

***MT 75.3 DETERMINATION OF pH VALUES**

OUTLINE OF METHOD

The pH value of a mixture of a sample with water or of an undiluted aqueous formulation is determined by means of a pH meter and an electrode system.

REAGENTS

Di-sodium tetraborate buffer solution 0.05 mol/l, commercially available or self prepared solution (see e.g. Note 1)

Potassium hydrogen phthalate buffer solution 0.05 mol/l, commercially available or self prepared solution (see e.g. Note 2)

Water, distilled or de-ionised, in equilibrium with CO₂ from the air (Note 3).

APPARATUS

pH meter capable of at least two point calibration

Electrode system e. g. glass electrode system, conditioned and stored according to the manufacturer's instructions

Measuring cylinder stoppered, 100 ml

PROCEDURE

(a) *Calibration*. Operate the pH meter and the electrode system according to the manufacturer's instructions. Calibrate the measuring system (pH meter and electrode) according to the manufacturer's instructions using at least two appropriate buffer solutions.

(b) *Measurement of pH values*

(i) *Measurement of diluted samples*. Weigh 1.0 g of sample (Note 4) into a measuring cylinder containing about 50 ml water (Note 5), make up to 100 ml with water and shake vigorously until completely mixed or dispersed. If necessary, transfer the solution or dispersion to a beaker (200 ml) and allow any suspended material to settle for 1 min. Ensure that the temperature of the sample/water mixture does not differ from the temperature of the borate buffer used the time of calibration. Immerse the electrode into the liquid and immediately start the stopwatch (Note 7). Record the pH values after 1 and 2 minutes, without stirring during the measurement. If the two pH values differ

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by more than 0.1 pH units, record and report the pH value minutes after immersing the electrode.

(ii) *Measurement of undiluted aqueous formulations.* Transfer enough sample to a beaker (100 ml). Continue as for (i) *Measurement of diluted samples* beginning at: 'Ensure that the temperature.....'

(c) *Reporting of the results.* Give the result to the nearest 0.1 pH unit and report the following conditions:

- (i) the concentration of the mixture that has been measured (normally 1 % w/v, or 1 % v/v for liquid samples)
- (ii) whether or not the pH has been measured in an undiluted sample
- (iii) the type of water that has been used
- (iv) the temperature at which the pH has been measured.

Note 1 Di-sodium tetraborate solution, 0.05 M (pH see below):

Dissolve 19.07 g di-sodium tetraborate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$) in water (2.2) and make up to 1000 ml. *Do not keep the solution for longer than one month.*

pH values:

Temperature [°C]	10	15	20	25	30
pH	9.29	9.26	9.22	9.18	9.14

Note 2 Potassium hydrogen phthalate solution, 0.05 M (pH: 4.00):

Dissolve 10.21 g potassium hydrogen phthalate ($\text{HOOC-C}_6\text{H}_4\text{-COOK}$) in water and make up to 1000 ml. The temperature coefficient of the pH of this buffer solution can be neglected between 10 and 30 °C. *Do not keep the solution for longer than one month.*

Note 3 If necessary, the water used should be freshly boiled and cooled to room temperature.

Note 4 In case of liquid samples which are not too viscous, e. g. emulsion concentrates, 1 ml (or more) of the sample may be used.

Note 5 Special types of water (e. g. CIPAC Standard Water D) instead of distilled or de-ionised water may be prescribed.

Note 6 During the measurement of pH values of samples a randomly fluctuating reading of the pH meter may be observed. The reason is normally that the concentration of ions in the sample (diluted or undiluted) is too low, or because of an interaction between the particles or droplets in suspension with the electrode. If the ion concentration is too low, some drops of a concentrated sodium chloride solution may be added to stabilise the reading.

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Note 7 When using an automatic pH meter, where the measurement stops automatically as soon as the pH changes are less than a pre-set drift value of 0.1 pH units/min, a measurement period less than 10 min is acceptable and the resulting pH value should be reported.