a means
whereby each successive motion of the turntable is timed
by the occurrence of the eluate portion to be collected.
A conventional method of effecting this result uses a
photocell and a light source placed along the path of
fall of the drop and arranged such that the reflected light
from the passing drop is detected by the photocell. The
output of the photocell is coupled to the turntable activat-
ing circuitry whereby a signal from the photocell triggers
the turntable rotation.

This method of detection, however, has been found
to be unsatisfactory with some fractionating column
operations such as isotope separation and ion exchange,
inasmuch as these operations sometimes involve a product
drop size so small that the drop presents insufficient light-
reflecting surface for reliable detection by the photo-
cell. In addition to this, very minute drops have a tend-
ency to leave the column orifice at various points about
the orifice rim and thus pursue slightly different paths of
fall. Some of these paths may not fully intercept the
light beam and consequently detection by the photocell
is further impaired.

In an attempt to overcome this problem, another method

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offers detection by means of passing the drops through
an electrostatic field established beneath the column ori-
fice. The charge disturbance created by the presence of
the drop in the field is detected by appropriate sensing
apparatus which apparatus, in turn, provides a signal
to the turntable activating circuitry. Since a relatively
large field area can be used, this system is reliable in
detecting drops with deviating paths of fall. However,
since the potential variation caused by the drop is related
to the size of the drop presented to the field, no ad-
vantage is gained in detecting very minute drop sizes.
With the recent increased interest in ion exchange and
isotope separation, a need for a more sensitive control
means has developed.

The present invention provides a means of eluate flow
detection to actuate a collector tube turntable which means
is independent of the size of the drops and of the vari-
ations in the path of fall thereof. The detection is ac-
complished by applying a D.C. voltage to the fractionat-
ing column, causing each drop therefrom, as it forms at
the column orifice, to collect an electrical charge. Upon
leaving the orifice, the charged drops pass the vicinity
of an electrically conducting plate, which is connected
to ground through a resistance, before entering the col-
lecting tubes, creating an electrostatic field between the
drop and the plate. An electrometer tube, coupled to
the plate, responds to the appearance of this potential
and produces an output signal, which signal activates the
turntable driving motor. The motor is provided with
controls whereby each signal causes the table to rotate
the adjacent tube into position under the column orifice
which tube will then receive the succeeding drop from
the column. By this means the drops are detected by
the charge rather than size and with the increased sensing
area offered by the plate the need for a specific path of
fall is avoided. In addition, a scaler included in the cir-
cuitry provides ready adaptability of the apparatus to
column operations requiring the collection of multiple
drops by each tube and to drop counting.

It is accordingly an object of this invention to im-
prove and implement fractionating column processes by
providing accurate and automatic operation of the prod-
uct collection apparatus used in conjunction therewith.

It is another object of the invention to provide elec-
trostatic detection of eluted products from the outlet ori-
fice of a fractionating column.

It is another object of the invention to provide reliable
means for the detection of globules of eluate from a
fractionating column which detection is independent of
the size of the globule.

It is a further object of the invention to provide reli-
able means for the detection of very minute globules of
eluate from the outlet orifice of a fractionating column
which detection is sensitive over varied paths of fall of
the globules from the orifice.

It is another object of this invention to provide an
ultra-sensitive means of controlling the rotation of a
turntable to separate and collect successive products from
an ion exchange column.

It is still another object of the invention to provide
means for successively positioning new collector vessels
under the outlet orifice of a fractionating column upon
each occurrence of a selected number of drops there-
from.

It is a further object of the invention to provide an
accurate means for counting liquid product droplets
emitted from the outlet of a chemical separation column.

The invention, both as to its organization and method
of operation, together with further objects and advan-
tages thereof, will be best understood by reference to
the accompanying drawing which shows the mechanical
components of the invention in perspective and in which

certain of the electrical components are shown schematically.

Referring now to the drawing there is shown a rectangular base 11 upon which the mechanical components of the invention are mounted. The upper surface of base 11 is provided with a broad and deep longitudinal groove 12 which extends from approximately the center of the base through one end. A long rectangular block 13 fits the groove 12 and is slideably held therein by rabbet and tenon fittings 14 on the sides of the groove and the block. This arrangement provides a translatable portion to the base 11 whereby components mounted on the block 13 may be adjustably positioned with respect to components mounted on the base proper. A vertical pipe-like support post 16, to which the upright fractionating column 17 is attached, is rigidly mounted on base 11 and is disposed along the center line of the base opposite from groove 12. The fractionating column 17 containing the liquid to be processed comprises a cylindrical glass tube 18 with a bottom closure 19 and having a minute outlet orifice 21, this structure being conventional and being well understood within the art. The column 17 is secured to support post 16 by two spaced double-ring clamps 22 such that the column is located on the side of the post toward groove 12 and is positioned with the outlet orifice 21 a substantial distance above the base 11.

A collector tube turntable 23, comprising a large circular rack disc 24 is mounted to rotate about a vertical shaft 26 which shaft extends upwardly from sliding block 13. Rack 24 holds the collector tubes 27 to receive the fractionated liquid. The tubes 27 in this embodiment are arranged in two concentric circles on the rack 24 are equally spaced about each circle with the spacing decreased around the inward circle so that the tubes are in radial alignment with respect to the rack 24. Two circles of tubes are used in this embodiment of the invention however conventional racks having provision for more or fewer circles of collector tubes may be employed. With the turntable 23 mounted on the sliding block 13, either of the circles of collector tubes 27 may be oriented under the column orifice by the translation of the block in groove 12. The spacing between the fractionating column 17 and the support post 16 is selected to accommodate the free passage of the outermost portions of the rack 24 when block 13 is fully inserted in groove 12 and the inner circle of collecting tubes 27 is positioned under the column orifice 21. An index mark 28 inscribed in the base 11 along the side of groove 12 indicates the retracted position of the block 13 in the groove and alignment of the leading edge 29 of the block with mark 28 will accurately orient the outer circle of collecting tubes 27 under the orifice 21. With the block 13 in either position, rotation of the turntable rack 24 will pass each tube of the corresponding circle under the orifice 21. To provide for rotation of the turntable 23 by a driving motor 31, the periphery of the rack 24 is indented by a series of rectangular notches 32 which notches are spaced equidistantly between collector tubes 27.

The driving motor 31 is carried on the outside end of block 13 and is affixed thereto by a bracket 36 with the motor shaft 34 extending upwardly. A crank arm 33 is radially attached to the upper end of shaft 34 for rotation beneath the rack 24. A pin 37 which projects upwardly from the free end of crank arm 33 engages the notches 32 in rack 24 and transfers the rotation of shaft 34 to the turntable 24. The shaft 34 is geared to motor 31 within the motor housing such that the normal motor operating speed turns the crank arm 33 at a slow speed which may typically be one revolution per second. Thus each revolution of crank arm 33 advances the turntable by one tube spacing. While faster turntable speeds may be used if desired, a rate of one

tube per second will amply accommodate most column operations.

The driving motor 31 is energized by a power supply 38 and is coupled thereto through both a normally closed microswitch 39 and normally open relay contacts 41 which relay is thus connected in parallel with the microswitch. The microswitch 39 is disposed near motor shaft 34 and is actuated by an eccentric cam 42 affixed to the shaft 34 whereby the apogee of the cam may contact the microswitch actuator button 43 to open the switch when crank arm 33 is directed away from rack 24. The relay 41 is energized by relay driver coil 41' which coil is also coupled to power supply 38 through a gate circuit 44. The gate 44 is triggered by an input signal from the drop detecting circuitry, to be hereinafter described, to actuate relay 41 momentarily, thus closing the motor circuit and initiating motor rotation upon the occurrence of a drop from column 18. This rotation causes cam 42 to lose contact with actuator button 43 and thereby close the microswitch 39. Power to the motor 31 will now be supplied through microswitch 39 for the remainder of the crank revolution whereupon the renewed contact of cam 42 with button 43 will open the motor circuit and halt turntable rotation. Thus in the static condition, the apogee of cam 42 contacts actuator button 43 and holds the microswitch 39 open and the unenergized condition of coil 41' results in relay switch 41 being open. The motor 31 is completely decoupled from power supply 38 and crank arm 33 is at rest and directed away from rack 24 as shown in the drawing.

The described rotation of the motor shaft 34 engages connecting pin 37 with a notch 32 of the turntable rack 24 for approximately 120° of travel of crank arm 33. The rack 24 is thereby turned sufficiently to bring a new collector tube 27 into position under the column 18 for each cycle of operation.

To impose an electric charge on the drops of eluate from the column 18 by which charge the drops may be detected, a positive potential from a D.C. voltage source 46 is applied to the column by a metal conducting band 47 encompassing the column at a point near the bottom closure 19. The inherent impurities of the column and closure materials allow this charge to become distributed over the surface thereof and to be transferred to the eluate as the globules thereof form at the outlet orifice 21.

To detect each charged globule as it leaves the orifice 21, a sensing plate 48, electrically connected to ground through a resistor 49, is disposed in a vertical position slightly aside from the path of fall of the drops from the column orifice 21 to the rim of the collector tubes 27. The sensing plate 48 is supported in this position by an insulated ring clamp 51 mounted on support post 16. As the charged drop passes the vicinity of the plate 48 an electrostatic field is created between the drop and the plate, thereby inducing a charge on the plate. An electrometer circuit 52, having an input 53 coupled to the plate 48, detects this charge and produces an output signal in response thereto. The output 54 of electrometer 52 is connected through an amplifier 56 to the input 57 of a scaler 58. The scaler 58 is of the type having an adjustable scaling factor whereby the number of input pulses necessary to produce a single output pulse may be selectively varied. This adjustment allows the apparatus to be used for column operations requiring the collection of various portions of the eluate in each tube 27. By an appropriate setting of the scaler 58 the output signal to the turntable actuating circuitry will occur upon the collection of any selected number of drops in each collector tube. A first output 61 of scaler 58 is provided for optional connection to a drop counting or recording device 59. A second output 62 of the scaler 58 is connected to a monostable multivibrator 63 in which the time duration of each scaler output pulse is extended. The output of the multi-

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vibrator is coupled to the gate signal input of gate 44 of the motor switching circuitry thereby acting to close the motor circuit and initiate motor rotation as previously described.

Considering now the operation of the invention, the liquid to be fractionated is contained in the column 18 and the product drops therefrom in the conventional manner. In the rest position the crank arm 33 and pin 37 are disengaged from the turntable rack notches 32 and the apogee of cam 42 is in contact with actuator button 43 thereby holding the normally closed microswitch 39 open. The normally open relay 41 is unenergized also and thus the power supply 38 is completely de-coupled from the driving motor 31. The sliding block 13 of the base 11 is positioned either at index 28 or fully inserted in the groove 12 corresponding respectively to the inner or outer circle of tubes to be used and a first tube of the circle of collector tubes 27 centered under the outlet orifice 21 of the column 18. The D.C. voltage from voltage source 46 is applied to the column through conducting band 47 to charge the eluate as it forms a globule at the orifice 21. Upon leaving the orifice 21, the charged drop passes the sensing plate 48 and enters the first collector tube 27.

While the drop passes plate 48, the electrostatic field established therebetween induces a charge on the plate. The electrometer 52 detects the charge and sends an output pulse to amplifier 56, after which the amplified pulse is received by the scaler 58. Assuming it is desired to collect a single drop in each tube 27 and thus that the scaling factor of scaler 58 has been set at one, a scaler output pulse is immediately transmitted to monostable multivibrator 63. However if two drops of eluate are to be collected per tube, scaler 58 is set for a scaling factor of two and a second input pulse from electrometer 52 must be received before an output signal is transmitted to the multivibrator 63. Higher settings of the variable scaler 58 will cause a correspondingly increased quantity of eluate to be collected in each tube 27.

Upon being triggered by the scaler output pulse the multivibrator 63 applies a time extended pulse to gate 44 whereby relay 41 is energized for the duration of this pulse. Power is now connected to the driving motor 31 through relay 41 and motor shaft 34 begins rotating crank arm 33 toward turntable rack 24. Rotation of the shaft also brings the apogee of cam 42 away from actuator button 43 allowing the contacts of microswitch 39 to close. Multivibrator 63 will now cut-off to de-energize relay 41, however, power is still connected to the motor through microswitch 39. The continued rotation of shaft 34 swings coupling arm 33 under rack 24 and pin 37 engages a peripheral notch 32 thereof. Rotation is thus transferred to the rack 24 by the pin 37 for a portion of the revolution of crank arm 33 whereupon the pin dis-engages the notch and the rack has been advanced by one tube spacing. A second collector tube 27, is now in position under the column 18 to receive the next drop from the orifice 21. Further rotation of the shaft 34 returns coupling arm 33 to the initial rest position, at which point the apogee of cam 42 opens microswitch 39. Power is now disconnected from the driving motor 31 and the apparatus remains at rest until the next drop from the column 17 falls into the newly positioned second tube 27, thereby initiating the cycle again.

Since it is possible to impose a detectable electrostatic charge on an otherwise undetectable fluid particle, the invention provides a more sensitive, accurate and reliable eluate detection means than has been heretofore available.

While the described embodiment of the invention utilizes the detection means to actuate rotation of a turntable rack, the invention is also applicable to actuating other mechanisms for processing and handling minute liquid products where the improved reliability and increased sensitivity to very small drop sizes are needed. The turntable, for example, may readily be replaced with a conveyor belt driven by essentially similar mechanism.

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Thus while the invention has been described with respect to a single embodiment, it will be apparent to those skilled in the art that numerous variations and modifications may be made within the spirit and scope of the invention and it is not intended to limit the invention except as defined in the following claims.

What is claimed is:

1. In apparatus for controlling a movable carrier to successively position a plurality of collector vessels for receiving globules of fluid product from the outlet of a chemical processing column, the combination comprising a D.C. voltage source applying an electrostatic charge to said fluid globules at said outlet of said column, an electrically conductive element spaced a small distance from the path of said globules between said outlet and said collecting vessels, an electrometer circuit having an input coupled to said conductive element and producing an output signal in response to an electrical charge induced thereon by the passage of said globules thereby, and an electrically actuated motor operating said carrier in response to said output signals from said electrometer circuit.

2. In an eluate drop collector control apparatus for use with a chemical separation column, the combination comprising a plurality of collector vessels, a movable carrier for positioning successive ones of said plurality of vessels under the outlet orifice of said separation column whereby successive portions of the eluate therefrom may be separately collected, a voltage source connected to said column and applying an electrostatic charge thereto for charging eluate drops emitted therefrom, a conductive element of substantial area slightly displaced from the line of fall of said drops from said orifice to said vessels, an electrometer circuit having an input connected to said conductive element and producing an output signal in response to each induced charge thereon, an electrically actuated motor operatively coupled to said carrier, a normally open relay, an electrical power supply connected to said motor through said relay, and means coupled to said electrometer circuit and said relay for temporarily closing said relay in response to output signals from said electrometer circuit whereby said carrier is actuated to position a successive collector vessel beneath said column outlet.

3. In apparatus for controlling a movable carrier to successively position each of a plurality of collector vessels beneath the outlet of a chemical process column for separately collecting eluate droplets therefrom, the combination comprising an electrical motor coupled to said movable carrier for driving said carrier, a relay having normally open switch contacts and having a driver coil for closing said contacts upon energization of said coil, an electrical power source connected to said motor through said switch contacts, a voltage source connected to said column whereby a static charge is imparted to said eluate droplets at said column, a conductive element having a substantial surface laterally displaced a small distance from the path of fall of said droplets between said outlet and said collector vessels, a resistor connected between said conductive element and electrical ground, an electrometer circuit having an input coupled to said conductive element and producing an output pulse upon the detection of a charge on said element, and a gate circuit coupled between said electrometer circuit and said relay driver coil and acting to energize said coil in response to an output pulse from said electrometer circuit whereby said motor is actuated to position a succeeding one of said collector vessels under said column orifice.

4. In apparatus for collecting fluid products from the outlet orifice of a fractionating column, the combination comprising a plurality of separate fluid collecting vessels each having an opening at the upper extremity thereof, electrically driven means for successively positioning said vessels beneath said orifice of said column whereby successive portions of said fluid product from said column

can be separately collected, a voltage source connected to said column and applying an electrostatic charge thereto whereby the globules forming at said orifice collect a portion of said charge, a conductive element of substantial area positioned beneath said orifice and displaced laterally therefrom, an electrometer means sensitive to electrical charges induced on said conductive element and producing an output pulse in response to said each of said charges, a scaling circuit an input coupled to said electrometer circuit and producing an output signal in response to a selected number of output pulses from said electrometer means, and relay means connected to the output of said scaling circuit, said relay means actuating said electrically driven means to position a succeeding one of said collector vessels beneath said orifice upon receipt of an output signal from said scaling circuit.

5. In product collector control apparatus for use with a fractionating column, the combination comprising a plurality of collector vessels, a movable carrier for sequentially positioning each of said vessels under the outlet orifice of said column, a motor operatively coupled to said carrier, a voltage source connected to said column for charging product globules forming at the output of said column, a conductive plate of substantial area disposed in a vertical plane beneath said column outlet and to one side of a vertical axis therethrough, an electrometer connected to said conductive element and producing an output signal upon the detection of a charge thereon, a monostable multivibrator coupled to the output of said electrometer and producing a time extended signal in response to a signal from said electrometer, a relay having normally open switch contacts and having a driver coil for closing said contacts in response to said time extended signal from said multivibrator to initiate rotation of said motor, and a microswitch connected between said power supply and said motor and positioned to interrupt operation thereof after a fixed travel of said carrier whereby said collector vessels are successively positioned beneath said orifice.

6. In apparatus for collecting fluid product from the outlet of a chemical processing column of the class having a plurality of collector vessels supported on a movable carrier for successive positioning beneath said outlet, the combination comprising an electrical motor operatively coupled to said carrier, a normally closed microswitch having a trip lever positioned to periodically open said microswitch during operation of said motor, a power supply coupled to said motor through said microswitch, a relay having normally open switch contacts connected between said power supply and said motor in parallel with said microswitch and having a driver coil for closing said switch contacts, a gate circuit connected across said power supply in series relationship with said relay driver coil, said gate circuit having a gate signal input for initiating conductance through said driver coil, a D.C. voltage source coupled to said column outlet for applying

a charge to product globules forming at said outlet, a conducting element disposed between said column outlet and said carrier, said element being spaced slightly from a vertical axis passing through said outlet, a charge detector circuit having an input coupled to said conducting element and producing an output signal in response to a charge thereon, a scaling circuit having an input coupled to said charge detector circuit and producing an output pulse in response to a selected number of output signals therefrom, and a multivibrator circuit having an input coupled to said scaling circuit and having an output coupled to said gate circuit signal input whereby said motor is actuated for a limited period following the passage of a selected number of fluid droplets by said conducting element.

7. Apparatus for separately collecting successive portions of fluid product from the outlet of a chemical separation column comprising, in combination, a horizontal circular rack rotatably disposed beneath said column outlet, a plurality of collector vessels mountable on said rack around a circle thereon which is concentric with the axis of rotation thereof whereby rotation of said rack successively positions said collector vessels under said outlet orifice, an electrical motor having a drive shaft and having linkage coupling said shaft to said rack for rotating said rack, a voltage source coupled to said column and applying an electrostatic charge to said outlet thereof whereby product globules forming at said outlet are charged, a conductive upright plate disposed beneath said orifice and aside from a vertical axis therethrough, an electrometer circuit coupled to said conductive plate and producing an output pulse in response to an electrical charge on said plate, a relay having normally open switch contacts and having a driver coil for closing said contacts, an electrical power supply coupled to said motor through said relay contacts, means temporarily energizing said relay driver in response to said output pulses of said electrometer circuit to initiate operation of said motor, a microswitch coupled between said motor and said power supply in parallel with said relay contacts, and a cam coupled to said motor shaft and positioned to close said microswitch for a fixed amount of rotation of said shaft whereby said motor is continued in operation for an interval sufficient to position a successive one of said collector vessels at said column outlet.

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