

Title: Determination of sulfur dioxide in wine

Work instructions

Task: Determine the content of free and bound sulfur dioxide in the wine in mg/l iodometrically.

Theory

Sulfurization of wine is one of the oldest technological operations in wine production. Sulfurization is the application of varying doses of SO₂ to the must and the wine to stabilize the wine. Since too high a SO₂ content is unhealthy, it is essential to control the SO₂ content in the wine. Total and free sulfur dioxide can be determined in wine. Free sulfur dioxide is in the form of sulfurous acid. Bound sulfur dioxide is bound in wine to various substances which are capable of binding it. It can be released from this bond by alkaline hydrolysis. Total sulfur dioxide is the sum of free and bound sulfur dioxide.

The determination of free SO₂ is based on direct iodometric titration, in which free SO₂ is oxidised by iodine according to the Eq:



The determination of total SO₂ is based on the observation that the bound form of SO₂ undergoes hydrolysis in an alkaline medium, releasing sulfate ions directly detectable by titration with iodine solution. The titration process is the same as for free SO₂, but both forms (free and bound SO₂) are already titrated.

Equipment: scales, weighing boats, volumetric flasks (100, 250, 500 ml), beakers, glass rod, iodine titration flask, graduated cylinder, burette, laboratory stand, clamp, holder, pipettes, pipetting balloon

Chemicals:

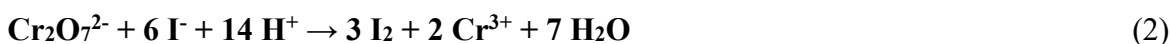
- 25% H₂SO₄ solution: 26 ml of 98% H₂SO₄ ($\rho_{20} = 1.84 \text{ kg/dm}^3$) is slowly added to 74 ml of distilled water. After mixing, the solution must be cooled.
- 0.01 M I₂ solution: dissolve 2.6 g of resublimated iodine (I₂) in 100 ml of 25% KI and make up to 1000 ml with deionised water in a volumetric flask. The solution is stored in a dark bottle.
- 0.5% starch solution: 0.5 g of starch is dissolved in 100 ml of cold water and boiled for about 2 minutes. The solution is stored in a refrigerator or preserved by the addition of 10 mg HgI₂ or by adding it to a saturated NaCl solution.

- 10% KI solution: the solution is prepared just before the analysis, never in stock.
- 0.1 M sodium thiosulfate solution: dissolve 24.8 g of Na₂S₂O₃·5H₂O in distilled water and make up to the mark in a 1000 ml volumetric flask. The solution is then standardised with potassium dichromate.
- 1/60 M potassium dichromate standard solution: dissolve an exact aliquot (0.4904 g; or write the aliquot) of K₂Cr₂O₇ dried at 130°C in a small quantity of distilled water, transfer quantitatively to a 100 ml volumetric flask and make up to the mark.
- 1 M NaOH solution: prepare by dissolving 40 g NaOH in water and make up to 1000 ml.
- aqueous acetaldehyde solution at a concentration of 8 g/l.

Procedures:

Standardization of 0.1 M Na₂S₂O₃ solution

1. Add 15 ml of 10% KI solution, 5 ml of 25% H₂SO₄ and 100 ml of deionised water to the iodine titration flask.
2. Pipette in 20 ml of standard 1/60 M K₂Cr₂O₇ solution with constant stirring.
3. Cap the flask and leave to stand for 15 minutes (brown iodine will precipitate out).
4. Then add the starch solution (5 ml) and titrate with 0.1 M Na₂S₂O₃ solution until the black-blue colour disappears.



Standardisation of a 0.01 M solution of I₂

1. Pipette 25 ml of 0.01 M I₂ solution and about 10 ml of 25% H₂SO₄ into the titration flask.
2. Dilute the mixture with distilled water to approximately 100 ml and titrate with standardised Na₂S₂O₃ solution on starch indicator solution (5 ml) until the blue-black colour disappears.



Sample treatment

1. For the determination of SO₂ in red wines, the wine sample is diluted appropriately (most often 1:1–25 ml of wine and 25 ml of distilled water are pipetted) in order to make it easier to distinguish the equivalence point. The dilution is considered in the calculation.
2. **Note:** The wines should be pipetted into the reaction with as little swirling as possible (pipette tip to the bottom of the titration flask and allow to drain slowly). By stirring too much, SO₂ is lost from the sample.

Determination of free SO₂

1. Pipette 50 ml of wine into the titration flask with the end of the pipette touching the bottom of the flask.
2. Add 10 ml of 25% H₂SO₄ solution and 5 ml of starch solution.
3. Titrate with I₂ solution (0.01 mol/l) until the colour turns blue-black and is maintained for 15 s. Mark the volume of the volumetric solution consumed in ml with V_a .
4. Repeat the determination two more times.

Determination of total SO₂

1. Pipette 50 ml of wine and 25 ml of NaOH solution (1 mol/l) into the titration flask. Cover the flask and leave to stand for 15 minutes.
2. Add 15 ml of 25% H₂SO₄ solution and 5 ml of starch solution.
3. Titrate with I₂ solution (0.01 mol/l) until a blue-black colour is obtained and maintained for 15 s. Mark the volume of the volumetric solution consumed in ml with V_b .
4. Repeat the determination 2 more times.

Determination of SO₂ with correction

1. Pipette 50 ml of wine into the titration flask with the end of the pipette touching the bottom of the flask.
2. Add 5 ml of acetaldehyde solution to the sample and leave to stand for 15 minutes.
3. Then add 10 ml of 25% H₂SO₄ solution and 5 ml of starch solution.
4. Titrate with I₂ solution (0.01 mol/l) until the blue colour is maintained for 15 s. Mark the volume of the volumetric solution consumed in ml with V_c .
5. Repeat the determination 2 more times.
6. From the data obtained, calculate the total and free sulfuric acid content in mg/l of the white wine.

Management of chemical substances

Chemicals	Form	H-statements	P-statements
I ₂	0.05 mol/l solution	H332, H312, H315, H400	P273, P280, P304, P340, P302, P352
Na ₂ S ₂ O ₃ ·5H ₂ O	0.1 mol/l solution	---	---
NaOH	Solid	H290, H314	P234, P260, P280, P303 + P361 + P353, P304 + P340 + P310, P305 + P351 + P338
KI	Solid	H372	P260, P264, P270, P314, P501

Chemicals	Form	H-statements	P-statements
$K_2Cr_2O_7$	Solution, indicator	H272, H302, H314, H317, H331, H334, H335, H340, H350, H360fd, H373, H410	P210, P273, P280, P303 + P361 + P353, P304 + P340 + P310, P305 + P351 + P338
Acetaldehyde	Liquid	H224, H319, H335, H341, H350	P202, P210, P233, P305 + P351 + P338, P308 + P313, P403 + P233
H_2SO_4	25% solution	H290, H314	P234, P280, P303 + P361 + P353, P304 + P340 + P310, P305 + P351 + P338, P363
Starch solution	Solution, indicator	---	---

Sources of risk and assessment of risk severity

Sodium thiosulfate is not a hazardous chemical. Iodine is harmful by inhalation and skin contact. It is highly toxic to aquatic organisms. Students work only with dilute iodine solutions. Students work with dilute acid solutions. Potassium dichromate is toxic and can cause genetic damage and cancer. Students do not encounter the solid; a dilute solution of the indicator is prepared in advance by the assistant. Acceptable risk when using personal protective equipment (lab coat, gloves, goggles).

Waste management method

Pour the residual chemicals into the prepared containers. Place broken glass in the container provided. Do not return the remaining standard solution to the storage bottles.

Risk reduction measures

Prevent the release of iodine into the environment. When in contact with iodine, avoid prolonged or repeated exposure. Wash your skin with plenty of soap and water.

Worksheet

Chemical calculations and evaluation

- Determine the content of free and bound sulfur dioxide in the wine in mg/l iodometrically.

Standardisation of Na₂S₂O₃

$c_{\text{theor.}}(\text{Na}_2\text{S}_2\text{O}_3)$		mol/l
$c_{\text{actual}}(\text{Na}_2\text{S}_2\text{O}_3)$		mol/l

Standardisation of I₂

$c_{\text{theor.}}(\text{I}_2)$		mol/l
$c_{\text{actual}}(\text{I}_2)$		mol/l

Determination

V_a		ml
V_b		ml
V_c		ml

Calculation

The correction factor $f(\text{I}_2)$ is defined as the ratio of the actual to the theoretical concentration of the measuring solution I₂:

$$f(\text{I}_2) = \frac{c_{\text{actual}}}{c_{\text{theor.}}} = \frac{c_{\text{actual}}}{0,01 \text{ mol/l}}$$

The free SO₂ in the sample is calculated according to the Eq:

$$c(\text{free SO}_2) \text{ (mg/l)} = 12,8 \cdot V_a \text{ (ml)} \cdot f(\text{I}_2)$$

The total SO₂ in the sample is calculated according to the Eq:

$$c(\text{total SO}_2) \text{ (mg/l)} = 12,8 \cdot V_b \text{ (ml)} \cdot f(\text{I}_2)$$

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The SO₂ concentration with interferences subtracted is calculated according to the Eqn:

$$c(\text{free SO}_2 \text{ with correction}) (\text{mg/l}) = 12,8 \cdot (V_a - V_c) (\text{ml}) \cdot f(I_2)$$

$$c(\text{total SO}_2 \text{ with correction}) (\text{mg/l}) = 12,8 \cdot (V_b - V_c) (\text{ml}) \cdot f(I_2)$$

Conclusion

Evaluate your work.