



End Point Titration Manager

Operational Qualification

Introduction

The operational qualification procedure demonstrates that an instrument runs according to the operational specifications in the selected environment. For an end point titration manager, this operation can consist of two steps:

- Calibration and checking of a pH measurement electrode system
- Titration of anhydrous sodium carbonate standard using a commercial standard solution of HCl.

Principle

The reaction has 2 steps



corresponding to 2 different end points.

The operational qualification takes into account the second equivalence point (pH 3.90) corresponding to the complete neutralisation of sodium carbonate.

Electrode and reagents

pHC2011-8 Combined pH Electrode (part no. E16M317)
 T201 Temperature Sensor (part no. E51M001)

IUPAC Series pH standards

pH 4.005 (part no. S11M002),

pH 7.000 (part no. S11M004) and

pH 10.012 (part no. S11M007)

100 g bottle of Merck "certified" sodium carbonate (part no. 1.06394.0100) or 500 g bottle of "pro analysis" Merck sodium carbonate (part no. 1.06392.0500)

1 l of "ready to use" Merck HCl

0.1 mol/l (part no. 1.09060.1000)

Distilled water

End Point titration settings

Electrode

pH: pHC2011-8
 Calibration request: YES
 Number of cycles: 2
 Number of buffers: 3
 Measurement: Stirring
 Temperature: probe

Calibration parameters

Stability: 15 mpH/min
 Acceptation time: 2.00 min

Max. stab. Time: 5.00 min
 Acceptation criteria: YES
 Iso. pH: 6.65 pH

Calibration solutions

1: 4.005 (IUPAC)
 2: 7.000 (IUPAC)
 3: 10.012 (IUPAC)
 Min. zero pH: 5.8 pH
 Max. zero pH: 7.5 pH
 Min. sensitivity: 95%
 Max. sensitivity: 103%

Titrant

ID: HCl
 Unit: M
 Titre: Entered

Method

Predose: see below
 Start timer: 45 sec
 Max. volume: see below
 Number of EP: 1
 Min. speed: 0.2 ml/min
 Max. speed: 5.00 ml/min
 Direction: decreasing pH
 End point: 3.90 pH
 Delay: 10 sec
 Proportional band: 3.00 pH

Results

Sample unit: mg
 Number of result: 1
 Acceptation criteria: YES
 Result unit: %
 Molar weight: 105.99
 Reaction: 1 smp + 2 titr
 Minimum value: 99
 Maximum value: 101

Procedure

Electrode calibration and checking

If the electrode is new, condition it by immersion in distilled water for at least 1 hour

Connect the pHC2011-8 combined pH electrode and the T201 Temperature Sensor

Using the above-mentioned settings, RUN an electrode calibration with 2 or more cycles

At the end of the last cycle, calibration results should be accepted

Ensure that the temperature of the standards does not differ by more than 2°C.

End point titration operation qualification

Fit the titration manager with HCl 0.1 M as titrant and install the titrant.

Preparation of Na₂CO₃

As indicated, dry approximately 5 g of anhydrous sodium carbonate in an oven for 4 hours at 250°C. Let it cool to room temperature in a desiccator with P₂O₅ or another humidity adsorber.

End point titration

To determine the necessary amount of sodium carbonate

With a 25 ml burette capacity

Weigh exactly 85-90 mg of Na₂CO₃

This weight corresponds to 1.6-1.8 meq or 16-18 ml of HCl 0.1M. Use a predose corresponding to 10 ml and a maximum volume of 25 ml

With a 10 ml buret capacity

Use the same settings as the 25 ml buret capacity

With a 5 ml buret capacity

Weigh exactly 40-45 mg of Na₂CO₃

Use a maximum volume of 10 ml

No predose.

Dissolve the weighed sodium carbonate quantitatively in the titration beaker with 50 to 80 ml of freshly distilled water. Immerse the electrode and the delivery tip in the solution. Using the above-mentioned settings, run a titration with 3 different samples. Results should be accepted between 99 and 101%.