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(54) **Burette.**

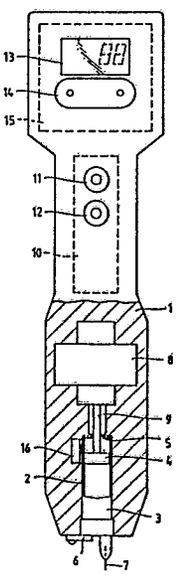
(57) The invention provides a burette having a body 1 adapted to receive at one end a releasable reagent container 3 having a removable end fitting with a cannula 7 such that the cannula 7 ruptures a self-re-sealing diaphragm of the reagent container 3 when the end fitting is mounted on the reagent container 3, and that the diaphragm is allowed to re-seal when the end fitting is removed.

The burette also includes an electric linear actuator comprising a stepping motor 8 and a reciprocable drive shaft 9, and includes a re-chargeable battery 10 with power to the stepping motor 8 controlled by a push button 11.

A further push button 12 re-sets a digital display means 13.

An interface socket 14 allows the burette to be connected to an external controller such as a microcomputer, and may serve as a power input connector for battery charging.

Fig.1.



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Description

"BURETTE"

The present invention relates to a burette which can be used for on-site titrimetric analysis, for example monitoring water quality.

Mechanically actuated burettes are known, for example from US Patent No. 4,086,062 issued 25 April 1978 to Clifford C. Hach, in which an actuator is manually operated to drive a plunger in a direction in which it expels liquid from the burette. The quantity of liquid dispensed can be indicated by way of a digital counter which records the number of turns of a rotor member serving to drive a linear actuator which engages the plunger.

An electrically operated pipette is also known, from EP-A-0 152 120 (Rainin Instrument Company) in which a stepper motor drives the pipette plunger in both an extension direction for dispensing liquid from the pipette and a retraction direction for taking up liquid into the pipette, again with a digital counter to indicate the displacement of the plunger and hence the quantity of liquid taken in or dispensed. EP-A-0 152 120 discloses the possibility of several different sizes of dispensing chambers for use with the same drive module in order to allow different quantity ranges of liquid to be handled in the pipette.

It is a disadvantage of the electrical pipette system of EP-A-0 152 120 that the liquid cannot safely be stored in the pipette and hence if on-site analysis is to be carried out it is necessary to take containers of the respective reagents and the pipette itself on-site so that, as required, liquid can be taken up into the pipette and then dispensed for the titrimetric analysis.

It is an object of the present invention to overcome the disadvantages of the prior art system and to provide a burette which is particularly convenient for use in on-site titration.

Accordingly, one aspect of the present invention provides a burette comprising a housing, means on the housing for receiving a releasable reagent container including a displaceable plunger and a discharge passage, linear actuator means for driving the plunger in a reagent discharge direction, and display means responsive to displacement of the linear actuator for indicating the dispensing travel of the plunger, wherein the removable reagent container includes a self-re-sealing diaphragm and means for piercing the diaphragm for dispensing of reagent from the container and for allowing the diaphragm to re-seal at termination of dispensing.

It is particularly convenient if the burette in accordance with the present invention can be equipped with interface means allowing the motorised burette to be controlled from a programmed or programmable controller which will effect standard titrimetric analysis according to a pre-determined programme.

Thus, a second aspect of the invention provides a burette including an electrically operated linear actuator arranged to drive a liquid-dispensing plunger in a liquid-dispensing direction to eject liquid from a discharge passage of the burette, power control means for energizing the linear actuator for displacement through known incremental distances during dispensing of the liquid, and interface means for connecting the burette to an external programmable or programmed controller for operating the burette in accordance with a predetermined programme for automatic titration operations.

In order that the present invention may more readily be understood the following description is given, merely by way of example, with reference to the accompanying drawings in which:-

FIGURE 1 is a schematic view of one embodiment of burette in accordance with the present invention;

FIGURE 2 is a block diagram of the electrical units incorporated in the pipette of Figure 1; and

FIGURE 3 is a side elevational view of the reagent container and cannula support of Figure 1.

The burette illustrated in Figure 1 comprises a main housing 1 which has at its front end a recess 2 for a pre-packed container 3 including a reagent to be used during titrimetric analysis for on-site water monitoring. A plunger 4 within the reagent container 3 is held in place by a flange 5 so that even when the container 3 is outside the burette body 1 the plunger will be held in situ.

The reagent container 3 is held in place by means of a clip 6 on the front end of the body 1, positioned so as not to obstruct a cannula 7 through which reagent is to be dispensed during analysis.

The dispensing of reagent from the container 3 is achieved by means of a stepping motor 8 co-operating with a linearly displaceable shaft 9 so that the stepping motor and the shaft together form a linear actuator. The shaft 9 is of a cross-sectional shape, for example splined, which prevents it from rotating and hence it is subject solely to linear reciprocation generated by way of a screw (not shown) within the rotor of the stepping motor 8.

The housing 1 further includes a rechargeable battery 10 to provide power to drive the stepping motor 8. On the exterior of the recess for the battery 10 are two control buttons 11 and 12 whose function will be described later.

At the extreme rear end of the burette housing 1 is a digital indicator 13, for example an LCD indicator, and an interface socket 14 allowing the burette to be connected by means of a suitable multi-pin plug to a battery-charging power supply and/or to a programmable or programmed controller (not shown), for example a microcomputer. This controller is then able to instruct the burette to carry out automatically pre-programmed analysis sequences, if desired.

As with any conventional burette, the burette shown in Figure 1 will normally be operated with the cannula 7 at the lower end and the display 13 uppermost where it can be viewed by the operator.

In the then uppermost end portion 15 of the housing 1 are various control circuits whose func-

tions will be described with reference to Figure 2.

It is a particularly important characteristic of the present invention that the reagent container 3 be of a self-sealing type so that the reagent in the container will be kept safe against inadvertent dilution or loss until such time as dispensing is required. Normally there will be some air trapped within the reagent container in order to allow for expansion of the liquid reagent. In such cases where the reagent is oxygen-sensitive, the container 3 may be topped up with an inert gas such as nitrogen which will of course remain trapped within the container during storage and transport of the containers and during use of the burette.

In order to guard against inadvertent operation of the linear actuator comprising the stepping motor 8 and the shaft 9, a switch 16 in the reagent container recess 2 is tripped only when a reagent container 3 is in place.

The block diagram shown in Figure 2 employs the same reference numerals as Figure 1 for those components which are shown in both drawings, but additionally illustrates the burette control circuit 17 and a burette driver circuit 18 which generate the appropriate pulses to drive the stepping motor 8.

As can be seen from Figure 2, the battery 10 has its output branched to drive the display 13, the control circuit 17, and the burette driver circuit 18. Equally, as mentioned above, it has an input from the interface 14 to allow for recharging of the battery when desired.

The interface 14 has two further outputs which join up with the output lines from the control buttons 11 and 12 so as to provide inputs to the control circuit 17. The button 11, and the appropriate by-passing output from the interface 14, causes the control circuit 17 to generate a stream of control pulses to the driver circuit 18 which in turn generates power pulses to the motor 8.

The push button 12 and the appropriate output from the interface 14 provides a means of resetting the digital display 13 to zero.

The push button 12 has an additional function in association with the control circuit 17 in that the control circuit 17 is such that when the two push buttons 11 and 12 are depressed simultaneously (or the corresponding outputs of the interface 14 are energised simultaneously) the control pulses emitted by the control circuit 17 and the corresponding drive pulses emitted by the driver circuit 18 cause the stepper motor 8 to cycle rapidly for advancing the drive shaft 9 into engagement with the plunger 4 of, for example, a newly inserted reagent container 3 to prime the burette. As soon as the operator sees a quantity of liquid expelled from the cannula 7 he releases the two push buttons 11 and 12.

Once this priming operation is complete, the operator then pushes button 12 alone in order to reset the display 13 to zero and then when dispensing of reagent from the container 3 during titrimetric analysis is required the operator presses button 11 alone, causing the stepper motor 8 to cycle slowly. When the operator releases the button 11 the stepper motor stops. Each pulse generated by the control circuit 17 is recorded on the digital display 13 and is delivered to any remote programme controller connected to the burette via interface 14. Each such pulse corresponds to a known fixed volume of liquid dispensed from the cartridge.

Preferably the concentration of the chemical reagent used in the container 3 is related to the incremental quantity of liquid dispensed per control pulse from the control circuit 17 such that the value visible on the digital display 13 at the end of the titration procedure corresponds to the concentration of the analyte being determined by titration, for example expressed in milligrams per litre.

Advantageously the burette includes pulse-emitting means to deliver, via the interface socket, pulses proportional to the volume of liquid dispensed.

As a preferred optional characteristic of the burette, the control circuit includes means for counting the number of control pulses emitted from the fully retracted position of the shaft 9, for example from the instant of installation of a fresh reagent container 3 tripping the switch 16, and the counter triggers the control circuit to cease to deliver pulses when the number of pulses counted is equivalent to travel of the shaft 9 to its limit position in which the plunger 4 has run out of travel. Alternatively some mechanical sensing means may be incorporated for signalling to the control circuit 17 when the plunger 4 is fully forward. In this way it is possible to ensure that no damage to the drive transmission occurs at full travel position.

A further optional characteristic is that when the reagent container 3 is removed, and the switch 16 tripped to its alternative position, the control circuit automatically instructs the driver circuit 18 to drive the stepper motor 8 in the reverse direction until the drive shaft 9 is fully retracted. When the shaft 9 reaches this rearward limit of travel, the contents of the counter register are reset to zero and power is disconnected from the driver circuit 17 leaving the burette in its quiescent state ready to receive a reagent container.

Figure 3 shows in more detail the reagent container 3 and its end fitting carrying the cannula 7. If desired, a short length of small bore plastic tubing may be slipped over the end of the cannula 7 to facilitate dispensing reagent without splashing.

As shown in this drawing, the reagent container 3 has its plunger 4 provided with a recess 19 intended to accommodate the air bubble when the container 3 is in the operative configuration, and hence the inadvertent dispensing of air along with the reagent liquid is avoided.

The end of the reagent container 3 opposite the plunger-retaining flange 5 is closed by a screw cap 20 which supports an end extension 21 on which an end fitting 22 is secured by way of a bayonet fastening means 23. Although not shown in Figure 3, the end fitting 22 includes a part of the cannula 7 having a sharpened point 24 which pierces a self-sealing diaphragm 25 all of which are shown in Figure 4. When the end fitting 22 is snapped in place by operation of its bayonet fastener 23, the sharpened tip 24 of the cannula is automatically driven

through the diaphragm 25 to pierce it. When, on the other hand, the end fitting 22 is released from the screw cap 20 the cannula tip 24 is withdrawn from the diaphragm 25, leaving the diaphragm to re-seal, and also allowing a neoprene sheath 26 on the cannula 7 to extend into the Figure 4 position to maintain the cannula tip 24 safe against pollution. It will of course be understood that when the cannula tip 24 pierces the diaphragm 25 the neoprene sheath 26 yields concertina-fashion to expose the cannula tip 24 to the diaphragm.

The diaphragm 25 is a plug of natural "india" rubber having a thickness of between 2.0 and 2.5 mm and subjected to radial compression on insertion, in order to enhance its self-sealing properties.

As indicated above, the provision of the interface 14 allows the illustrated burette either to be used as a hand-held on-site digital burette for titration, or to be used as part of a fully automated titration apparatus when linked to an appropriate controller such as a microcomputer. Starting from the above-mentioned quiescent state, when the drive shaft 9 is fully retracted and the drive circuit 17 de-energised, one complete sequence of operation of the burette for titrimetic determination of total alkalinity in water will now be described.

A 25 cm^3 aliquot of the water to be analysed is placed in a small flask or beaker, and the water is then dosed with one or two drops of BDH 4.5 indicator, a proprietary chemical indicator solution which undergoes a colour change at pH 4.5 to cause the solution to become blue in colour.

A pre-filled reagent container 3 containing 0.0618 $mol.dm^{-3}$ sulphuric acid is then inserted into the burette housing 1 and is clipped in place by operation of the clip 6 of Figure 1.

The priming procedure described above, under which the drive shaft 9 is advanced rapidly by simultaneous depression of the two push buttons 11 and 12, is carried out to bring the drive shaft into engagement with the plunger 4 of the reagent container, and to ensure that the liquid/air interface in the container 3 reaches the discharge end of the cannula 7.

The operator then slowly dispenses the acid from the container 3 into the sample flask, by depressing the push button 11, while agitating the sample in order to ensure thorough mixing. The operator continues to add acid in this way until the blue colour of the indicator just changes to a pale orange/yellow.

At this point the value shown on the digital display 13 of the burette is noted, and by virtue of the specially chosen gearing of the stepper motor 8 and the dimensions of the reagent container 3, related to the above-mentioned concentration of the sulphuric acid in the container 3, the value displayed corresponds to the total alkalinity of the water sample expressed in $mg.dm^{-3}$ $CaCO_3$.

Once this titrimetric analysis is complete, it is possible either to use the burette immediately for similar titrimetric analysis on a further sample of water, or to remove the end fitting 22 (Figures 3 and 4) with the cannula to cause the self-sealing diaphragm 25 to re-seal and the neoprene sheath 26 to recover the tip 24 of the cannula 7. The burette can then be transported to a fresh site for further titrimetric analysis with the same reagent container 3 or be stored for some future use.

When the next titrimetric analysis with the same reagent container 3 is required, the end fitting 22 is first of all re-attached to re-open the diaphragm 25, and then the button 11 is depressed to cause the stepper motor 8 to drive the plunger 4 forwardly until the end fitting 22 (Figures 3 and 4) and the cannula 7 are once again filled with reagent.

Once the container 3 has been depleted of reagent solution, the container can be removed by release of the clip 6, whereupon the switch 16 will be released to trigger the control circuit 17 to reverse the stepper motor 8 in order to retract fully the shaft 9 and to return the burette to its quiescent state.

In addition to the particular example of titrimetric analysis mentioned above, various other titrimetric determinations can be carried out in an analogous manner, using a suitable combination of reagent and indicator for each particular analyte to be determined, and preferably using a reagent concentration related to the incremental volume of liquid dispensed so as to give a direct read out, as in the above example.

Other applications for the invention, for example when used in conjunction with a remote-controlling device, may include the performance of fully automatic titrimetric analysis, the intermittent or continuous dispensing of bio-chemical reagents in luminescence assays of biological material, and the intermittent or continuous dispensing of calibration standards or samples or reagents into chemical sensing systems for industrial process monitoring.

The advantages of the burette in accordance with the present invention over the prior art apparatus for titration include :-

1. The liquid reagents can be provided in sealed disposable plastic cartridges which self-re-seal after use, thereby simplifying greatly the handling of the reagent and eliminating the dangers of chemical spillage. In practice the diaphragm 25 can re-seal satisfactorily at least 20 times, so a considerable number of separate titrimetric analysis operations can be carried out on one reagent container 3 before it is eventually empty and needs to be disposed of.

2. The ability to have a direct read-out of analyte concentration (by appropriate relationship of the reagent concentration to the rate of dispensing of the reagent) considerably simplifies the operator's task of titrimetric analysis.

3. The ability to pre-pack the reagents with reduced concentration as compared with those employed in the past ensures that the operator in the field is much less at risk to inadvertent dispensing of the reagent onto the skin or other body tissue.

4. The ability to connect the burette to a remote controller such as a microcomputer, using the interface 14, considerably extends the versatility of the burette.

5. The ability to recharge the battery by way of

the interface 14 adds to the convenience of operation in the field.

Claims

1. A burette comprising a housing for reagent to be controllably dispensed by the burette, a displaceable plunger, a discharge passage, and linear actuator means for driving the plunger in a reagent discharge direction, characterised by means (6) on the housing (2) for receiving a releasable reagent container (3) which includes the plunger (4) and the discharge passage (7); and by display means (13) responsive to displacement of the linear actuator (8, 9) for indicating the dispensing travel of the plunger (4), the removable reagent container including a self-re-sealing diaphragm (25) and means (22, 24) for piercing the diaphragm for dispensing of reagent from the container and for allowing the diaphragm to re-seal at termination of dispensing.

2. A burette according to claim 1, characterised in that the diaphragm piercing means comprise a releasable end fitting (22) having attached thereto a diaphragm piercing member (24) communicating with said discharge passage (7) whereby fastening of the end fitting in place on the reagent container automatically pierces the diaphragm, and removal of the end fitting from the reagent container allows the diaphragm to re-seal.

3. A burette according to claim 2, characterised in that the diaphragm-piercing member (24) comprises a discharge cannula of the reagent container end fitting, said cannula forming the discharge passage.

4. A burette according to claim 3, characterised in that the cannula (24) carried by the reagent container end fitting includes a resilient sheath (26) which is automatically retracted upon diaphragm piercing and will automatically extend upon release of the cannula from the diaphragm.

5. A burette according to any one of the preceding claims, characterised in that the linear actuator (8) is electrically powered and the burette includes power control means (18) for driving the linear actuator in both directions of travel of the plunger.

6. A burette according to claim 5, characterised in that the burette further includes a rechargeable battery (10) connected to the power control means (18).

7. A burette according to either of claims 5 and 6, and further characterised by a switch (16) responsive to the presence of a reagent container (3) in the burette body (2), and control means (17) responsive to said switch for driving the linear actuator to retract upon removal of a reagent container, until the actuator arrives at a fully retracted configuration.

8. A burette according to claim 7, characterised in that the control means (17) further de-energises the linear actuator automatically at its fully retracted end of travel position.

9. A burette according to any one of claims 5 to 8, further characterised by interface means (14) allowing the burette to be controlled from a remote programmable or programmed control means.

10. A burette including an electrically operated linear actuator (8) arranged to drive a liquid-dispensing plunger (14) in a liquid-dispensing direction to eject liquid from a discharge passage (7) of the burette, and power control means (18) for energizing the linear actuator for displacement through known incremental distances during dispensing of the liquid, characterised by interface means (14) for connecting the burette to an external programmable or programmed controller for operating the burette in accordance with a predetermined programme for automatic titration operations.

11. A burette according to claim 10, further characterised by a rechargeable battery (10) for powering the linear actuator (8) and energizing the power control means, the interface means (14) comprising a multi-pin plug and socket connector having means for re-charging the battery (10) from outside, and conductors for connecting the power control means (18) to a remote programmable or programmed controller.

12. A method of titrimetric analysis using a burette according to any one of claims 1 to 9, characterised by selecting the concentration of reagents in the reagent container such that the quantity of reagent dispensed per unit of the value displayed on the display means allows the displayed value to be taken as the concentration of the appropriate analyte in a sample.

13. A method according to claim 12, characterised in that the titrimetric analysis is for measurement of total alkalinity of water, and the reagent is sulphuric acid; and in that the water sample is first of all dosed with a coloring indicator which indicates the alkalinity of the sample and which responds to attainment of a pH value of 4.5 during neutralisation of the alkalinity upon gradual dispensing of the acid reagent.

Fig. 1.

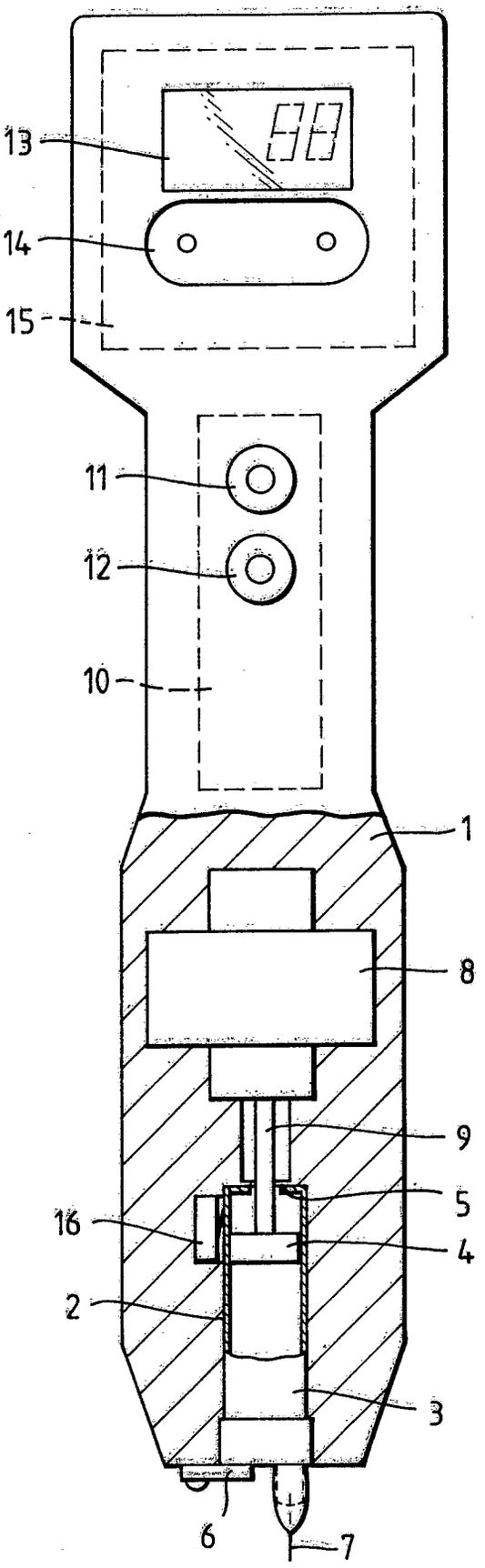

Fig.2.

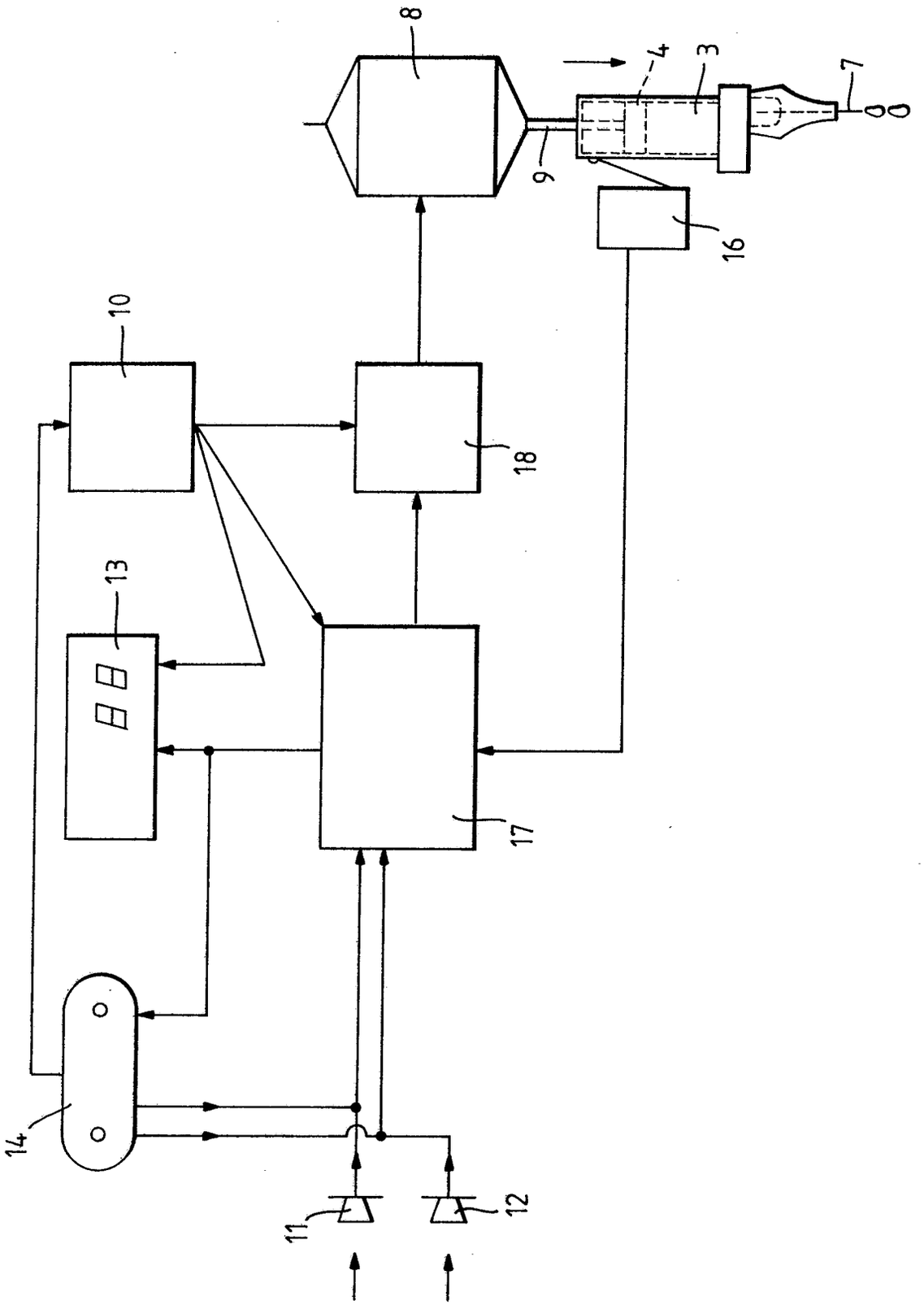

Fig. 3.

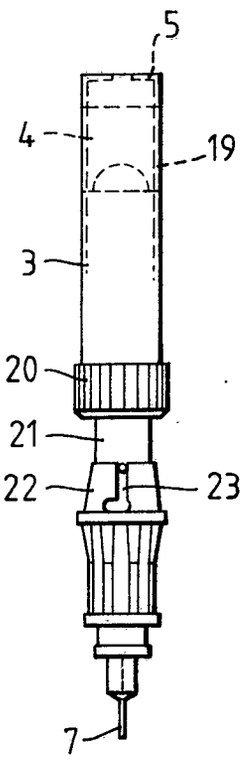

Fig. 4.