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COLUMN HEAD FOR BATCH FRACTIONATING COLUMNS

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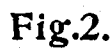


Fig.1.

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2 Sheets-Sheet 2

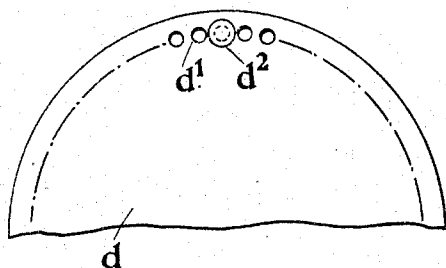


Fig. 4.

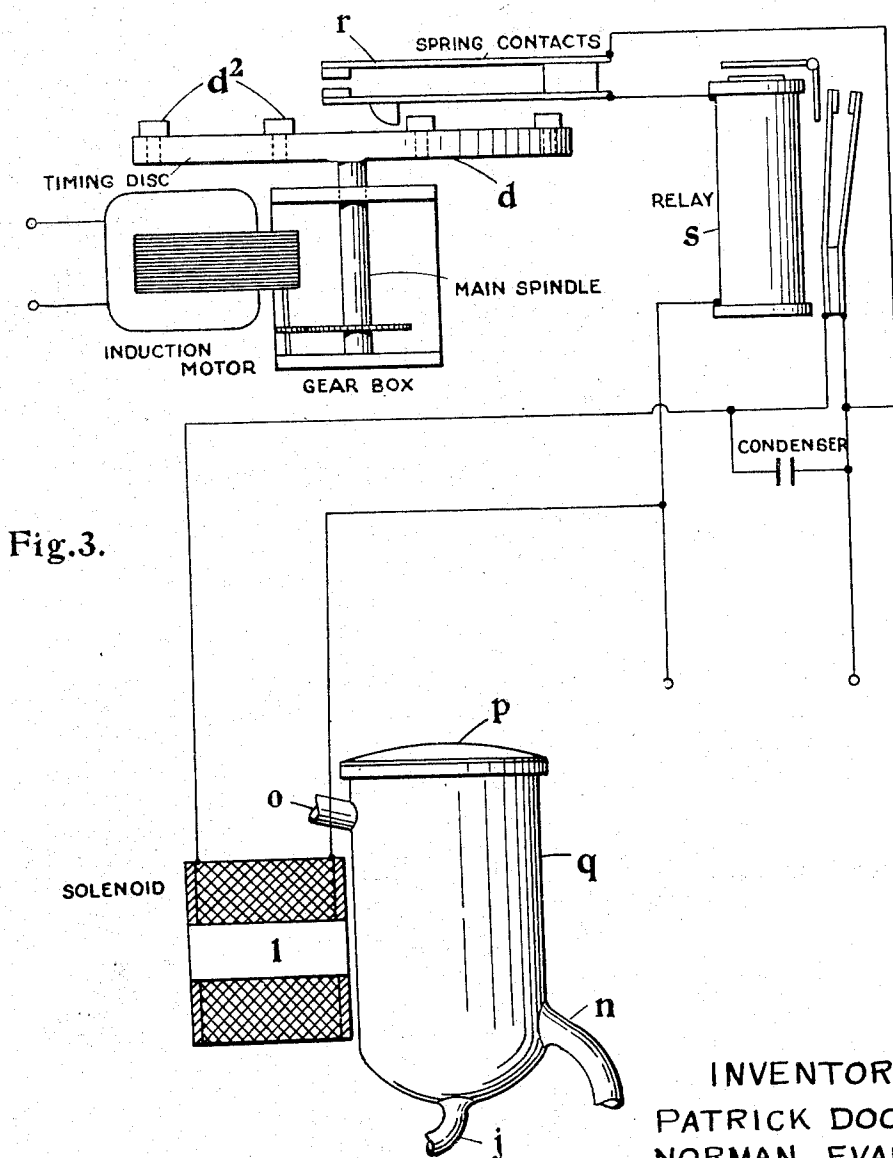


Fig. 3.

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COLUMN HEAD FOR BATCH FRACTIONATING COLUMNS

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9 Claims. (Cl. 202—163)

This invention relates to column heads for batch fractionating columns.

In batch fractionating columns the functions of a column head are respectively to measure the overhead temperature, to condense the reflux and, if a total condenser be used, the distillate, to remove the distillate at the required off-take rate, and to return the reflux to the column. When it is desired to fractionate under conditions of precision, the off-take rate will in general be small, and the reflux rate will necessarily be correspondingly higher. Under these conditions it is essential that the column head should have as low a hold-up of liquid as possible. Difficulty has been experienced in designing satisfactory column heads to meet this requirement, and to ensure a constant and easily controlled rate of take off. The problem thus presented is particularly important in laboratory apparatus. The invention is however of more general application.

According to the invention a take-off device is provided in the column head and comprises a small container or bucket adapted to be filled with condensate, the contents being discharged into the distillate receiver at determined intervals. This may for example be done by means of an electro-magnet which on being energised causes the container or bucket to be tipped for discharging its contents into the distillate receiver. The intervals at which the magnet is energised may be controlled by a clockwork mechanism or other means.

The invention comprises the constructions hereinafter described with reference to the accompanying drawings.

The invention is diagrammatically illustrated by way of example in the accompanying drawings as applied for laboratory use, in which,

Figure 1 is a sectional elevation of a column head equipped according to the invention, the condenser and the column being omitted.

Figure 2 is a detail view of the bucket equipment employed.

Figure 3 is a diagram of a timing mechanism that may be employed for the periodic tipping of the bucket, and

Figure 4 is a detail part view of the rotary disc of the timing mechanism.

In carrying the invention into effect the column head comprises a lower part *a* (Figure 1) adapted to be set in the upper part of the column (not shown), and from the part *a*, the main limb *c* of the column head rises, and near its upper end is connected by transversely extending and downwardly inclined tubing *c*² with a smaller

limb *b* of the column head, the upper end of which limb is connected to a condenser.

The limb *c* of the column head is surrounded by a vacuum jacket *c*¹ advantageously having silvered internal surfaces, which guard against heat loss and serves as a radiation shield for the thermometer, which is accommodated within that part of the limb *c* extending upward beyond the tubing *c*², the bulb of the thermometer being conveniently set in the position indicated at *d*. Thus the vapour from the column passes upward through the rising main part *c*, and films of liquid are deposited within the downwardly inclined tubing *c*² and drain into the cooler *b*, which comprises a cooling jacket *e* through which cooling water may be circulated, whereby the deposited films of liquid are cooled well below the boiling point of the liquid in order to avoid inaccuracies that might result from possible re-vaporisation, and the liquid leaves the cooler through the spout *g*.

The liquid flowing through the spout *g* is thus cooled well below its boiling point, as otherwise a certain amount of vaporisation would occur in the casing *q* into which the liquid thence passes, which would allow an undeterminable quantity of distillate in the form of vapour to flow out from the casing *q* through the outlet pipe *n*. It is however necessary that the cooler *e* should act in the manner described as a film cooler, and that the spout *g* should be filled with air and not vapour, as otherwise the cooler would act as a condenser, and discharge a hot condensate. To ensure the operation of the cooler *e* as a film cooler, a small septum is interposed at *f* in the tubular limb *b* of the column head below the passage *c*², whereby an annular space of a small and uniform width is provided of the order of 1 millimeter (1 mm.) between the peripheral edge of the septum and the tube permitting the condensate to flow through without accumulating a liquid head above the septum, but sufficiently narrow to be self-sealing by surface tension. Under these conditions the presence of air in the cooler is ensured by venting it into the casing *q*, through the vent hole *o* in the spout *g*, while the casing *q* is itself vented to atmosphere through the outlet pipe *n*, whose bore is sufficiently wide to ensure that in operation it is not choked with liquid passing to the receiver.

The liquid passes from the cooler *e* through the spout *g* within a casing *q* into a bucket *h* mounted therein and provided with an inlet spout *h*¹ and an outlet spout *h*², which are so positioned that when the bucket is full there is a con-

tinuous stream of liquid through the bucket from inlet to outlet. The bucket h is pivotally supported at i^1 within open vertical slots provided at the upper end of a pair of vertical supports i , disposed at the respective opposite sides of the bucket h , and the bucket and supports are accessible for adjustment within the casing q by removal of the cover-plate p . The supports i are mounted upon a cylindrical base part i^2 , which together with the supports are advantageously made of brass, the base part i^2 being adapted to fit within a cylindrical part q^1 of glass fused at a number of positions at its lower edge to the base of the casing q when the latter is made of glass: the lower part of the cylindrical flange q^1 being provided with an aperture or apertures such as q^2 through which the distillate may discharge on its accumulation in the bottom of the casing q , through the outlet spout j , that discharges through the part a of the column-head into the column. By such means the whole of the support can be rotated to bring the bucket h below the spout g .

Thus the liquid passes from the spout g , through the bucket h and from the output spout h^2 it flows over the outer surface of the bucket and into the outlet pipe j , whereby it is returned to the column as reflux, or through the outlet pipe n .

The bore of the outlet tube j is advantageously contracted or constricted towards its lower outlet end to form at the end an orifice of small diameter in order that the constricted bore near the end may be self-sealing by surface tension, whereby the admission of vapour from the column into the casing q may be avoided.

The bucket itself may advantageously be made of a non-magnetic material such as brass, and a counterweight k for example of mild steel may be suspended beneath it on a downwardly depending rod k^1 (Figure 2), while an electro-magnet l may advantageously be mounted outside the casing q in which case the wall of the casing is made of some non-magnetic material such as brass or glass. Thus when the electro-magnet l (Figure 1) is energised the counter-weight k is attracted by the electro-magnet l , and the bucket thus tips through a right-angle, whereby its contents are discharged into the wide mouth n^1 of the outlet pipe n into the distillate receiver. The upper inlet end n^1 of the outer pipe n has a wide mouth that is adapted for the reception of the liquid discharged from the bucket h on its being tilted, and the outlet pipe n for the main part of its length is of a diameter sufficiently large to avoid any possibility of its being choked with liquid. Thus the outlet pipe n may also serve the purpose of venting the casing q to the atmosphere, the presence of air in the cooler e being ensured by venting it into the casing q through the vent hole o on the spout g .

The form of the bucket h itself may be varied widely so long as certain conditions are observed to ensure its effective operation. Free surfaces tend to be undefined owing to the vagaries of surface tension, with consequent variation in the volume of liquid contained in the bucket. For this reason the bucket h should be formed so that it has comparatively small free-surfaces. The bucket should not therefore have an open top. To ensure accurate operation it has been found that the top h^3 of the bucket should be sloped upwards towards the outlet h^2 at a small angle such as 8° . By such means all air is swept out of the bucket h , with the further advantage that the

top h^3 does not easily permit of a pool of liquid forming upon it, and possibly being tipped into the outlet pipe n . The tendency for the liquid to run backwards over the top of the bucket h instead of spilling straight down the side, may lead to appreciable inaccuracies which may be minimized by the provision of a suitably shaped outlet spout. Thus when the outlet spout h^2 is disposed to make an angle of about 45° with the horizontal this tendency is avoided. Again in order to give a clear sweep through for purging the bucket, the inlet h^1 may advantageously be carried down to the bottom of the bucket h and the outlet h^2 taken from the top of the opposite side as indicated in Figure 2, and at a substantial angle to the base such as 60° .

In order to ensure that even when the bucket h is full the liquid will continue to flow through it, the bucket h may be formed so that the top of the inlet h^1 is at a higher level than the top of the outlet h^2 .

The bucket h may for example contain about 1.5 ml. of liquid. If the boil-up rate of the column be 1800 ml./hr. and operation is intended at a reflux ratio of 40:1, the distillate off-take rate will be 45 ml./hr. Thus the bucket will have to be tipped 30 times per hour. This is easily accomplished by a simple clock mechanism actuating the electro-magnet through a relay.

The means described thus provide an intermittent off-take, it being understood that immediately the bucket h has righted itself, after being tilted and emptied, it is filled again with distillate, and during this short period the reflux rate in the column drops. As soon as the bucket h is full the column runs under total reflux and continues to do so until the bucket is again tipped, and then rights itself. It has been found that there is no objection to this short periodic stoppage of reflux at the column head as the time during which the stream is cut off is small, and for this reason it is preferred to use a bucket of small capacity tipping fairly frequently rather than one of larger capacity tipping less frequently.

In operation there will normally be a sufficient capacity for liquid in the upper part of the column to ensure that the column is not seriously starved of reflux during the period in which the bucket is filling. On the other hand if a bucket of large capacity be used, the time taken before reflux again begins to flow may be long enough to allow the column to drain, and thus seriously affect its operation. Although there is also a correspondingly longer time under total reflux, in which to re-establish its operation, it has been found desirable to avoid too considerable a fluctuation in the actual operation of the column.

The timing mechanism may take a number of forms and advantageously is of simple construction such as diagrammatically indicated in Figures 3 and 4. When it comprises the use of a solenoid l this should be energised at regular intervals, and it should remain energised to ensure effective drainage of the bucket h , and advantageously means provided for variation in the intervals of time at which the solenoid l is energised.

Satisfactory operation is secured in the use of a synchronous motor or clockwork mechanism whereby a disc such as d is rotated at constant speed, the disc near its periphery being provided in circular series at equal intervals with a large number of holes d^1 , (Figures 3 and 4) for example sixty. Pegs d^2 may be placed in any determined number of these holes at regular inter-

vals, the pegs being so set that they trip a switch *r* by which a relay *s* is energised, which in turn energises the solenoid *l*. The pegs *d*² are so designed that the switch *r* is tripped for a sufficiently long period to allow effective drainage of the bucket *h*. The interval advantageously should be a minimum of one second. There is however no need for the time to be accurately determined as long as the minimum is exceeded. Such a mechanism has the advantage of being flexible since by suitable adjustment of the number of pegs *d*², the switch *r* may be made to operate from one up to sixty times per revolution, thus giving a wide range of reflux ratios.

The electro-magnet *l* is advantageously mounted with its horizontal axis in line with the bucket pivot pins. If it be too great a distance from the casing *q* containing the bucket, the latter will not properly tip. If on the other hand it be too close, the tipping action is apt to be too vigorous.

The adjustment of the distance of the electro-magnet so as to give the necessary force may be made by placing it fairly well away from the wall of the casing *q* (with the end of the pole piece about one-half an inch from the wall of the casing) and gradually moving inward the electro-magnet until the tipping is satisfactory. The adjustment should be made with the bucket full of liquid, and the final adjustment of position of the electro-magnet is best carried out when the column is running under total reflux.

We claim:

1. A column-head for batch fractionating columns, comprising a film-cooler having a condensate discharge outlet, a liquid "take-off" device including a closed bucket having an inlet and an oppositely disposed outlet respectively extending upward and outward from the bucket, and a depending counter-weight, the bucket inlet being normally disposed beneath said cooler outlet to receive a stream of condensate discharging therefrom, the bucket and counter-weight being adapted to be tilted as a system on a substantially horizontal axis disposed transversely beneath and between the inlet and outlet of the bucket, on the bucket being filled with condensate, and means for tipping the bucket and discharging the contents of the bucket through the bucket outlet, whereupon the bucket-counterweight system returns to normal position, in which the bucket inlet is disposed beneath the discharging stream of condensate from said cooler.

2. A column head for batch fractionating columns, comprising a casing, a film-cooler having a condensate discharge pipe terminating within the upper end of said casing, a liquid "take-off" device within said casing including a closed bucket having an inlet and an oppositely disposed outlet respectively extending upward and outward from the bucket, and a depending counterweight, the bucket inlet being normally disposed beneath said cooler discharge pipe in position to receive a discharging stream of condensate therefrom, a supporting structure within said casing upon which said bucket and counterweight are pivotally mounted for tilting as a system on a substantially horizontal axis disposed transversely beneath and between the inlet and outlet of said bucket, on the bucket being filled with conden-

sate, an outlet pipe for said casing disposed beneath said bucket in diametrically opposite position to the bucket inlet for receiving the contents of said bucket when the latter is tilted and for conducting said contents from said casing, means for tilting said bucket on said axis to discharge the contents of said bucket through the bucket outlet into said outlet pipe, and a drain pipe for returning that part of the condensate falling to the bottom of said casing as reflux to the column.

3. A column-head for batch fractionating columns, as specified in claim 2, in which the counter-weight is of a magnetic metal, the enclosing casing within which the "take-off" device is mounted and the bucket are of non-magnetic material, and which includes an electro-magnet disposed externally of the said casing in lateral position adjacent the counter-weight and near the wall of said casing, and means for controlling the intervals at which the electro-magnet is energised for attracting the counter-weight and tipping the bucket, whereby its contents are discharged through an outlet from said casing.

4. A column-head for batch fractionating columns, as specified in claim 2, in which the film-cooler comprises a cooling jacket operative to cool the condensate well below its boiling point before the condensate is discharged into the bucket of the "take-off" device.

5. A column-head for batch fractionating columns, as specified in claim 2, in which the presence of air in said film-cooler is ensured by venting it into the casing in which the liquid "take-off" device is mounted by a vent hole in the discharge pipe through which cooled condensate from the cooler passes into the said casing, the said casing being itself also vented to atmosphere through said outlet pipe having a bore of large diameter.

6. A column-head for batch fractionating columns, as specified in claim 2, in which the bore of the pipe through which the cooled liquid from the cooler passes into the casing in which the "take-off" device is mounted, is constricted towards the lower end, whereby it is self-sealed by surface tension.

7. A column-head for batch fractionating columns, as specified in claim 2, in which the bore of the drain pipe through which the liquid condensate passes as reflux to the column, from the bottom of the casing in which the "take-off" device is mounted, is gradually constricted towards its outer end whereby it is self-sealed by surface tension.

8. A column-head for batch fractionating columns, as specified in claim 2, in which near the upper end of the cooler a septum is set leaving between the peripheral edge of the septum and the cooler tube a narrow annular space, whereby the accumulation of a head of liquid above the septum is avoided, the annular space being sufficiently narrow to be self-sealing by surface tension.

9. A column-head as specified in claim 2, in which the narrow annular space is of the order of one millimeter in width.

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