Application Bulletin

Of interest to:	Wine growers and bottlers Food analysis laboratories	A G 7

Simple wine analysis

Summary

This bulletin describes simple methods for wine analysis, namely for pH value, total titratable acid, free sulphurous acid, total sulphurous acid as well as ascorbic acid (vitamin C) and other reductones.

Instruments and accessories

- 2.691.0020 pH Meter
- 2.765.0010 or 2.776.0010 (or 2.775.0010) Dosimat with 2.728.0040 Magnetic Stirrer
- Additional 6.3014.223 Exchange Unit 20 mL (with flat PCTFE/PTFE stopcock) with 6.1608.004 PP bottle (for NaOH)
- 6.0239.100 combined LL pH glass electrode with 6.2104.020 electrode cable
- 6.0309.100 double Pt sheet electrode with 6.2104.080 electrode cable
- Possibly 6.1110.100 Pt 1000 temperature sensor with 6.2104.080 electrode cable

Reagents

- Buffer solution pH = 4.00 (Metrohm no. 6.2307.100)
- Buffer solution pH = 7.00 (Metrohm no. 6.2307.110)
- Electrolyte solution c(KCl) = 3 mol/L (Metrohm no. 6.2308.020)
- Stock solution c(NaOH) = 1 mol/L:
  Dissolve 40.0 g NaOH in CO_2-free dist. water and make up to 1 L.
- Titrant c(NaOH) = 0.1 mol/L:
  The above stock solution is diluted 1 : 10 with CO_2-free dist. water.
- Sulphuric acid w(H_2SO_4) = 25%
- Titrant iodide/iodate solution:
  Dissolve 0.5573 g potassium iodate KIO_3 (dried at a temperature not exceeding 150 °C) in 700 mL dist. water, add 3.5 g potassium iodide KI and make up to 1 L with dist. water.
- Potassium iodide KI puriss. p.a.
- Glyoxal solution w(glyoxal) = 40%:
  80 g glyoxal are mixed with 100 mL dist. water. Adjust the pH value of this mixture to 7.0 with c(NaOH) = 1 mol/L and make up to 200 mL with dist. water. The solution has to be stored in the refrigerator using an amber bottle.

Analysis

1. pH value

- Connect the combined pH glass electrode to the pH Meter. Then calibrate with buffer solutions pH = 7.00 and pH = 4.00.
- Rinse the electrode with dist. water and immerse it in the wine sample. When the drift value is reached, read off or print out the pH value. Indication of the value to two decimal places.
- *Example:* **pH value = 3.60**

2. Total titratable acid

- The combined pH glass electrode to be used has already been calibrated prior to the pH measurement. Carbon dioxide must first be removed from the sample.
- Pipette 10 mL wine sample and 10 mL dist. water into a 50 mL beaker. Add a drop of a suitable anti-foaming agent (such as octanol) and heat to boiling point. Immediately allow to cool, then immerse the electrode and titrate to pH = 7 with c(NaOH) = 0.1 mol/L while stirring. Let the titrant consumption be **A** mL.
- *Calculation:* Total acid = **A** * 0.75 (calculated as tartaric acid)
- *Example:* Consumption = 2.80 mL c(NaOH) = 0.1 mol/L
  Total acid = 2.80 * 0.75 = **2.10 g/L**

3. Free sulphurous acid

- Connect the double Pt sheet electrode to the I_{pol} input of the pH Meter.
- Pipette 50 mL sample into a 100 mL beaker and mix with ca. 1 g KI and 5 mL w(H_2SO_4) = 25%. Now slowly titrate with iodide/iodate solution until the indicated value shows a big change, and remains in this state for at least 5 s. If not, add more titrant until the conditions described are reached.* Let the titrant consumption be **B** mL.
- *Calculation:* Free sulphur dioxide = **B** * 10 (calculated as SO_2)
- *Example:* Consumption = 2.31 mL iodide/iodate solution
  Free sulphurous acid = 2.31 * 10 = **23.10 mg/L**

- Brand-new electrodes or electrodes that have been out of use for a long time, may respond sluggishly. In such a case, they will need to be regenerated as follows:

  Short across the two plugs of the double platinum electrode with a wire and connect to the negative pole (cathode) of a 6 V battery. Connect the positive pole (anode) of the battery to an iron nail. Then immerse the electrode and the nail in dilute sulphuric acid solution that still contains some sulphurous acid and leave for 2 ... 3 min. After removal, rinse the electrode thoroughly with dist. water.

4. Total sulphurous acid

- The combined sulphurous acid must first be released by adding NaOH.
- Pipette 25 mL c(NaOH) = 1 mol/L and 50 mL sample into a 100 mL beaker. Place on the magnetic stirrer and stir gently for 15 min. Then add 10 mL $w(H_2SO_4)$ = 25% and ca. 1 g KI and titrate with iodide/iodate solution under the same conditions as described in the section «Free sulphurous acid». The calculation is the same as above.
- *Example:* Consumption = 7.50 mL iodide/iodate solution
  Total sulphurous acid = 7.50 * 10 = **75.0 mg/L**

5. Ascorbic acid (vitamin C) and other reductones

- Free sulphurous acid interferes with the determination and must therefore be bound by adding glyoxal.
- Mix 50 mL sample with 2 mL glyoxal solution and allow to stand for 5 min. Then add 5 mL $w(H_2SO_4)$ = 25% (no addition of KI) and titrate with iodide/iodate solution as described under «Free sulphurous acid». Let the titrant consumption be **C** mL.
- *Remark:*
  If the determination of the free sulphurous acid yields a result exceeding the maximum permitted value, check for the presence of ascorbic acid (vitamin C) according to the method described last. The real content of free sulphurous acid is then calculated as follows:
  mg/L SO_2 = (**B** – **C**) * 10
- The content of **vitamin C** is calculated as follows:
  mg/L vitamin C = **C** * 27.5

Example

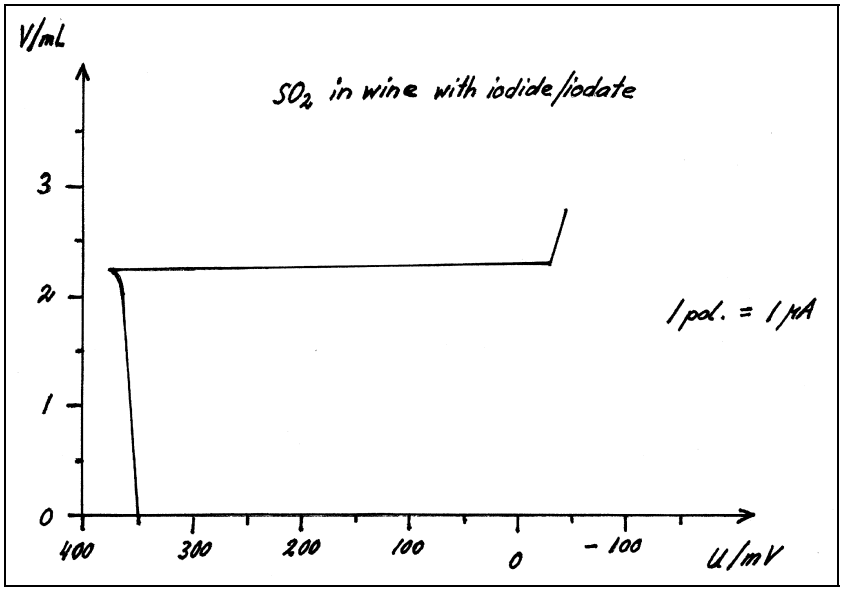

Fig. 1: *Determination of the free sulphurous acid in a wine sample by manual titration with iodide/iodate solution.*

Literature

- Schweizerisches Lebensmittelbuch, chapter 30.
- H. Tanner, H. R. Brunner
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  Verlag Heller Chemie- und Verwaltungsgesellschaft mbH, Schwäbisch Hall, 1979.