



Method 9.3 – Boiler and boiler feed water: total hardness

1. Rationale

The method is applicable to boiler water and boiler feed water and determines the total hardness of the water.

2. Principle

Water is termed hard when it does not form an immediate lather with soap. The hardness is due to the presence of dissolved calcium, magnesium and iron compounds. This method uses either one of two methods for quantifying the total hardness of a water sample. A titration method is used for boiler feed water in which the metals are complexed with a standard EDTA solution. The soap method is used for boiler water in which the sample is mixed with increasing amounts of a soap solution until a permanent lather is obtained.

3. Apparatus

- 3.1 **Burettes:** 10 and 50 cm³
- 3.2 **Conical flask:** 250 cm³
- 3.3 **White tile** or sheet of paper
- 3.4 **Measuring cylinder:** 100 cm³
- 3.5 **Pipette:** 10 cm³
- 3.6 **Analytical balance** readable to 0.0001 g
- 3.7 **Volumetric flasks:** 250 and 500 cm³
- 3.8 **Bottle,** glass with stopper: 250 cm³
- 3.9 **Glass mortar and pestle**

4. Reagents

- 4.1 **EDTA solution** (0.01 M)

Ethylene diamine tetra acetic acid (EDTA), disodium salt dihydrate is mildly irritating to the skin, eyes and respiratory tract. Work in a fume cupboard while wearing gloves and safety glasses.

Weigh 1.8612 g EDTA, dissolve in distilled water and dilute to 500 cm³ in a volumetric flask.

4.2 Eriochrome Black T indicator powder

Mix 0.1 g of Eriochrome Black T with 30 g potassium chloride or sodium chloride. Grind the mixture with a glass mortar and pestle.

4.3 Ammonium chloride

Ammonium chloride (NH₄Cl) is a corrosive base and contact with the skin and eyes should be avoided. Wear safety glasses and gloves during handling.

4.4 Ammonia buffer solution (pH 10)

Ammonia (NH₄OH) is a corrosive base and contact with the skin, eyes and through inhalation should be avoided. Use in a fume cupboard while wearing gloves and safety glasses.

Add 142 cm³ concentrated ammonia to 17.59 g ammonium chloride (NH₄Cl) and dilute with distilled water to 250 cm³ in a volumetric flask.

4.5 Soap solution (Wanklyn)

Wanklyn's soap solution is available from BDH and is an ethanolic soap solution with 1 degree of hardness on the Wanklyn scale, *i.e.* 1 cm³ is equivalent to 1 mg calcium carbonate (CaCO₃).

5. Procedure

5.1 EDTA titration

Measure 100 cm³ of the boiler feed water sample in a 250 cm³ conical flask and add 10 cm³ of the ammonium buffer solution. Add a small amount of indicator powder. Titrate against a white background using the white tile or paper with 0.01 M EDTA solution from the 50 cm³ burette until the colour changes to blue.

5.2 Soap test

Measure 100 cm³ of the boiler water sample in a stoppered glass bottle. Add soap solution from the 10 cm³ burette in aliquots of 0.2 cm³. Shake after each addition and continue until a permanent lather is obtained. Determine the reagent blank for each batch of soap solution by using 100 cm³ distilled water instead of the water sample.

6. Expression of Results

6.1 EDTA titration

1 cm³ of the 0.01 M EDTA solution will react with 10 mg/litre of calcium carbonate (CaCO₂). Therefore, multiply the titre of the titration with 10 to give the total hardness in the sample in mg/litre CaCO₃.

6.2 Soap test

1 cm³ of the soap solution is equivalent to 1 g CaCO₃. Therefore, for a 100 cm³ sample volume, multiply the difference between the titre and the blank by 10 to give the total hardness in the sample in mg/litre CaCO₃.

7. References

SASTA (1985). *Laboratory Manual for South African Sugar Factories*. 3rd Edition: 350.