

Carbon Dioxide Content in Wine - Determination by Titration

Chemical Concepts and Techniques:

Carbon dioxide in wine derives mainly from the alcoholic and malolactic fermentation processes. Winemakers may also use the gas during wine storage as a protective gas in tanks that have some headspace. As CO₂ is heavier than air, adding dry ice to a tank of wine creates a blanket of CO₂ gas that provides (temporary) protection from oxidation. Carbon dioxide used in this way slowly dissolves into the wine or is lost to the atmosphere.

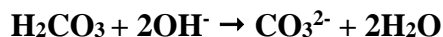
Retention of residual dissolved CO₂ is a function of temperature (CO₂ is more soluble in cold wines). Levels of residual sugar and alcohol do have an effect – at constant temperature CO₂ retention is inversely related to alcohol content.

Still table wines contain <3g/L CO₂. High levels of CO₂ give the wine a characteristic ‘spritzy’. The saturation level of CO₂ in wine at 20°C will depend on alcohol content, but is around 1.5g/L. White wines generally contain higher CO₂ levels as they have been stored at lower temperatures, therefore retaining more CO₂. Dissolved carbon dioxide contributes to apparent acidity – a saturated wine may contain enough CO₂ to be equivalent to 2.0g/L titratable acidity as tartaric acid.

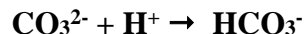
In solution, CO₂ reacts with water to form carbonic acid –



To determine CO₂ content by titration, it is first required to convert the carbonic acid to the carbonate ion by the addition of strong base:



The sample is then titrated with standard acid to **pH 8.6**. This process neutralises any excess hydroxide ions and converts the carbonate ions to bicarbonate:



Further titration to **pH 4.0** with standardised acid converts all the bicarbonate to carbon dioxide and water (carbonic acid)



Equipment Required:

25mL burette

100mL Schott™ bottles with screw caps or similar

10mL volumetric pipette

100mL beaker

Pasteur or plastic transfer pipettes

Electronic pH meter, preferably with probe support arm to suspend electrode in solution

Magnetic stirrer and stirbar (optional)

Plastic jug or other container for ice bath

Reagent Preparation:

Note that the 50% w/v Sodium Hydroxide solution must be prepared by Laboratory staff, not students.

50% w/v Sodium Hydroxide: Take extreme care during preparation. Weigh approximately 50g of sodium hydroxide. Add the solid NaOH slowly and carefully and with immediate stirring, to around 100mL distilled water in a large beaker. Stand the beaker in an ice bath during preparation as extreme heat is evolved. Mix well, cool and store in a plastic container.

0.10M Standard Hydrochloric Acid: Available commercially (standardisation not necessary in this case).

Freshly boiled, cooled distilled water: preferably stored with an in-line CO₂ (soda lime) trap.

pH buffers: Commercial grade, pH 4.0 and 7.0 recommended

Method:

Determination of the sample titre:

1. Calibrate the pH meter using at least two buffer solutions.
2. Carefully measure 100mL of the sample into a 100mL Schott® bottle or other suitable vessel with screw cap. Avoid any splashing of the solution as this can affect the CO₂ content.
3. Seal the bottle and place in an ice bath to bring the temperature down to about 5°C.
4. Remove the cap and quickly (but carefully) add 1mL of 50% sodium hydroxide solution (a Pasteur pipette is fine for this).
5. Reseal the bottle immediately and mix well. **Allow the sample to return to room temperature.**
6. Pipette 10.0mL of the treated wine into a beaker. Add sufficient freshly boiled, cooled distilled water to ensure that the solution level is high enough to cover the pH electrode fully. Place the pH probe in the solution and confirm that the initial pH is >11.
7. Add a magnetic stirbar and commence stirring if using a magnetic stirrer.
8. Rinse and fill the burette with the 0.1M hydrochloric acid solution.

9. Commence the titration, mixing the solution well as acid is added. Titrate until the pH of the solution reaches 8.6. Record the burette reading (B_1)
10. Continue the titration until the pH of the solution reaches 4.0. Record the second burette reading (B_2).
11. Calculate the Sample Titre $B_2 - B_1$.

Determination of the blank titre:

1. Measure a further 100mL of the sample into a beaker and degas. This can be done by heating rapidly until boiling (do not allow to boil more than a few seconds).
2. Transfer to a 100mL Schott® bottle, seal and place in an ice bath to bring the temperature down to about 5°C.
3. Repeat steps 4 – 11 above.
4. Calculate the Blank Titre $B_2 - B_1$.

Calculation of the net CO₂ content:

1. Calculate the CO₂ content using the following equation:

$$\text{CO}_2 \text{ (g/L)} = [\text{Sample titre(mL)} - \text{Blank titre(mL)}] \times 0.44$$

Points to consider:

- **50% sodium hydroxide solutions are very corrosive.** If caustic solution are spilt on skin, rinse **immediately** under plenty of cold running water, rubbing the affected area until no sensation of ‘soapiness’ or ‘sliminess’ remains. Students must wear eye protection when handling strong caustic solution, as caustic burns to the eye can cause blindness. If caustic solution is spilt on clothing, remove the clothing immediately and rinse well with water.
- The initial pH of the treated wine must be >11 to ensure that all carbonic acid has been converted to carbonate. If the pH is not >11, prepare a fresh sample using 2mL of NaOH solution rather than 1mL.
- The dilution water must be freshly boiled to remove dissolved CO₂, cooled and stored with an in-line CO₂ to avoid error.
- The addition of the NaOH solution causes a slight dilution error – in this case about 1%. For best accuracy, multiply the final result by 1.01 to account for this dilution error.
- It is not necessary to record an initial burette reading for the first acid addition. The titration does not commence until the pH reaches 8.6, when the true initial burette reading must be recorded.
- It is not practical to try and attempt this titration using indicators instead of a pH metre. Indicators change colour over a range of pH values, not a fixed point, so would not provide sufficient accuracy in this case. Also, the pH range of the titration would require the use of two different indicators (one for pH 8.6, one for pH 4.0) with possible colour interference.
- It is important that the titration be carried out once the solution has returned to room temperature. **pH measurements are very temperature dependent** and

must be made at the same temperature that the meter was calibrated at to be accurate. Attempting measure pH on a solution not at room temperature will cause drifting as the pH changes due to the solution warming up.

References:

Jean L. Jacobson, *Introduction to Wine Laboratory Practices And Procedures*, 2006
Iland, Ewart, Sitters, Markides & Bruer, *Techniques for Chemical Analysis and Quality Monitoring during Winemaking*, 2000
Zoecklein, Fugelsang, Gump & Nury, *Production Wine Analysis*, 1990