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**Urea for industrial use — Measurement of the variation of pH
in the presence of formaldehyde — Potentiometric method**

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FOREWORD

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International Standard 2752 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	Ireland	Sweden
Belgium	Italy	Switzerland
Bulgaria	Netherlands	Thailand
France	New Zealand	Turkey
Germany	Poland	United Kingdom
Hungary	Romania	U.S.S.R.
India	South Africa, Rep. of	

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 — Measurement of the variation of pH in the presence of formaldehyde — Potentiometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a potentiometric method for the measurement of the variation in pH caused by the addition of formaldehyde to a solution, of conventional concentration and pH, of urea for industrial use.

2 PRINCIPLE

Measurement, at $20 \pm 0,5$ °C, of the quantity of 0,01 N sodium hydroxide standard volumetric solution required to bring the pH of a solution containing 50 g of sample in 100 ml of water to a pH of 8,3 after the addition of formaldehyde.

3 REAGENTS

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

3.1 Formaldehyde solution, 350 to 370 g/l, stabilized with approximately 10 % (V/V) of methanol.

3.2 Hydrochloric acid, 0,01 N standard volumetric solution.

3.3 Sodium hydroxide, 0,01 N standard volumetric solution.

3.4 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.

3.5 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 acid,

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.

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 Calibration of the pH meter

Calibrate the pH meter (4.1) at $20 \pm 0,5$ °C, by means of the buffer solutions (3.4 and 3.5).

5.3 Preparation of the test solution

Pour 100 ml of water into a beaker of suitable capacity (400 ml, for example) and add the test portion (5.1). Warm slightly (without exceeding 30 °C) until the test portion is completely dissolved and then cool to $20 \pm 0,5$ °C. Insert the two electrodes of the pH meter (4.1) in the solution and adjust the pH to 8,3 by the addition of one of the standard volumetric solutions (3.2 or 3.3), as appropriate.

5.4 Determination

Into a beaker of suitable capacity (400 ml, for example), pour 100 ml of water and 10 ml of the formaldehyde solution (3.1). Bring the temperature to $20 \pm 0,5$ °C, insert the two electrodes of the pH meter (4.1) and adjust the pH to 8,3 by the addition of one of the standard volumetric solutions (3.2 or 3.3), as appropriate.

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Mix this solution with the test solution (5.3), cover the beaker and leave undisturbed for 5 min at $20 \pm 0,5$ °C. By means of the burette (4.2), add the standard volumetric sodium hydroxide solution (3.3) in small portions, keeping the beaker covered as much as possible. Stir continuously and read the pH value 10 to 15 s after each addition. Continue the addition until a pH value of 8,3 is obtained.

6 EXPRESSION OF RESULTS

The variation in pH is expressed, in millilitres of 0,05 N standard volumetric sodium hydroxide solution, by the formula

$$\frac{V}{5}$$

where V is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.3) used in order to adjust the pH value to 8,3.

NOTE — This result is expressed in millilitres of 0,05 N standard volumetric sodium hydroxide solution to enable a comparison to be made with the result obtained from the determination of the buffer coefficient. (See ISO 2751.)

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.