Boric Acid

DOC316.52.93094

Inflexion potentiometric titration
Application: In nuclear cooling water

1. Introduction

This working procedure deals with the titration of boric acid using sodium hydroxide:

$$H_3BO_3 + NaOH \rightarrow H_2BO_3^- + Na^+ + H_2O$$

2. Principle

Boric acid is a weak acid. To improve the quality of the titration by a strong base, mannitol is added to the sample to form a complex with boric acid resulting in a medium-strong acid compound with a unique pH jump.

This method is suitable for the determination of boric acid in the range of 0.15 to 3.00 g/L.

Boric acid concentration in g/L can be directly calculated from the equivalent volume.

3. Electrode and reagents

Electrodes: Intellical combined pH electrode with integrated temperature sensor, PHC705

Titrant: NaOH 0.1 mol/L; Use a commercial solution or dissolve 4.00 g of sodium hydroxide in 1 L of CO_2-free boiled water

Reagent: Mannitol 75 g/L; Dissolve 75 g of D-mannitol in water and dilute to 1 L

Standard for titrant calibration: Dihydrate oxalic acid, molar weight = 126.07 g/mol

pH standards: Colored 4.01, 7.00, 10.01 (part numbers 2283449, 2283549, 2283649)

Deionized water

4. Ranges and settings

4.1. Default parameters

The working procedure is described using the following parameters:

- V sample = 20 mL
- V mannitol solution = 10 mL
- Syringe volume = 10 mL

4.2. Working range

For most samples, 1 syringe (10 mL) of titrant should be sufficient to reach the equivalent point. It provides the following range:

Veq (mL)	0.5	9.5
Concentration in Boric Acid (g/L)	0.15	2.85

The syringe is allowed to refill once during titration, so the range is widened:

Veq (mL)	0.5	19
Concentration in Boric Acid (g/L)	0.15	5.70

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4.3. Settings

Name	Default parameter	Unit
Sample		
Name	Sample	
Amount	20	[mL]
Amount min	0	[mL]
Amount max	22	[mL]
Titrant		
Name	NaOH	
Titrant concentration	0.1000	[mol/L]
Syringe	Syringe 1	
Probe		
Recommended probe	PHC705	
Leveling		
Active	No	
Time	30	[s]
Automatic addition		
Active	Yes	
Stirring speed	25	[%]
Pump	One	
Time	6	[s]
Manual addition		
Active	No	
IP titration		
Stirring speed	25	[%]
Measured parameter		[pH]
Predose	0	[mL]
Max volume stop point	20	[mL]
Stop on last EQP	True	
Delay	2	[s]
Min increment size	0.08	[mL]
Max increment size	0.8	[mL]
Result 1 name	Boric Acid	
R1 resolution	3 decimals	
R1 min	0.15	[g/L]
R1 max	5.7	[g/L]
R1 QC min	0.15	[g/L]
R1 QC max	5.7	[g/L]

4.4. Modification of the settings

The parameters are defined in order to have the best compromise between accuracy and titration time.

For high concentration with a high titrant volume, titration time can be reduced with an addition of titrant (predose) at the beginning of the titration. Enter the predose volume (in mL) and the stirring time after the addition in the application edit window.

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5. Titration procedure

5.1. Leveling

To use this method, an external pump is required. All elements (probes, tubes from the titrator and the tube from the external pump) have to be well installed on the probe holder. The beaker has to contain a level of sample higher than the position of the tube of the external pump. When the beaker is attached to the probe holder, this method allows the system to automatically remove the excess sample by a defined pump working time, and always keep the same sample volume before launching the analysis.

In order to define this volume, autoleveling calibration sequence has to be previously executed (see section **8.3 Autoleveling calibration**).

When this option is active, the working time of the external pump must be set (default 30 s). The minimum working time must allow the pump to be removing air during the last few seconds of the external pump activation.

Note: *Do not forget to re-edit the sample amount with the expected value when deactivating the leveling method.*

5.2. Titration

Rinse the pH probe with deionized water. If leveling is disabled, use a pipette to collect precisely 20 mL of sample.

Pour the sample into the 50 mL beaker, put in a magnetic stir bar, dip the probe and the delivery tip in the solution and then start the application. 10 mL of mannitol solution will be automatically added[1].

At the end of the titration, a first window displays boric acid concentration in g/L. A second window appears displaying the titration curve and the equivalent point coordinates.

By pressing **next** it is then possible to:

- Replicate the sample. This is used to study the repeatability by analyzing several samples successively. At the end of each titration, a window displays the average value, the standard deviation (SD in g/L) and the relative standard deviation (RSD in %).
- Analyze a new sample. Another titration can be started but no Standard Deviation and RSD value will be made.

Always rinse the pH probe and the delivery tip between measurements.

6. Results

6.1. Result calculation

The calculation used is:

$$\text{Boric acid (g/L)} = \frac{C_{\text{titrant}}\,(\text{mol/L}) \times V_{\text{titrant}}\,(\text{mL})}{V_{\text{sample}}\,(\text{mL})} \times M_{\text{boric acid}}\,(\text{g/mol})$$

$$= \frac{0.1\,(\text{mol/L}) \times V_{\text{titrant}}\,(\text{mL})}{20\,(\text{mL})} \times 61.83\,(\text{g/mol})$$

[1] Manual addition of the mannitol solution is also possible: in **Edit** mode, deactivate **Automatic addition** and activate **Manual addition**

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6.2. Experimental results

These results are indicative and have been obtained for three boric acid solutions, for five successive determinations each time.

Solution at 0.2 g/L

Measurement	Parameter	Unit
Mean concentration	0.201	[g/L of boric acid]
Standard deviation	0.003	[g/L of boric acid]
Relative standard deviation	1.3	[%]
Mean titration duration	72	[s]

Solution at 1.5 g/L

Measurement	Parameter	Unit
Mean concentration	1.486	[g/L of boric acid]
Standard deviation	0.007	[g/L of boric acid]
Relative standard deviation	0.4	[%]
Mean titration duration	102	[s]

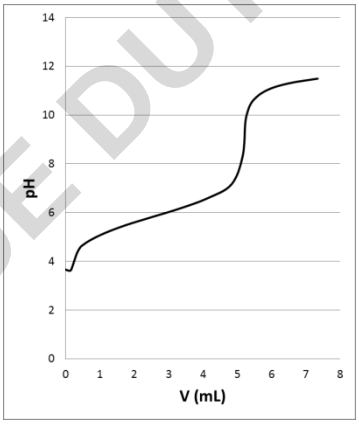
Solution at 3 g/L

Measurement	Parameter	Unit
Mean concentration	2.942	[g/L of boric acid]
Standard deviation	0.003	[g/L of boric acid]
Relative standard deviation	0.1	[%]
Mean titration duration	145*	[s]

*with one burette filling

6.3. Example of a titration curve

This curve has been obtained during the analysis of one of the five samples at 1.5 g/L.



7. Recommendations

Always rinse the pH probe and the delivery tip between measurements.

Refill the electrode regularly with KCl saturated solution to maintain the level of internal solution around 1 cm (0.4 inches) below the refill hole.

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8. Appendices

8.1. Electrode calibration

For the precision of the titration, it is recommended to calibrate the probe with pH standards at the same temperature as the samples. For calibration, the pH is compensated at 25 °C.

Pour a sufficient amount of the first buffer into a 50 mL beaker and add a magnetic stir bar. Place it on the titrator under the probe holder and dip the probe into the beaker. Start the electrode calibration sequence. Rinse the probe between two buffers. Repeat this operation for each buffer (at least two buffers are recommended).

At the end of the calibration, the results of the slope and offset are displayed and the user can accept or reject this result.

Default settings for electrode calibration

Name	Default parameter	Unit
Stirring speed	25	[%]
Stability criteria	0.05	[pH/min]
Stability max time	300	[s]
Slope limit min	97	[%]
Slope limit max	102	[%]
Offset limit min	29	[mV]
Offset limit max	89	[mV]

8.2. Titrant calibration

The sodium hydroxide solution can be calibrated. The exact concentration can be determined from an acid-base titration using oxalic acid.

Weigh 30 mg of dihydrate oxalic acid in a 50 mL beaker and use a graduated cylinder to add 25 mL of deionized water. Put in a stir bar, dip the probe and the delivery tip into the solution and launch the titrant calibration sequence. When prompted, type in the exact weighed amount (three digits). At the end of the titrant calibration, titer (eq/L) is displayed and the user can reject, replicate, or save the result.

Default settings for titrant calibration

Name	Default parameter	Unit
Titrant		
Name	NaOH	
Titrant concentration	0.1000	[mol/L]
Syringe	Syringe 1	
Standard		
Name	Oxalic acid	
Amount	30	[mg]
Amount min	25	[mg]
Amount max	40	[mg]
Molar weight	126.07	[g/mol]
EP titration		
Stirring speed	25	[%]
Measured parameter		[pH]
Predose	2	[mL]
Max volume stop point	8	[mL]
Stop on last EQP	True	
Delay	0	[s]
Min increment size	0.08	[mL]
Max increment size	0.8	[mL]
EP Ordinates	8.55	[pH]
Result name	Titer	
Result resolution	4 decimals	
Result min	0.09	[mol/L]
Result max	0.11	[mol/L]

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8.3. Autoleveling calibration

The aim of this method is to calibrate the volume of sample by leveling. The result of this calibration will be used as sample volume for the following titrations.

This option is **ONLY** available from the calibration menu if **Method Leveling** is set to Active (**Yes**). Refer to the documentation delivered with the external pump for a correct installation, paying particular attention to the suction tube from the pump.

Prepare a 1.5 g/L boric acid solution or use a sample of known concentration (in this case, change the concentration in **Edit** mode > **Autoleveling calibration**).

Pour a sufficient amount of the solution into a beaker allowing the external pump tube to be immersed in the liquid. In the calibration menu select **Autoleveling calibration** and then **Boric Acid**.

The result in mL is compared to minimum and maximum amounts defined for the sample volume. The calculation used is:

$$V_{sample} = \frac{V_{titrant}(mL) \times C_{titrant}\ (mol/L)}{C_{boric\ acid\ solution}\ (g/L)} \times M_{boric\ acid}\ (g/mol)$$

$$= \frac{V_{titrant}(mL) \times 0.1\ (mol/L)}{1.5\ (g/L)} \times 61.83\ (g/mol)$$

Autoleveling calibration uses the same settings as for a sample analysis (see section **4.3 Settings**).

Default settings for autoleveling calibration

Name	Default parameter	Unit
Sample		
Amount min	0	[mL]
Amount max	22	[mL]
Autoleveling calibration		
Solution name	Boric Acid	
Concentration	1.5	[g/L]
Resolution	3 decimals	

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