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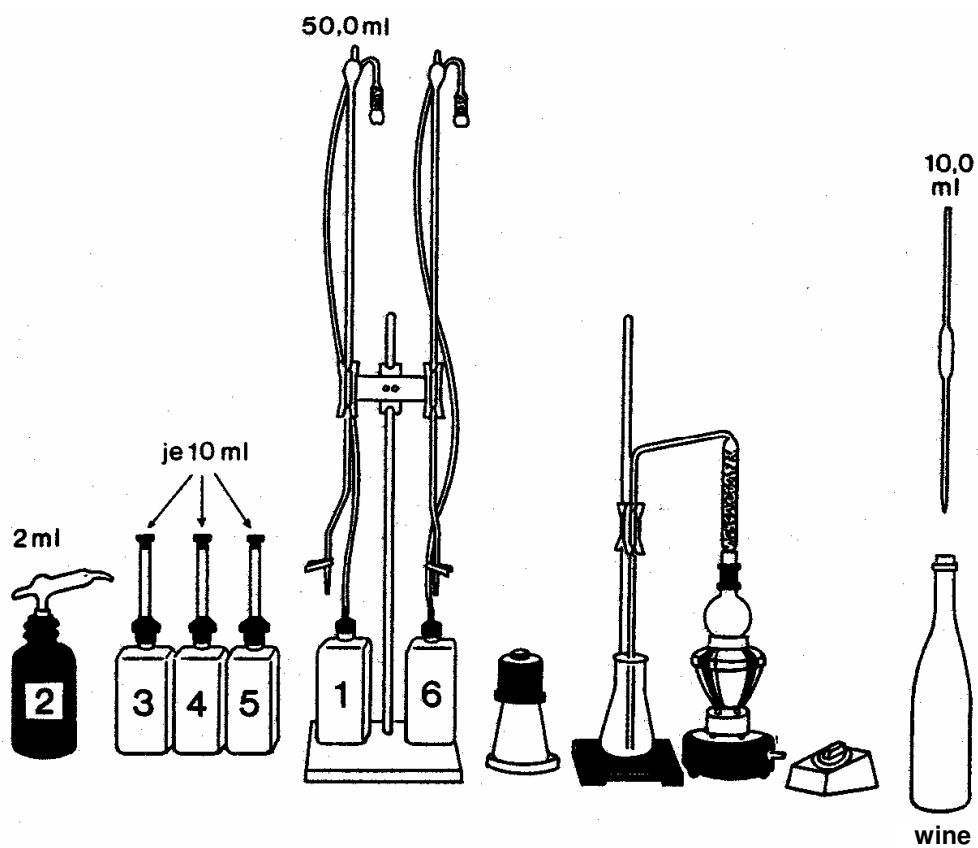
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Getränkeanalytik

Distillation of the total sulphurous acid according to Dr. Rebelein

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page 1/4



Distillation of the total sulphurous acid:

- Heat laboratory burner for approx. 5 minutes.
- Check burettes. The meniscus must be on a level with the top mark of the burettes. Remove drops from the burette jets. Fill up dose cylinders.
- Fill 50,0 ml of "Sulphur 1" into 200 ml conical flask (reaction flask), wipe off burette jet on the inside wall of the flask, place flask under distillation tube.
- Put one level spatula of pumice, 1-2 drops of silicone anti-foam solution, and 2 ml of "Sulphur 2" (tip pipette) into 100 ml flat-bottomed flask.
- Add 10 ml of the liquid to be analysed. (When pipetting the initial solution mind correct pipetting technique.)
- Add 10 ml of "Sulphur 3" (dose cylinder) and immediately connect flask to silicone stopper of distillation tube.
- Swivel distillation flask onto laboratory burner. The distillation tube must reach down to the bottom of the reaction flask. If necessary raise reaction flask by putting aluminium disks underneath.
- Set alarm clock to 3 minutes and distil.
- After distillation raise distillation tube approx. 5-10 cm and turn the tube by 180°, remove the distillation flask and rinse tip of distillation tube well into reaction flask with distilled water.
- Place rubber cap over reaction flask, put flask into Petri disk and cool with running water.
- Once the liquid has cooled down to room temperature (after 2-3 minutes) add according to the given order 10 ml each of "Sulphur 4" and "Sulphur 5", (dose cylinder), simultaneously shaking the flask.
- Titrate the now deep blue liquid with "Sulphur 6" until discoloured. Read burette level. Example: The meniscus of the titration solution reads 232. The analysed liquid contains 232 mg of total sulphurous acid per litre.
- Fill up burette for the next determination to starting mark. Do not leave burettes completely or partly empty.

Preparation of the sample:

The carbonic acid must not be removed by shaking and filtering from the liquid to be analysed because otherwise SO₂ would also be lost. If necessary measure the sample exactly with a graduated cylinder.

Through the addition of 1-2 drops of silicone anti-foam solution foaming can be avoided during distillation. If, for some reason, liquid boils over, it is recommended to distil approx. 10 ml of distilled water to clean the distillation tube.

Pipetting technique:

Rinse pipette first with liquid to be analysed! Suck in the liquid approx. 2 cm above the ring mark of blowing out pipette, 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. Drain the contents of the pipette without loss into the distillation flask with the tip of the pipette put against the wall of the flask approx. 2 cm above the level of the liquid already in the flask. About 15 seconds after the draining has finished blow pipette empty and wipe tip of pipette.

Blank Titration for Checking the Solutions:

Pipette 50,0 ml of "Sulphur 1" into 200 ml conical flask. Add 10 ml each of "Sulphur 4" and "Sulphur 5". Titrate the reaction liquid with "Sulphur 6" until discoloured. The result thus obtained must read 0 mg of total SO₂ per litre with a tolerance of ± 2 mg/l.

Notes Concerning the Distillation of the Total Sulphurous Acid acc. to Dr.Rebelein:

The sulphurous acid is distilled quantitatively into an alkaline oxidation mixture and is titrated back after acidifying the not used oxidising agent. The SO₂ distillation tube could also be used for the determination of alcohol. However it would be better to use the alcohol distillation tube with glass balls for the following reason. In the determination of sulphurous acid an extreme quantity of methanol must be used. During the distillation period of 3 minutes all sulphurous acid is quantitatively removed from the distillation tube and collected in the receiver, but not the excess methanol which, for the following determination, would positive rather than negative. Thus, if the same distillation tube is used for an alcohol determination after an SO₂ test it would first be necessary to distil water for ten minutes in order to completely free the still from the methanol.

Determination of Free Sulphurous Acid without Consideration of reductones and Ascorbic Acid:

- Pipette 10,0 ml of sample into 100 ml flat-bottomed flask.

While shaking the flask add 10 ml each of "Sulphur 3" and "Sulphur 4" in the listed order.

- Titrate with "Sulphur 1" until a pale blue colour persists for at least 30 seconds (reductones and ascorbic acid oxidise slower than sulphurous acid; thus in their presence, the initial blue colour will soon disappear again. This "faded" or "retarded" titration is not completed until the blue colour persists for at least 30 seconds!).
- Multiply the burette reading by 10. This figure indicates the free SO₂ with reductones and ascorbic acid.
Example: The meniscus of the titration solution reads 2,5 ml. The analysed sample contains 10 x 2,5 = 25 mg of free sulphurous acid per litre.
- Pipette 10,0 ml of sample into 100 ml flat-bottomed flask.
- While shaking the flask add 10 ml each of "Sulphur 3" and "Sulphur 4" in the listed order.
- Titrate with "Sulphur 1" until a pale blue colour persists for at least 30 seconds.
- Add 1 level spatula of pumice, 1-2 drops of silicone anti-foam solution as well as 2 ml of "Sulphur 2" into the titrated liquid in the 100 ml flask and connect flask to the distillation tube.

Specific Determination of Free SO₂ with Consideration of reductones and Ascorbic Acid:

Variant 1:

The following are to be determined in 2 operations:

- 1 a) free SO₂ including reductones and ascorbic acid acc. To the previous determination
- 1 b) reductones and ascorbic acid separately

The genuine free SO₂ is obtained from the difference 1 a) - 1 b).

1 b) Determination of Reductones and Ascorbic Acid:

- Pipette 10,0 ml of the sample into 100 ml flat-bottomed flask.
- Add 2 ml of glyoxal and shake slightly. (glyoxal binds free SO₂)
- After 5 minutes add 10 ml each of "Sulphur 3" and "Sulphur 4" while shaking.
- Titrate with "Sulphur 1" until a pale blue colour persists for at least 30 seconds.
- Multiply burette reading by 10. This figure indicates reductones and ascorbic acid only in mg/l.

Calculation of Genuine Free SO₂:

SO ₂ , reductones, ascorbic acid (1 a)	66 mg/l
reductones, ascorbic acid (1 b)	- 18 mg/l
genuine free SO ₂	<u>48 mg/l</u>

Variant 2:

The latent sulphurous acid remaining after oxidation of free SO₂, reductones, and ascorbic acid is distilled and its quantity deducted from the total sulphurous acid. This method is suitable for the examination of darkly coloured samples.

- Pipette 50,0 ml of "Sulphur 1" into 200 ml conical flask and place flask under distillation tube. Place 100 ml flask on burner and lower distillation tube to base of 200 ml flask.
- Set alarm clock to 4 minutes instead of 3 because of larger quantity of liquid in comparison to a total SO₂ distillation. Distil.
- Raise distillation tube, remove 100 ml flask and rinse outlet of distillation tube with distilled water into 200 ml reaction flask.
- Put rubber cap on 200 ml flask, place flask into petri dish, and cool to room temperature with running water.
- While shaking the flask add 10 ml each of "Sulphur 4" and "Sulphur 5" in listed order.
- Titrate liquid with "Sulphur 6" until discoloured. The burette reading indicates the content of latent sulphurous acid of sample in mg/l.

Calculation of Genuine Free SO₂ (Variant 2):

In order to determine the specific free sulphurous acid (free sulphurous acid without reductones and ascorbic acid) the figure found for the latent sulphurous acid must be deducted from the figure found for the total SO₂. The difference of is the specific free SO₂ in mg/l.

Example:	
Total SO ₂	280 mg/l
Latent SO ₂	- 245 mg/l
Calculated free SO ₂	<u>35 mg/l</u>

Distillation of the total sulphurous acid - parts list -

- 1 laboratory burner
- 1 laboratory alarm clock
- 2 Stands with staffs 600 x 12 mm for burettes
- 1 SO₂- distillation tube (with glass rings) with silicone stopper 21 / 16 x 25 mm
- 1 holder for distillation tube
- 1 burette 50 ml TTS for „sulphur1“
- 1 burette „total sulphur according to Dr. Rebelein“ TTS for „sulphur 6“
- 1 holder for burettes 10 – 14 / 12
- 1 blowing out-pipettes 10 ml
- 1 tip pipette 2 ml with silicone stoppers 31/25 x 30 mm for „sulphur 2“
- 3 dose cylinder 10 ml for „sulphur 3“, „sulphur 4“ und „sulphur 5“
- 2 flat-bottomed flask 100 ml NS 19/26 with thermic protection (distillation flask)
- 2 conical-flasks 200 ml (Reaction flask)
- 1 aluminium disk 130 x 130 x 2,5 mm for levelling the burner/reaction flask
- 1 rubber cap size 4a as hood
- 1 petri dish approx 95 mm Ø
for putting into the reaction flask during the cooling with tap water
- 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 map with information-material

original allocation of equipment of reagents:

- sulphur 1 500 ml in polyamide bottle
- sulphur 2 1000 ml in glass bottle
- sulphur 3 500 ml in polyamide bottle
- sulphur 4 500 ml in polyamide bottle
- sulphur 5 500 ml in polyamide bottle
- sulphur 6 500 ml in polyamide bottle

Need per analysis:

- (50,0 ml / burette)
- (2 ml / tip pipette)
- (10 ml / dose cylinder)
- (10 ml / dose cylinder)
- (10 ml / dose cylinder)
- (variable / burette)

recommendable accessories:

- draining rack for laboratory glass
- pipette stand from polypropylene