

SOP 14

Procedure for preparing sodium carbonate solutions for the calibration of coulometric C_T measurements

1. Scope and field of application

This procedure describes the preparation of a set of aqueous sodium carbonate solutions which are to be used for the calibration of the determination of total dissolved inorganic carbon by extraction/coulometry. Such solutions provide an alternative to the use of gas loops for the calibration of this apparatus.

2. Principle

Six different standard solutions are prepared which contain carefully weighed amounts of pure sodium carbonate, such that the concentrations are at approximately $500 \mu\text{mol dm}^{-3}$ intervals from 0–2500 $\mu\text{mol dm}^{-3}$. Care is taken during the preparation and use of these solutions to minimize contamination by atmospheric carbon dioxide.

3. Apparatus

- $6 \times 1 \text{ dm}^3$ calibrated volumetric flasks (SOP 13),
- Balance capable of weighing the sodium carbonate to $\pm 1 \mu\text{g}$.

4. Reagents

- High purity sodium carbonate, > 99.95% (If you wish to prepare high purity sodium carbonate yourself, follow the procedure given in IUPAC, 1969.),
- Deionized water (If desired, carbon dioxide can be removed by boiling and allowing to cool in a stream of nitrogen.),
- Cylinder of N_2 gas.

5. Procedure

5.1 Preparation of solutions

Dry the sodium carbonate to constant weight in an oven at 280°C for > 2 hours and store it in a desiccator over phosphorus pentoxide until it is used.

The following procedure is carried out for each solution prepared:

- Weigh out the necessary amount of salt in a small container, such as a plastic vial, which has been treated to reduce static charge (*e.g.*, with an anti-static strip).
- Flush the clean dry flask with nitrogen for 5 minutes.
- Fill the flask $\frac{3}{4}$ full with deionized water, filling from the bottom of the flask through a piece of tubing.
- Using a funnel, transfer the sodium carbonate from the vial to the flask. Rinse the vial and the funnel into the flask to ensure quantitative transfer of the sodium carbonate into the flask.
- Adjust the volume of solution contained in the flask to the calibration mark.
- Make sure that the headspace is filled with nitrogen gas and then close the flask with a lightly greased stopper. Once it is closed, shake it gently to dissolve all the sodium carbonate and to mix the solution.

These solutions will last for at least 1 week, provided that the headspace is kept filled with nitrogen and that the flasks are resealed after each use.

5.2 Calculation of concentrations of solutions

First correct the weight of Na_2CO_3 for air buoyancy effects (SOP 21):

$$m(\text{Na}_2\text{CO}_3) = w(\text{Na}_2\text{CO}_3) \left(\frac{1 - \rho(\text{air})/\rho(\text{weights})}{1 - \rho(\text{air})/\rho(\text{Na}_2\text{CO}_3)} \right). \quad (1)$$

Then combine this with information on the calibrated volume of the flask, $V(\text{flask})$ (SOP 13) and on the purity of the sodium carbonate (if known):

$$\frac{c(\text{Na}_2\text{CO}_3)}{\text{mol dm}^{-3}} = \frac{m(\text{Na}_2\text{CO}_3)/\text{g}}{105.988} \cdot \frac{1}{V(\text{flask})/\text{dm}^3} \cdot \text{purity}. \quad (2)$$

5.3 Example calculation

$$\begin{aligned} w(\text{Na}_2\text{CO}_3) &= 0.21230 \text{ g}, \\ V(\text{flask}) &= 1.0001 \text{ dm}^3, \\ \rho(\text{weights}) &= 8.0 \text{ g cm}^{-3}, \\ \rho(\text{Na}_2\text{CO}_3) &= 2.532 \text{ g cm}^{-3}, \\ \text{purity of Na}_2\text{CO}_3 &= 99.95\%. \end{aligned}$$

Thus

$$\begin{aligned}m(\text{Na}_2\text{CO}_3) &= 0.21230 \left(\frac{1 - 0.0012/8.0}{1 - 0.0012/2.532} \right) \\ &= 0.21237 \text{ g}\end{aligned}$$

and

$$\begin{aligned}c(\text{Na}_2\text{CO}_3) &= \frac{0.21237}{105.988} \cdot \frac{1}{1.0001} \cdot 0.9995 \\ &= 2002.5 \mu\text{mol dm}^{-3}.\end{aligned}$$

6. Quality assurance

The set of six calibration standards are used in a linear regression procedure (SOP 23) to calibrate the extraction/coulometric system for measurement of total dissolved inorganic carbon. If the relative standard error of the slope obtained is too high ($> 0.15\%$), the data should be examined to ascertain the source of error. If necessary, additional standard solutions should be prepared. Furthermore, if the value of the intercept—the background level of carbon dioxide in the deionized water—is too high ($> 15 \mu\text{mol dm}^{-3}$), the solutions should be rejected and made again with freshly boiled deionized water.

7. Bibliography

IUPAC. 1969. Sodium carbonate and sulphamic acid as acid-base primary standards. *Pure Appl. Chem.* **18**: 445–455.