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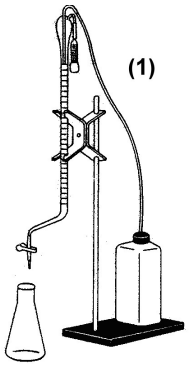
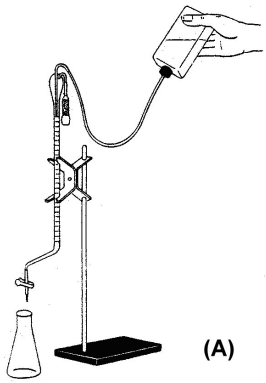
Getränkeanalytik

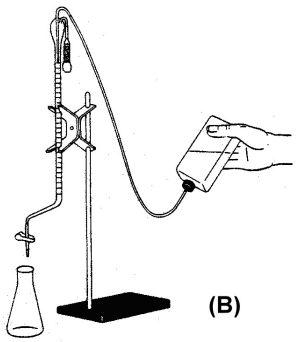
Information on the working equipment for analysis methods according to Dr. REBELEIN

version 10/2005





Burette filling in this way (falling pressure)



or in that way (finger pressure)

(1) AUTOMATIKUS burettes:

TTS (with titration ball, titration clamp, silicone hose, Teflon tip) and TTG (as before, however with rubber hose instead of silicone hose) are supplied for:

10 : 0,1 ml with 10 mm outside Ø
20 : 0,1 ml with 10 mm outside Ø
25 : 0,1 ml with 12 mm outside Ø
50 : 0,1 ml with 14 mm outside Ø

The reaction solutions are filled from supply bottles (preferably 500 ml poly bottles) into the burettes by falling pressure (A) or by compressing the bottle with the opening directed downward (B). Place down bottle as soon as the rising liquid has reached the uppermost mark. The liquid exceeding the zero mark flows automatically back into the supply bottle; the meniscus adjusts automatically to the zero mark. Check the quality of the zero-point adjustment after each burette filling!

The automatic burette filling in the closed system protects the solutions and facilitates the work. Storage bottle cleaning problems are thus avoided. After having emptied a supply bottle, simply connect the next bottle with new solution is to the burette.

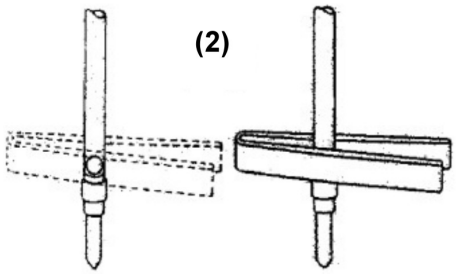
Shake the supply bottles briefly before refilling the burettes. Why? Due to the differences in temperature, the liquid contained in a closed bottle forms after some time condensation water at the walls not being covered by the liquid. If this condensation water is not mixed with the solution before the removal, a slight change in concentration will be the consequence.

Automatic burettes should after each titration be refilled up to the zero mark. It is thus avoided that salts crystallise out after the evaporation of the wetting film at the inner walls of the burettes, preventing at the same time that the titration precision is affected when these salts dissolve after refilling the burettes later.

As soon as the forming of drops at the inner surfaces of the burette can be observed (non-uniform wetting), a through inner cleaning will be required. Use the commercially available cleaning agent for laboratory equipment and follow the supplier's working instructions.

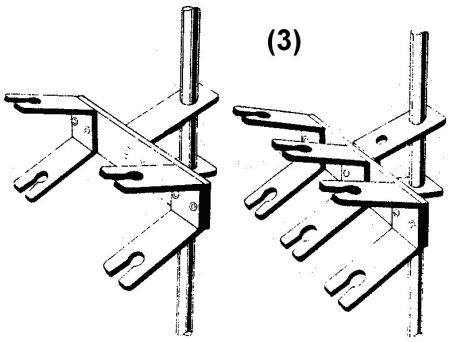
The most advantageous method is to add the cleaning liquid by means of a 500-ml poly bottle screwed onto the connection line to the burette. Rinse in the same way with distilled water.

The titration burettes for the sugar and alcohol determination have a graduation for 0 - 30 g sugar and 0 - 120 g alcohol respectively (the meniscus of the receiver solutions must before titration be exactly on the division mark 30 g sugar and 120 g alcohol respectively!), but they have only a reading range of 0 - 28 g sugar and 0 - 110 g alcohol respectively. Why? The oxidation solutions have indeed a capacity of 30 g sugar/l or 120 g/l alcohol, but since a chemical reaction depends on the concentrations of its participants and the concentration ratios become more unfavourable the closer the fringe to the reaction range, the reaction process in the fringe area is slower than normally. To avoid any moment of uncertainty (retarded reaction), the fringe ranges are not evaluated when determining the sugar and alcohol content, for the reason of which the evaluation ends at 28 g sugar/l and 110 g/l alcohol respectively, i.e. values that are safe under the standardised procedure conditions. Beverages with higher sugar and alcohol contents are diluted (see working instructions).



(2) Titration ball – titration clamp – burette valve:

This easily exchangeable valve consists of a hose piece with a Teflon delivery tip, the titration ball inside the hose and the titration clamp placed around the hose outside at the level of the titration ball. When pressing the clamp jaws together, the hose enclosed in the titration ball expands towards the side that is not under pressure, and the liquid within the burette can exit via the pass-through lock. Neither the rough nor the fine dosing represents for an inexperienced person any problems.



(3) Automatikus burette holders:

are available for stand rods of 12 mm ∅ (figure after the slash) in simple version (for 1 burette), double version (for 2 burettes), and triple version (for 3 burettes):

simple	10/12	12/12	14/12
double	10-10/12	12-12/12	14-14/12
trible	10-10-10/12	12-12-12/12	14-14-14/12

The figures before the slash represent the burette ∅ in mm. Any combination from 10 mm - 12 mm - 14 mm for 2 or 3 burettes is available, but always with stand rods of 12 mm Ø.

Assembly: Press stand rod – or burette holder – slightly together, insert stand rod from below into the unslotted holes of the stand rod holder, push the burette from the top into the slotted holes of the burette holder. For adjusting the burette, press the burette holder with one hand slightly together, moving the burette with the other hand in the required extent up or down. Adjust the stand in height after having pressed the stand rod holder with one hand together.

Stop watch:

The digital stop-watch gives an acoustic signal after the expiration of the set time. It is provided with three buttons to which the following functions are allocated:

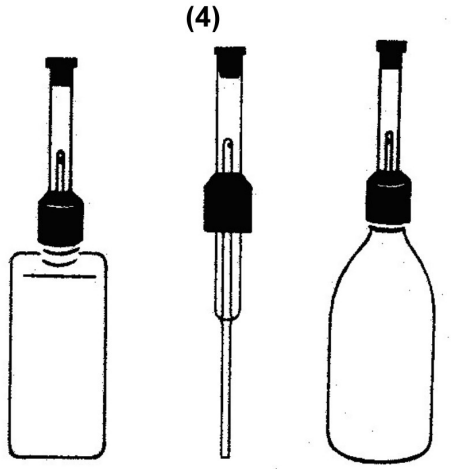
- „min" and "sec" for setting the desired period of time
- „start/stop", to start the countdown and/or to stop the acoustic signal

To reset the set time to 0, press "min" and "sec" buttons simultaneously.

(4)-(6) Dosing cylinder:

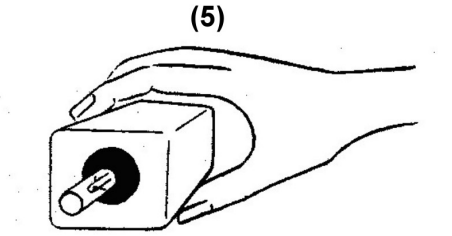
(4) Glass dosing cylinders 2 ml - 5 ml - 10 ml - 12 ml and
(6) Poly dosing cylinders 10 ml with intermediate 5 ml mark

are destined for the approximate measurement of equivalent volumes from 500 ml and 1000 ml poly bottles.



The dosing cylinders for the reaction solutions are mounted in such a way that the polyethylene hose reaches all the way down to the bottom of the bottle after having screwed the dosing cylinders onto the reagent bottles.

Fill the liquid into the dosing cylinder by pressing the supply bottle together until the level slightly exceeds the liquid exit opening of the ascending pipe. The liquid being above the opening flows spontaneously back into the bottle as soon as the pressure on the bottle is released, the nominal volume remaining in the dosing cylinder is used for the addition described in the respective analysis instructions (5).

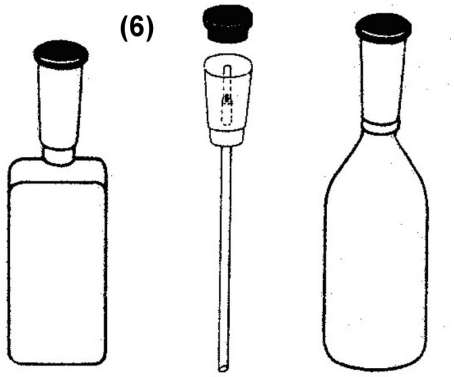


Hold rectangular bottles preferably at opposite corners for emptying the dosing cylinders.

Plugs and caps respectively protect dosing cylinder inside from contamination and have to be removed when using the dosing cylinder and to be attached again after use.

Aggressive liquids can also be filled with loosely attached cap to avoid splashing when pressing the bottle together.

The dosing cylinders can also be used to measure simple or multiple volumes. If e.g. the approximate addition of 20 ml of a liquid is required, the 10-ml dosing cylinder is filled and emptied twice.



The poly dosing cylinder permits a measuring in 5 ml-steps. If for example the approximate addition of 25 ml of a liquid is required, the liquid is filled twice up to the 10-ml mark and once up to the 5-ml mark.

The dosing cylinders have from time to time to be cleaned thoroughly. Insert the dosing cylinders after cleaning only into the reagent bottles when they are completely dry.

Laboratory glass:

Laboratory glass calls for a thorough care. Special laboratory glass cleaner is used for its cleaning (follow the cleaning instructions). The impeccable condition of the burettes, pipettes and volumetric flasks is required. The cracking of the uniform wetting film and the starting of drop formation at the inner surfaces of burettes are indications for the fact that they have to be cleaned.

Infrared laboratory heater:

To ensure standardised heating powers, before use a heating-up phase of 5 minutes has to be adhered to. The infrared laboratory heater is completely *maintenance-free*.

Disposal of the REBELEIN reagents

according to Dr. R. Miltenberger

1. Formula for the precipitation of chromium after the alcohol determination:

- Collect titrated alcohol solutions occurring from the alcohol determination according to REBELEIN;
- Add to 1 litre of titrated solution carefully (using safety goggles!) approx. 200 ml of caustic soda lye (32%) in portions (50 ml each);
- Check the pH value, that should be at around pH 7 – 8, by touching gently a litmus paper or using a pH meter;
- Observe colour change from dark blue via turquoise to a light blue grey. A voluminous $Cr(OH)_3$ deposit occurs;
- Allow deposit to settle over night;
- Filter deposit by means of a coarse filter, whereby the filtrate being drained must be clear;
- **Check for chromium** in the filtrate by adding some drops of hydrogen peroxide to the filtrate sample in the test tube:

  Full yellow = chromium exists
  Slightly light yellow = no chromium exists

- Mix filtrate, if applicable, with filtrate from the sugar determination according to REBELEIN in the ratio 3:1 (pH value nearly neutral) and dispose of it;
- Collect filter paper with blue grey chromium hydroxide residues from the drying chamber and dispose of it with the hazardous waste.

Chemical reagents:

Caustic soda solution 32 % highest grade, Merck Nr. 5587
Hydrogen peroxide 30 %, highest grade, Merck Nr. 8597
Litmus paper blue, Merck Nr. 9486
Coarse filter, Nr. 604½, ∅ 150 mm, Schleicher & Schüll

2. Formula for the precipitation of copper after the sugar determination:

- Collect titrated alcohol solutions occurring from the sugar determination according to REBELEIN;
- Add to 1 litre of the collected solutions 20 ml of hydrochloric acid and 0.5 g of thioacetamide (safety goggles!);
- Heat preparation in the outlet up to the boiling point and allow it then to cool down;
- Observe colour change from violet blue via white (after having added thioacetamide) to black (after boiling);
- Allow deposit to settle over night, filtering it then by means of a folded filter. The filtrate must be clear, a slight yellow colouring is possible;
- **Check for copper** in the filtrate by adding some drops of ammonia to the filtrate sample in the test tube:

  Dark blue colouration = copper in the filtrate
  Other colouration = no copper exists

- Dry filter paper with black copper sulphide (a drying chamber can be used), collect it and dispose of it with the hazardous waste.
- Mix filtrate, if applicable, with filtrate from the alcohol determination (after precipitation of chromium) in the ratio 1:3 and dispose of it.

Chemical reagents:

Hydrochloric acid 32 % highest grade, Merck Nr. 313
Thioacetamide p.A., Merck Nr. 8170
Ammonia 25 %, highest grade, Merck Nr. 5422
Filter Nr. 595 ½, ∅ 150 mm, Schleicher & Schüll