

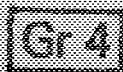
BRITISH STANDARD 2690 : Part 11 : 1971

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**METHODS OF TESTING
WATER USED
IN INDUSTRY**

**Part 11. Anionic, cationic and non-ionic
detergents and oil**

BRITISH STANDARDS INSTITUTION



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BRITISH STANDARDS INSTITUTION

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The Institution desires to call attention to the fact that this British Standard does not purport to include all the necessary provisions of a contract.

In order to keep abreast of progress in the industries concerned, British Standards are subject to periodical review. Suggestions for improvements will be recorded and in due course brought to the notice of the committees charged with the revision of the standards to which they refer.

A complete list of British Standards, numbering over 5000, fully indexed and with a note of the contents of each, will be found in the British Standards Yearbook. The BS Yearbook may be consulted in many public libraries and similar institutions.

This standard makes reference to the following British Standards:

- BS 1328. Methods of sampling water used in industry.
- BS 1427. Routine control methods of testing water used in industry.
- BS 1583. One-mark pipettes.
- BS 1739. Filter flasks.
- BS 1792. One-mark volumetric flasks.
- BS 1923. Glass filter funnels.
- BS 2021. Separating and dropping funnels.
- BS 2071. Soxhlet extractors.

British Standards are revised, when necessary, by the issue either of amendment slips or of revised editions. It is important that users of British Standards should ascertain that they are in possession of the latest amendments or editions.

The following BSI references relate to the work on this standard:
Committee references C/37, C/37/1, C/37/1/1
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CO-OPERATING ORGANIZATIONS

The Technical Committee which has been working under the supervision of the Chemical Divisional Council, and which is responsible for the preparation of this British Standard, consists of representatives from the following Government departments and scientific and industrial organizations:

- Association of Shell Boiler Makers
- British Launderers' Research Association
- *British Steel Industry
- *Chemical Industries Association
- Department of Employment and Productivity
- *Department of Trade and Industry—Laboratory of the Government Chemist
- *Electricity Council, the Central Electricity Generating Board and the Area Boards in England and Wales
- Institute of Fuel
- Institution of Marine Engineers
- Institution of Chemical Engineers
- *Institution of Gas Engineers
- Institution of Mechanical Engineers
- Institution of Municipal Engineers
- *Ministry of Defence, Navy Department
- National Coal Board
- Royal Institute of Chemistry
- Society of Laundry Engineers and Allied Trades Ltd.
- Individual firms

The Government departments and scientific and industrial organizations marked with an asterisk in the above list, together with the following, were directly represented on the Committee entrusted with the preparation of this British Standard:

- Gas Council
- Institute of Petroleum
- London Transport Executive
- Society for Water Treatment and Examination
- Water-tube Boilermakers' Association
- An individual firm

BRITISH STANDARD METHODS OF TESTING WATER USED IN INDUSTRY

Part 11. Anionic, cationic and non-ionic detergents and oil

FOREWORD

This British Standard has been prepared under the authority of the Chemical Divisional Council.

Since the original publication of BS 2690 in 1956 an addendum was published in 1960 extending the methods to cover the testing of very pure waters. Since that time various further advances have been made in the development of methods of analysing water and the standard is now being revised in separate parts so that it will in the future be simpler to revise these parts and also in order that new methods may be made available as soon as possible. The following have already been published or are due for early publication:

- Part 1. Copper and iron.
- Part 2. Dissolved oxygen, hydrazine and sulphite.
- Part 3. Silica and phosphate.
- Part 4. Aluminium, calcium, magnesium and fluoride.
- Part 5. Alkalinity, acidity, pH value and carbon dioxide.
- Part 6. Chloride and sulphate.
- Part 7. Nitrite, nitrate and ammonia (free, saline and albuminoid).
- Part 8. Cyclohexylamine, morpholine and long-chain fatty amines.
- Part 9. Appearance (colour and turbidity), odour, suspended and dissolved solids and electrical conductivity.
- Part 10. Sodium, potassium and lithium.

NOTE. The coherent system of units adopted by ISO, known as the *Système International d'Unités* (SI) has been used in this part of the revision. The siemens (S) is the name given to the unit formerly called the 'mho' (reciprocal ohm), and conductivities are accordingly expressed in terms of microsiemens per metre ($\mu\text{S/m}$), where

$$1 \text{ micromho/cm} \equiv 100 \mu\text{S/m}$$

Also, wavelengths are expressed in terms of nanometres (nm) rather than 'millimicrons' ($\text{m}\mu$); these units are equivalent.

1. GENERAL

1.1 Reagents. All reagents shall be of recognized analytical reagent quality unless otherwise specified.

1.2 Apparatus. British Standards relating to apparatus used for carrying out the tests in this British Standard are listed in Appendix A.

1.3 Water. Subject to the special requirements of certain methods, pure water having a conductivity of less than $50 \mu\text{S/m}$ at 20°C when freshly prepared and measured out of contact with air is to be used whenever water is required.

This can be achieved by treating water with the correct grades of ion exchange resin.

1.4 Ranges. Where the range of amounts of the substance being determined is stated at the beginning of a test it is usually given in either micrograms (μg) or milligrams (mg); occasionally the range is given in p.p.m., i.e. mg/litre. The procedures described for the various tests cover the stated ranges, but may in certain cases be modified by taking a smaller volume of the sample and diluting with water or by taking a larger volume of sample and evaporating. The test portion shall then be adjusted to the appropriate volume before proceeding with the determination.

1.5 Sampling. It is important to obtain a representative sample. Appropriate methods of sampling are described in BS 1328*.

1.6 Interferences. It should be realized that although the methods given will be applicable in almost all cases, occasionally some substances may be present which will render the method unsuitable.

1.7 Colorimetric methods. It is recommended that the spectrophotometer used for colorimetric determination should have a band width not exceeding 1 % of the wavelength at which the measurement is carried out.

2. ANIONIC AND CATIONIC DETERGENTS

2.1 Anionic detergents

2.1.1 Range. The range is up to 100 μg .

2.1.2 Maximum size of sample. The maximum size of the sample is 100 ml.

2.1.3 Introduction. Anionic detergents react with methylene blue to form a coloured salt which is soluble in chloroform. The result is expressed in terms of the actual anionic detergent in the sample, if known, or in terms of sodium dioctyl sulphosuccinate if the type of anionic detergent present is not known.

2.1.4 Interferences. Under the conditions described in the method the presence separately, of

chloride (Cl^-) 4.5 g

nitrate (NO_3^-) 1.5 g

in the test solution does not cause significant interference.

Low results are obtained on the filtrates from waters containing suspended solids such as sand, chalk or boiler ash, as adsorbed anion active material is retained in the residue.

2.1.5 Reagents. The following reagents are required for anionic detergents:

(1) *Chloroform*.

(2) *Sulphuric acid*, N, dilute 5N reagent solution 1+4.

*BS 1328, 'Methods of sampling water used in industry'.

(3) *Sulphuric acid*, 0.25N, dilute 5N reagent solution 1 + 19.

(4) *Methylene blue*, 0.1 g/l solution. Dissolve 0.020 g methylene blue B.P. in 150 ml of N sulphuric acid. Add 0.040 g of sodium dihydrogen orthophosphate dihydrate and 0.200 g of sodium arsenite. Stir to dissolve. Dilute to 200 ml with N sulphuric acid.

Transfer the solution to a 500 ml separating funnel and wash with a 20 ml portion of chloroform. Reject the chloroform layer. Wash with two further 20 ml portions of chloroform and reject the washings.

(5) *Wash solution*. Dissolve 50 g sodium dihydrogen phosphate dihydrate in 800 ml of 0.25N sulphuric acid. Dilute to 1000 ml with 0.25N sulphuric acid. Mix well.

(6) *Anionic detergent standard solutions*

a. *Anionic detergent stock solution*. Dissolve 1.00 g of the appropriate anionic detergent or, if this is not known, 1.00 g of sodium dioctyl sulphosuccinate* in water in a 1000 ml one-mark volumetric flask and make up to the mark. Mix well.

b. *Anionic detergent working solution*. Pipette 10.00 ml of the stock solution into a 1000 ml one-mark volumetric flask and make up to the mark with water. Mix well. Prepare this solution as and when required.

1 ml $\equiv$ 10 μ g of anionic detergent.

2.1.6 Preparation of calibration graph. To a series of 250 ml separating funnels add by pipette 0, 2, 4, 6, 8 ml and 10 ml of the anionic detergent working solution. Treat each solution as follows:

Dilute to 100 ml with water and add 4 ml of methylene blue solution. Mix well. Add 10 ml of chloroform, shake the funnel for 30 s and allow the two phases to separate. Run the chloroform layer into a 100 ml separating funnel. Re-extract the solution with two further 5 ml portions of chloroform and combine the extracts in the 100 ml separating funnel.

Add 10 ml of the wash solution to the combined extracts in the 100 ml separating funnel, shake the funnel and allow the two phases to separate. Run the chloroform layer into a second 100 ml separating funnel. Wash the chloroform layer with two further 10 ml portions of the wash solution. Reject the washings.

Filter the washed chloroform layer through a filter funnel containing a plug of glass wool into a 25 ml one-mark volumetric flask and dilute to the mark with chloroform. Mix well.

Measure the optical density of each chloroform solution at a known temperature between 20 °C and 25 °C in a spectrophotometer at the wavelength corresponding to maximum absorption (approximately 625 nm but

*A suitable grade is obtainable under the trade name 'Manoxol OT'.