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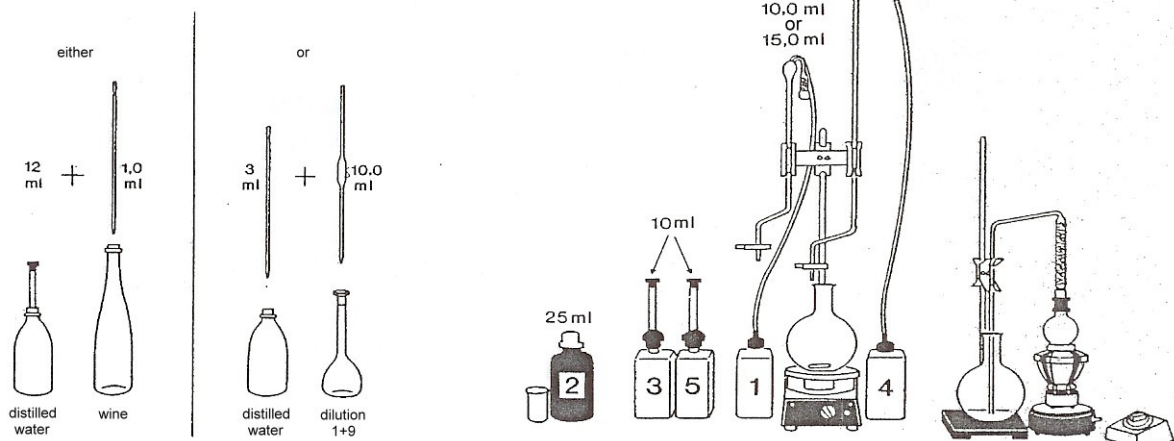
Getränkeanalytik

Determination of alcohol according to Dr. Rebelein

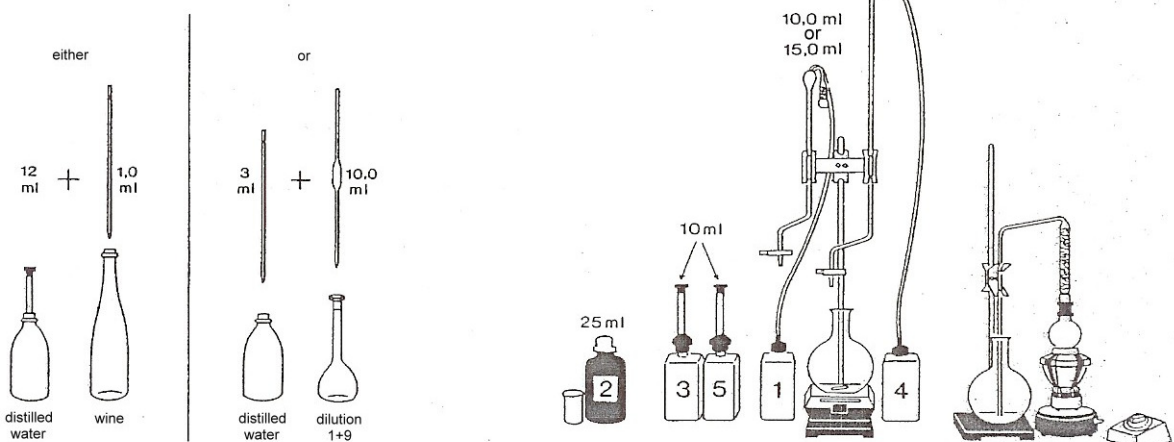
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Equipment with lum stirrer



Equipment with mini-stirrer



Guidance for determination of alcohol in wines:

(For the determination of alcohol in spirits, that means liqueurs, brandy etc...., please see special information sheet!)

- Heat laboratory burner for 5 minutes.
- Check burettes. The meniscus must be on a level with the top mark of the burette. Remove drops from burette jets. Fill up dose cylinders.
- Let 10,0 ml of "Alcohol 1" slowly flow into a reaction flask (500 ml flat bottomed flask) wipe off drop on burette tip on inside wall of flask.
- Pour "Alcohol 2" into 50 ml glass beaker up to 25 ml ring mark and transfer into 500 ml reaction flask (Caution: 65% nitric acid, analytical grade, Merck) and place reaction flask under distillation tube.
- Pipette liquid to be analysed into distillation flask (100 ml flat-bottomed flask NS 19/26). (Consider the chapter "Preparation of sample")
- Swivel distillation flask onto reflector burner and lower distillation tube down to the bottom of the reaction flask. If required raise flask by putting aluminium disks underneath.
- Set alarm clock to 4 minutes and distil.
- After distillation raise distillation tube by 5-10 cm and swivel tube by 180° to the side, take off distillation flask, and rinse outlet of distillation tube well with distilled water into reaction flask.
- Fill up reaction flask approx. to 300 ml mark with distilled water, simultaneously rinsing the inside wall of the flask.
- Put in magnetic pin and place the flask on the stirrer. Switch on stirrer/illumination.
- Pour 10 ml of "Alcohol 3" into reaction flask (dose cylinder).
- Titrate with "Alcohol 4" until the initial red-brown colour tends to yellow/ yellow-green. Now interrupt titration, add 10 ml of "Alcohol 5" (dose cylinder), and continue titration carefully drop by drop till the deep blue colouring changes into a water-brightly blue. The dripping distance should last amount to 3 to 5 seconds.
- Read alcohol content (g/l) off burette. Example: The meniscus reads 78,2. The examined liquid contains 78,2 grams of alcohol per litre.

- Checking the titration: the titration was correct, if the titrated liquid turns again to dark blue through the addition of one drop of "Alcohol 1". The liquid was over titrated if several drops are necessary to obtain a change of colour.

In the case of alcohol contents up to 110 g/l (13.93 Vol. %) this instruction is applied.

If an alcohol content between 110 and 170 g/l (13.93 - 21.54 Vol. % is expected, 15.0 ml instead of 10.0 ml of "Alcohol 1" are used. The addition of nitric acid ("Alcohol 2") remains the same (25 ml). Generally 60.0 g/l of alcohol must be added to the read-off titration rates.

Example: 15.0 ml of "Alcohol 1" were used, 75.8 g/l of alcohol were titrated. The beverage contains $75.8 + 60.0 = 135.8$ g/l of alcohol (17.20 Vol. %).

In the case of beverages with more than 170 g/l (21.54 Vol. %) of alcohol, the order developed particularly for spirits applies.

Preparation of sample:

There are two possibilities for the selection:

1. Combine 12 ml of distilled water (dose cylinder), 1 levelled spatula of pumice, 1-2 drops of silicone anti-foam solution, and 1,0 ml of the sample in distillation flask and connect this immediately with the silicone stopper of the distillation tube.
2. Combine 3 ml of distilled water (measuring pipette), 1 levelled spatula of pumice, 1-2 drops of silicone Anti-Foam solution and a 1 + 9 dilution of sample/distilled water (blowing out pipette), fill up flask with distilled water and connect this immediately to the silicone stopper of the distillation tube.

Making of a dilution 1 + 9:

- pour 10-20 ml of distilled water in 100 ml measuring flask,
- pipette 10,0 ml of the sample (blowing out pipette),
- fill up flask to ring mark with distilled water (meniscus must be exactly on a level with ring mark), and mix.

Correct pipetting technique:

(Rinse pipette first with the liquid to be pipetted!) Suck liquid approx. 2 cm above ring mark, dry pipette with cellulose cloth, adjust to the mark. In doing so, place the tip of the pipette onto the inside wall of a glass beaker specially supplied for this purpose and wipe the last droplet off the tip. The contents of the pipette are draining without loss with the tip of the pipette slightly immersed in the water. After the draining of the liquid the tip is placed onto the inside wall of the flask approx. 2 cm above liquid level. After about 15 seconds blow pipette out and wipe tip of pipette.

Blank Titration for Checking the Solutions:

Pour 10.0 ml of "Alcohol 1" and 25 ml of "Alcohol 2" into 500 ml reaction flask, top up with distilled water to 300 ml mark, add 10 ml of "Alcohol 3", titrate with "Alcohol 4" to yellow/ yellow-green, add 10 ml of "Alcohol 5", continue titration to water-brightly blue. At a tolerance of + 0.5 g/l the blank titration should produce an alcohol content "0 g/l".

Water Test by Blank Distillation:

The quality of the distilled water used should also be checked from time to time. For this purpose distil 12 ml of the used distilled water instead of the liquid to be analysed and titrate the distillate of the water. If the blank titration results without distillation differ distinctly from the results with distillation, the distilled water is not perfectly.

Determination of Alcohol in Fruit Juice:

If an exact determination of small quantities of alcohol in fruit juices is required, 3 ml of distilled water (measuring pipette) and 10.0 ml of the undiluted sample (blowing out pipette) instead of 12 ml of distilled water and 1.0 ml of the sample are used, and the result is divided by 10. Example: an alcohol rate of 48.2 g/l is titrated. The fruit juice contains $48.2 : 10 = 4.82$ g of alcohol per litre.

Determination of Alcohol in Fermented Fruit and Corn Mash:

For the determination 1 ml of blank mash liquid or 10 ml of a blank dilution 1 + 9 mash liquid / distilled water is used. The result in g/l is read off the titration burette. The real content of alcohol is found out tabularly (1 g of alcohol = 0.1267 Vol. %). In order to determine the alcohol content of the original mash the alcohol content found for the mash liquid must be reduced by the empirical rate of the share of draff and residue (3 % in the case of corn, 6-8 % in the case of apples, 8-10% in the case of pears, 11-12 % in the case of plums, 15 % in the case of cherries).

Determination of Alcohol in Beer:

In the case of Pilsner, lager, and bitter beer 2.0 ml of decarbonized beer are used for the examination. The rate read off the burette has to be halved. In the case of stout beer the normal quantity (1.0 ml of decarbonized beer) is used. The rate read off the burette is valid. Hence the comprehended range of the titration burette reaches in the case of 2.0 ml of sample up to 55 g of alcohol per litre (approx. 5.5 weight %), in the case of 1.0 ml of sample up to 110 g of alcohol per litre (approx. 11 weight %).

Notes concerning the Determination of Alcohol according to Dr. Rebelein:

The alcohol driven over into the oxidation mixture is immediately oxidised quantitatively to acetic acid. The remaining oxidising agent is titrated back with sodium thio-sulphate. The genuine alcohol content of the liquid can be read off the burette.

Acetic acid and sulphurous acid do not influence the determination. (In the case of the simple alcohol determination method without neutralisation the volatile acids passing on to the distillate are included.)

In the case of series-determinations the distilled water can be put in as many distillation flasks as required. However, the sample to be examined must be put in only immediately before the distillation. After the pipetting of the sample into the distillation flask the flask should be connected to the distillation tube immediately.

The magnetic stirrer is working properly when a deep whirl is forming in the liquid (usually when set to half speed). In case the magnetic pin should start to jump, interrupt titration and switch off the stirrer. Do not restart the stirrer before the magnetic pin has come to rest. Continue titration only after the full stirring action can be observed again.

Safety instructions:

It is advisable to wear safety goggles when working with the 65% nitric acid ("Alcohol 2"). Nitric acid leaves a temporary yellow colour on the skin which-however harmless- can be avoided by immediately rinsing the acid off with water.

While handling the solutions R and S-sentences on the back labels of them are to be considered. Further references are to be inferred from the safety data sheets.

Determination of alcohol according to Dr. Rebelein - parts list -

- 1 laboratory burner
- 1 laboratory alarm clock
- 1 Lum stirrer with thread M 10 in the adapter, staff 600 x 12 mm, 2 magnetic pins 4 cm, 1 magnetic pin grip arm (the lum stirrer with staff is also the stand for burettes)

alternatively

- 1 mini-stirrer with 2 magnetic pins 4 cm, magnetic pin grip arm
- 1 Stand with staff 600 x 12 mm for burettes (necessary, if no lum stirrer is ordered)
- 1 burette 20 ml TTS for „alcohol 1“
- 1 burette „alcohol according to Dr. Rebelein“ TTS for „alcohol 4“
- 1 holder for burettes 10 – 10 / 12
- 1 Stand with aluminium staff 600 x 12 mm for distillation tube
- 1 alcohol distillation tube (with glass beads) with silicone stoppers 21 / 16 x 25 mm
- 1 holder for distillation tube
- 2 blowing out-pipettes 1 ml
- 1 blowing out-pipette 5 ml
- 2 blowing out-pipettes 10 ml
- 1 measuring pipette 12 ml
- 1 dose cylinder 12 ml with round polyamide bottle 500 ml for distilled water
- 2 flat-bottomed flask 100 ml NS 19/26 with thermic protection (distillation flask)
- 2 flat-bottomed flask 500 ml with marking on 300 ml (Reaction flask)
- 1 Beaker high form 50 ml with Ringmark 25 ml for „alcohol 2“
- 2 dose cylinder 10 ml for „alcohol 3“ und „alcohol 5“
- 1 aluminium disk 130 x 130 x 2,5 mm for Levelling the burner/reaction flask
- 1 5-litre-bottle for distilled water with pinchcock/air cleaner for distilled water
- 5 litre distilled water in the can
- 1 polyamide bottle 500 ml for distilled water
- 1 x pumice stones for analysis (Riedel-de-Haen)
- 1 spatula for pumice stones
- 1 x 100 ml silicone Anti-Foam solution for distillation
- 1 measuring flask 100 ml for dilutions
- 1 map with information-material
- 1 „Tables for wine analytics“ by Dr. Schmitt
- 1 board „Conversion of the alcohol from g/l in Vol.%“

original allocation of equipment of reagents:

Need per analysis:

- | | |
|---|-------------------------|
| <input type="checkbox"/> alcohol 1 500 ml in polyamide bottle | (10,0 ml / burette) |
| <input type="checkbox"/> alcohol 2 1000 ml in glass bottle | (25 ml / beaker) |
| <input type="checkbox"/> alcohol 3 500 ml in polyamide bottle | (10 ml / dose cylinder) |
| <input type="checkbox"/> alcohol 4 500 ml in polyamide bottle | (variable / burette) |
| <input type="checkbox"/> alcohol 5 500 ml in polyamide bottle | (10 ml / dose cylinder) |

recommendable accessories:

- eye protector
- Dispenser 25 ml for dosage of 65 % Nitric acid directly from 2,5 Litre Merck-original bottles into the reaction flask
- draining rack for laboratory glass
- pipette stand from polypropylene