Name of the project:	**Digitization of chemistry experiments to improve the quality and support chemistry teaching in secondary schools**
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Tittle: 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 SO_2 to the must and the wine to stabilize the wine. Since too high a SO_2 content is unhealthy, it is essential to control the SO_2 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 SO_2 is based on direct iodometric titration, in which free SO_2 is oxidised by iodine according to the Eq:

$$\mathbf{SO_3^{2-} + H_2O + I_2 \rightarrow SO_4^{2-} + 2I^- + 3H^+} \quad (1)$$

The determination of total SO_2 is based on the observation that the bound form of SO_2 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 SO_2, but both forms (free and bound SO_2) 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% H_2SO_4 solution: 26 ml of 98% H_2SO_4 (ρ_{20} = 1.84 kg/dm^3) is slowly added to 74 ml of distilled water. After mixing, the solution must be cooled.
- 0.01 M I_2 solution: dissolve 2.6 g of resublimated iodine (I_2) 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 HgI_2 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 $Na_2S_2O_3 \cdot 5H_2O$ 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 $K_2Cr_2O_7$ 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 $Na_2S_2O_3$ solution

1. Add 15 ml of 10% KI solution, 5 ml of 25% H_2SO_4 and 100 ml of deionised water to the iodine titration flask.
2. Pipette in 20 ml of standard 1/60 M $K_2Cr_2O_7$ 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 $Na_2S_2O_3$ solution until the black-blue colour disappears.

$$\mathbf{Cr_2O_7^{2-} + 6\ I^- + 14\ H^+ \rightarrow 3\ I_2 + 2\ Cr^{3+} + 7\ H_2O} \quad (2)$$

$$\mathbf{I_2 + 2\ Na_2S_2O_3 \rightarrow 2\ NaI + Na_2S_4O_6} \quad (3)$$

Standardisation of a 0.01 M solution of I_2

1. Pipette 25 ml of 0.01 M I_2 solution and about 10 ml of 25% H_2SO_4 into the titration flask.
2. Dilute the mixture with distilled water to approximately 100 ml and titrate with standardised $Na_2S_2O_3$ solution on starch indicator solution (5 ml) until the blue-black colour disappears.

$$\mathbf{I_2 + 2\ Na_2S_2O_3 + \rightarrow 2\ NaI + Na_2S_4O_6} \quad (4)$$

Sample treatment

1. For the determination of SO_2 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, SO_2 is lost from the sample.

Determination of free SO_2

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% H_2SO_4 solution and 5 ml of starch solution.
3. Titrate with I_2 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 SO_2

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% H_2SO_4 solution and 5 ml of starch solution.
3. Titrate with I_2 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 SO_2 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% H_2SO_4 solution and 5 ml of starch solution.
4. Titrate with I_2 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
$Na_2S_2O_3 \cdot 5H_2O$	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

Experimental data

1. Record the volume of the sodium thiosulphate volumetric solution on $K_2Cr_2O_7$.

Titration	V($Na_2S_2O_3$) [ml]
1	
2	
3	

2. Record the volume of the sodium thiosulphate volumetric solution used in the standardisation of the iodine solution.

Titration	V($Na_2S_2O_3$) [ml]
1	
2	
3	

3. Record the volume of iodine solution used in the determination of free SO_2

Titration	V(I_2) [ml]
V_{a1}	
V_{a2}	
V_{a3}	

4. Record the volume of iodine solution used in the determination of total SO_2

Titration	V(I_2) [ml]
V_{b1}	
V_{b2}	
V_{b3}	

5. Record the volume of iodine solution used in the determination SO_2 concentration with interferents subtracted

Titration	V(I_2) [ml]
V_{c1}	
Vc_2	
Vc_3	

Calculations

1. Calculate the exact concentration of the sodium thiosulphate measuring solution.

2. Calculate the exact concentration of the iodine measuring solution.

3. Calculate the correction factor $f(I_2)$ that is defined as the ratio of the actual to the theoretical concentration of the measuring solution I_2:
$$\boldsymbol{f(I_2) = \frac{c_{actual}}{c_{theor.}} = \frac{c_{actual}}{0,01\ mol/l}}$$

4. Calculate the free SO_2 content in the sample according to the Eq:
$$\boldsymbol{c(free\ SO_2)\ (mg/l) = 12,8 \cdot V_a\ (ml) \cdot f(I_2)}$$

5. Calculate the total SO_2 in the sample according to the Eq:
$$\boldsymbol{c(total\ SO_2)\ (mg/l) = 12,8 \cdot V_b\ (ml) \cdot f(I_2)}$$

6. Calculate the SO_2 concentration with interferents subtracted according to the Eq:
$$\boldsymbol{c(free\ SO_2\ with\ correction)\ (mg/l) = 12,8 \cdot (V_a - V_c)\ (ml) \cdot f(I_2)}$$
$$\boldsymbol{c(total\ SO_2\ with\ correction)\ (mg/l) = 12,8 \cdot (V_b - V_c)\ (ml) \cdot f(I_2)}$$

Questions

1. Write the equation of the reaction that takes place in the standardisation of a volumetric solution of iodine to sodium thiosulphate.

2. Write the equation of the reaction that takes place in the standardisation of a volumetric solution of sodium thiosulphate.

3. Write the equation of the reaction that takes place in the determination of SO_2.

4. Why it is necessary to determine SO_2 in wine?

5. Explain the principle the indication of the equivalence point in iodometry.

6. Indicate the sources of errors in this method. How would you minimise these errors?

Conclusion

Briefly summarize the objective of the experiment, the main results and compare them with the expected values.