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Urea for industrial use — Determination of the buffer coefficient — Potentiometric method

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FOREWORD

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International Standard ISO 2751 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in June 1972.

It has been approved by the Member Bodies of the following countries :

Austria	Israel	Sweden
Belgium	Italy	Switzerland
Bulgaria	Netherlands	Thailand
France	New Zealand	Turkey
Germany	Poland	United Kingdom
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India	Romania	
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This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

No Member Body expressed disapproval of the document.

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Urea for industrial use — Determination of the buffer coefficient — Potentiometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a potentiometric method for the determination of the buffer coefficient of a solution, of conventional concentration, of urea for industrial use.

2 PRINCIPLE

Measurement, at $20 \pm 0,5$ °C, of the quantity of 0,05 N acid solution required to effect a pH change from 8 to 6 for a solution containing 100 g of urea in 1 000 ml of solution.

3 REAGENTS

Distilled water, or water of equivalent purity, recently boiled and cooled to room temperature, shall be used in the test.

3.1 Hydrochloric acid, 0,05 N standard volumetric solution.

3.2 Sodium hydroxide, 0,05 N standard volumetric solution.

3.3 Buffer solution of disodium tetraborate, 0,01 M.

Dissolve $3,81 \pm 0,01$ g of disodium tetraborate decahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) in water. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Store this solution away from atmospheric carbon dioxide and renew it at least monthly.

The pH of this buffer solution at 20 °C is 9,22.

3.4 Buffer solution of potassium hydrogen phthalate and sodium hydroxide.

Dissolve $10,21 \pm 0,01$ g of potassium hydrogen phthalate ($\text{COOH}-\text{C}_6\text{H}_4-\text{COOK}$) in about 500 ml of water and add, with continuous stirring, 70,90 ml of 0,5 N standard volumetric sodium hydroxide solution.

Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Store this solution away from atmospheric carbon dioxide and renew it at least monthly.

The pH of this buffer solution at 20 °C is 5,40.

3.5 Nitrogen, free from carbon dioxide.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 pH meter fitted with a glass electrode and a calomel electrode, sensitivity : 0,05 pH unit.

4.2 Burette, 25 ml, accurate to 0,05 ml, complying with the requirements of ISO/R 385.

5 PROCEDURE

5.1 Test portion

Weigh, to the nearest 0,05 g, 50 g of the test sample.

5.2 Preparation of the test solution

Transfer the test portion (5.1) to a beaker of suitable capacity (600 ml, for example). Add approximately 300 ml of water and stir until the sample is completely dissolved. Transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

5.3 Calibration of the pH meter

Calibrate the pH meter (4.1) at $20 \pm 0,5$ °C, by means of the buffer solutions (3.3 and 3.4).

5.4 Determination

Transfer the test solution (5.2) to a dry beaker of suitable capacity. Insert the two electrodes of the pH meter (4.1) and measure the pH at $20 \pm 0,5$ °C, passing a slight flow of nitrogen (3.5) over the surface of the solution and maintaining it throughout the operations.

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If the pH value is equal to or greater than 8,5, add the hydrochloric acid standard volumetric solution (3.1) by means of the burette (4.2), in fractions of 0,25 ml, each time reading the pH after having stirred the solution for 5 to 10 s, until a pH of approximately 5,5 is obtained.

If the pH value is lower than 8,5, add the sodium hydroxide standard volumetric solution (3.2), from the burette (4.2), slowly in fractions of 0,25 ml, reading the pH value at each addition, after having stirred the solution for 5 to 10 s, until a pH of approximately 8,5 is obtained. Then add, from the burette (4.2), the same quantity of hydrochloric acid standard volumetric solution (3.1), in order to obtain the starting pH value, operating as indicated above, until a pH of approximately 5,5 is reached.

5.5 Preparation of the calibration graph

Using graph paper, plot a graph showing the pH values (10 mm = 0,1 pH unit) as ordinates and the corresponding number of millilitres of hydrochloric acid standard volumetric solution (3.1) (10 mm = 0,5 ml) as abscissae.

6 EXPRESSION OF RESULTS

From the calibration graph (5.5) read the number of millilitres of hydrochloric acid standard volumetric solution (3.1) required to achieve the variation of pH from 8 to 6. This number represents the buffer coefficient.

7 TEST REPORT

The test report shall include the following particulars :

- a) the reference of the method used;
- b) the results and the method of expression used;
- c) any unusual features noted during the determination;
- d) any operation not included in this International Standard or regarded as optional.