Volumetry. A little theory

CRISON has more than 25 years' experience in the manufacture of potentiometric titrators which has given us in-depth knowledge of the kind of instruments required for precise handling of liquids.
In labs the continual demand for increased productivity means that routine tasks need to be automated as far as possible, such as:
- Preparing solutions.
- Repetitive reagent dispensing.
- Dilution of standard solutions.
- Titrations with indicator.
- Reagent pipetting.

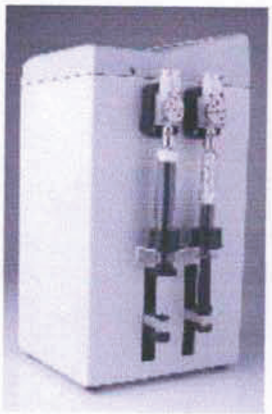
CRISON's contribution in this field has been to offer a high-quality range of precision burettes at reasonable prices. These burettes fill the gap between manual digital dispensers and expensive motorised precision burettes.
The new range comprises:
- The Burette 1S, a single-syringe burette.
- The MultiBurette 2S, a 2-syringe burette.
- The MultiBurette 4S, a 4-syringe burette.

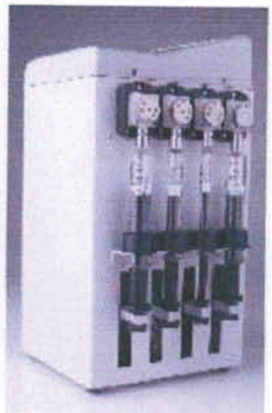
These models all come in two different versions, according to use:
- D, with Dispense function, titration and auto-calibration.
- DPD, with Dispense, Pipette and Dilute function, and titration and auto-calibration.
- Customised Burettes, based on the models described above, which can be adapted to each client's particular requirements.

Remote control
By operating the Multi-Burettes from a PC, the operator can perform multiple and simultaneous dispensings.

Burette 1S

Essential components
There are three essential components to the burette:
- High-resolution plunger displacement mechanism, actioned by a stroke-by-stroke motor.
- The precision syringe, which can be of different volumes, depending on the client's requirements.
- Instantly switcheable reagent input and output electrovalve. This is a great improvement over motorised valves, which are much slower.

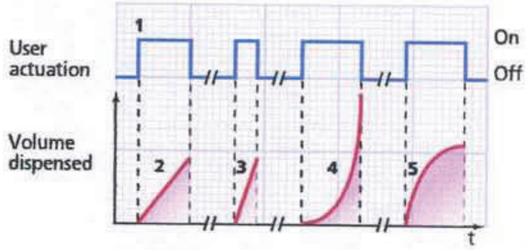




Multi-syringe burettes
In the 2S and 4S Multi-Burette, the two or four syringes are actioned via a single mechanism.
The syringe plungers can remain free or be attached to the cursor.
The cursor in movement drags the plungers of the attached syringes.
Only one reagent is dispensed: the one from the valve which has been activated.
The other syringes will return the reagent to the original bottle. This makes it easy to handle different reagents with different programmes via a single module.

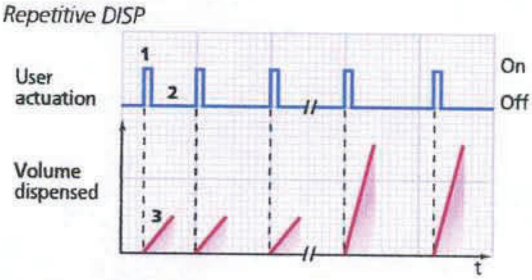
Functions
A summary of the various possibilities offered by the new range of CRISON burettes is given below.

Dispensing
Manual DISP



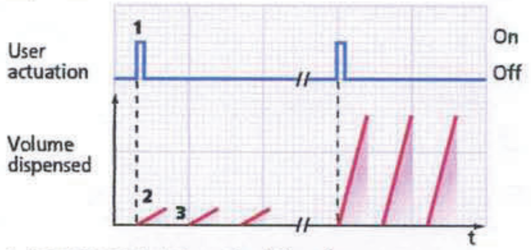
1- Dispensing occurs whilst the user presses the corresponding key.
2- Slow, constant dispensing.
3- Fast, constant dispensing.
4- Variable dispensing speed, from slow to fast.
5- Initial speed fast, end speed slow.

Repetitive DISP



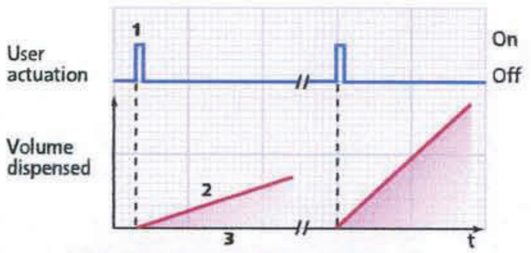
1- A simple keystroke causes the previously programmed dispensing to be performed.
2- The time between the various dispensings is decided by the user.
3- The flow and volume dispensed are constant.

Sequential DISP



1- A single keystroke starts a series of dispensings.
2- The volume of the dispensings can be programmable and repetitive.
3- The time between additions is constant. It must be programmed.

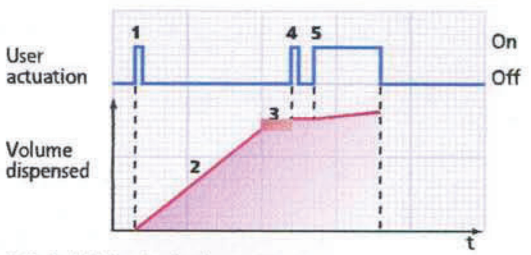
Cumulative DISP



1- A single keystroke starts the dispensing process.
2- The volume dispensed is as programmed.
3- The length of time of dispensing is as programmed.
4- Other options: Select a flow for a certain length of time or select a volume with a fixed flow rate.

Titration

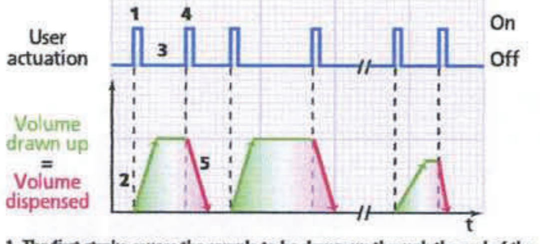
TITR



1- Stroke initiating the titration process.
2- Initial addition, maximum dispensing flow.
3- Automatic addition to the pre-selected flow rate.
4- Stroke to stop automatic addition.
5- Continual stroke until titration finalises.

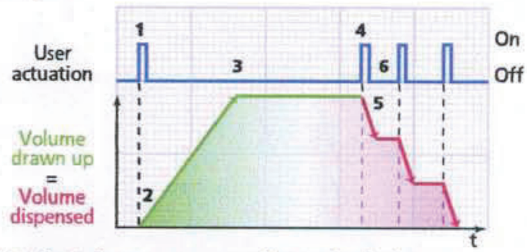
Pipetting

Simple PIP



1- The first stroke causes the sample to be drawn up through the end of the outlet tube.
2- Volume drawn up, programmable.
3- The time between the draw-up and dispensing is decided by the user.
4- The second stroke causes the sample to be dispensed.
5- The volume dispensed is equal to the volume drawn up.

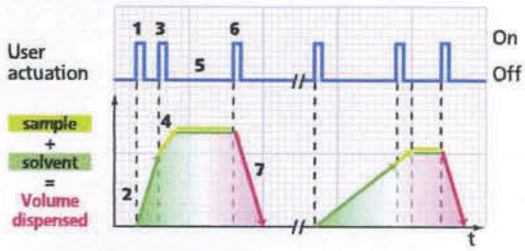
Multiple PIP



1- The first stroke causes an amount of the sample to be drawn up.
2- Volume drawn up.
3- The time between the draw-up and dispensing is decided by the user.
4- Stroke which starts the first partial dispensing.
5- Volume dispensed. As per programme.
6- The time between the various partial dispensings is decided by the user.

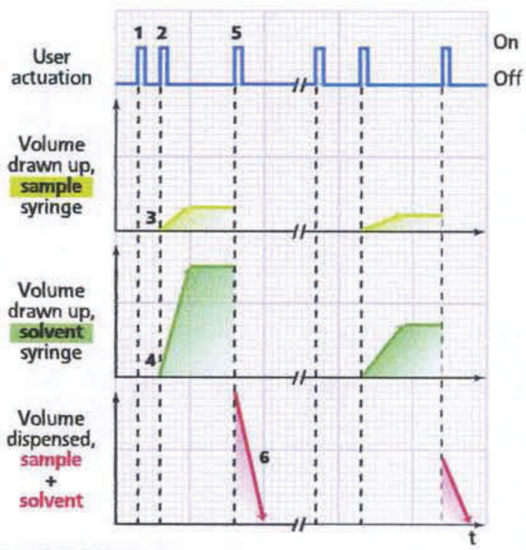
Dilution

DIL with one syringe



1- Stroke initiating the process.
2- The solvent is automatically loaded from the corresponding bottle and positioned.
3- Stroke which causes the sample to be drawn up through the end of the outlet tube.
4- Volume drawn up.
5- The time between drawing up the sample and dispensing is decided by the user.
6- Stroke which causes final dispensing.
7- Dispensation of the sample plus the solvent.

DIL with two syringes



1- Stroke initiating the process.
2- Stroke which causes the sample to be drawn up through the end of the outlet tube and corresponding loading of the solvent from the bottle.
3- Volume of sample drawn up.
4- Volume of solvent drawn up.
5- Stroke causing final dispensing with the two syringes.
6- Total dispensing, sample plus solvent.

Titration. A little theory

Titration is the quantitative determination of a substance when it reacts to a known substance, a titrant reagent.

$$aA + bB \rightleftharpoons cC$$

Together with gravimetry it is one of the oldest known methods of analysis.
Titration is used as a standard method of quantitative analysis.
The advantages over other techniques are:
- It is a direct method.
- It is exact and reproducible. Under optimal conditions, better than 0.1 %.
- Can be automated. Both for analysing a single sample or a whole series.

Potentiometric titration

The classical method of indication has been observing how an indicator changes colour. These days, the most frequently-used method of indication is the potentiometric method.
The electrode used will depend on the reaction to be monitored, pH, metallic, ion selective, etc.

The current potentiometric titrator

By combining the various elements, this instrument performs potentiometric titrations automatically.

Essential components

- Sensors: pH, redox, ISE etc. electrode
  Temperature probe (ATC).
- Process measurer-monitor.
- Burette(s) for adding the titrant reagent or other reagents.
- Stirrer: Magnetic or rod.

Handy complements

- A printer for registering results.
- A computer for storage and later processing of data.
- Auxiliary peristaltic pumps, either for prior treatment of the samples or to increase the degree of automation.
- Automatic sample changer, to automate the consecutive analysis of several samples.

Classic titrations

End point titration

The instrument adds reagent until a previously-selected pH or mV value is attained.
This method is generally applied in norms for analysis where an end-point value is specified or when adapting a method with an optical indicator.

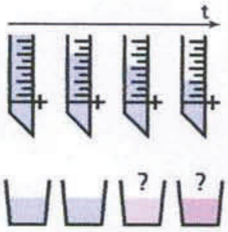


Figure A

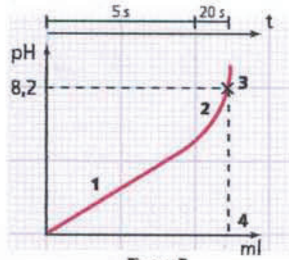


Figure B

Fig. A. Titration with optical indicator, phenolphtalein.
Example: Standardisation of HCl with NaOH.
Observations:
The volume of the titrant reagent consumed must be very high.
The point at which it "turns" is high subjective.
Short titration time for experts.

Fig. B. Potentiometric PF titration.
Example: Standardisation of HCl with NaOH.
1- Fast initial addition.
2- Slow addition in the approximation zone.
3- Exact determination of the volume at the end point.
4- Low level of reagent consumption.

Location of equivalence points

The instrument analyses the variation in potential generated by the successive additions of the titrant reagent on the sample, and using these data it locates the equivalence point.
This type of titration can be used to study unknown samples, to determine the components of a sample, to determine one or more equivalence points, etc.

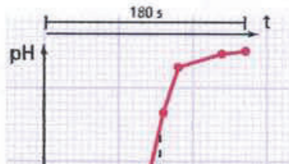


Figure A

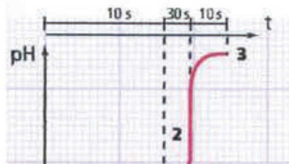


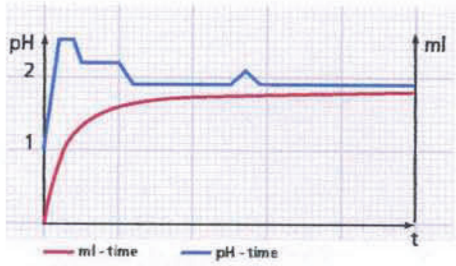
Figure B

Fig. A. Manual potentiometric titration
Example: Determination of chlorides in water.
- Long analysis time.
- Curve not well defined. Difficult to locate inflection point.

Fig. B. Automatic potentiometric titration. Location of inflection point.
Example: Determination of chlorides in water.
1- Fast initial addition.
2- Slow addition, for perfect definition of the inflection area.
3- Titration ends after locating the inflection point. This means saving reagent and time.

pH-stat

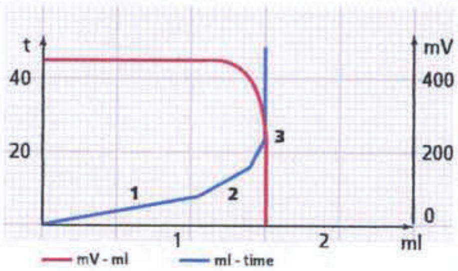
This is a variation on end point titration, applied to kinetic analysis.
The information on the kinetics of the reaction can be obtained from the amount of titrant reagent consumed, in relation to the time.



Example: Monitoring an antiacid.
The automatic procedure includes:
- Pre-titration to neutralise the substrate.
- Stabilisation of the medium.
- Introduction of the sample and commencement of the process.
- Evolution of the pH.

Karl Fischer Titration

This is an end-point titration adapted to the specific Karl Fischer reaction, to determine the water content of a substance.
The detection system used in this case is bipotentiometry. Based on the measurement of potential, applying a constant current between two platinum electrodes.



Example: Monitoring the humidity of a solvent.
1- Fast initial addition until a significant drop in potential occurs.
2- Addition at average speed.
3- Slow-speed addition, through to the end.

Why use an automatic titrator?

Quality and safety

These days, a titrator is not only an instrument for performing titrations. It is a necessary tool for testing labs who require a certain quality and need to follow GLP's.

Productivity

The high degree of automation provided by a complete automatic titration system immediately translates into significant time savings for the user and the rapid obtainment of results.

Economy

The use of high-precision burettes means that consumption can be greatly reduced, both as regards the sample and the reagents.
The automatic titrator frees the user from tedious, routine jobs, allowing him/her to take on more creative tasks.