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Water quality — Adsorption of substances on activated sludge — Batch test using specific analytical methods

Qualité de l'eau — Adsorption des substances sur la boue activée — Essai de lot utilisant des méthodes analytiques spécifiques

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 18749 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 5, *Biological methods*.

Introduction

This test is used as a screening test to determine the degree of adsorption of substances on activated sludge or primary sludge in waste water treatment plants. General information on the adsorption and desorption of test compounds may also be obtained by other tests (see e.g. Reference [5] in the Bibliography).

Water quality — Adsorption of substances on activated sludge — Batch test using specific analytical methods

WARNING — Activated sludge and sewage contain potentially pathogenic organisms. Take appropriate precautions when handling them. Handle with care toxic test compounds and those whose properties are unknown.

1 Scope

This International Standard specifies a screening test method for the determination of the degree of adsorption of substances on to the activated sludge or primary sludge in a waste water treatment plant.

The conditions described in this International Standard normally correspond to the optimum conditions for the adsorption to occur at the chosen activated-sludge concentration and water hardness during the test period.

The method applies to substances for which an analytical method with sufficient accuracy is available and which, under the conditions of the test and at the test concentration used,

a) are water-soluble;

b) or, if only slightly water-soluble, allow sufficiently stable suspensions, dispersions or emulsions to be prepared;

c) are not significantly removed from the test solution during the test by known abiotic processes such as stripping or foaming;

d) do not deflocculate activated sludge;

e) are not readily biodegradable (for a discussion of biodegradability, see ISO/TR 15462).

An important parameter that can influence the reliability of the test results is the stability of the test compound during the test. If no information on the stability is available, it is recommended that this be checked before the test. If any transformation (e.g. due to hydrolysis) is observed, it is recommended that the degree of adsorption of the transformation products be determined, if possible. Since biodegradability of the test compound may also lead to an incorrect assessment of the degree of adsorption, it is recommended that the biodegradability be investigated in advance using standard biodegradation tests which are preferably based on oxygen consumption or on carbon dioxide production and in which adsorption has no influence on the test result. If biodegradation cannot be excluded, sterilized sludge may be used (see Clause 7). There is generally no need to carry out adsorption tests on substances which are readily biodegradable as they are sufficiently removed biologically in waste water treatment plants. Substances which are easily adsorbed on activated sludge in waste water treatment plants are preferably removed by adsorbing them in sludge digesters and degrading them anaerobically. For such substances, high adsorption may be a reason for carrying out anaerobic biodegradation tests. An overview of standardized biodegradation tests is given in ISO/TR 15462.

The test compound concentrations used in this method are usually very low and therefore no negative effects are to be expected on the capacity of the activated sludge to adsorb even toxic test compounds. When there is any doubt, it is recommended that microscopic investigations of the flocs and suitable toxicity tests such as that specified in ISO 8192 be carried out.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
activated sludge
biomass and inert matter produced in the aerobic treatment of waste water by the growth of bacteria and other microorganisms in the presence of dissolved oxygen

2.2
degree of adsorption on activated sludge
percentage of a test compound eliminated by any process except biodegradation and stripping under the conditions of a specific aqueous batch test by activated or primary sludge, determined by comparing the concentration at the beginning of the test with that at the end

2.3
distribution coefficient
the ratio of the concentration of a test compound in the sludge to its concentration in the aqueous phase

2.4
concentration of suspended solids in an activated sludge
amount of solids obtained from a known volume of activated sludge by filtration or centrifugation under specified conditions and drying at about 105 °C to constant mass

3 Principle

The method determines the degree of adsorption, and, optionally, the distribution coefficient and mass balance, of water-soluble organic substances on activated or primary sludge using an aqueous batch-test system. The test mixture usually comprises an inorganic-salt test medium, activated sludge and the test compound. The hardness of the test medium, the concentration of suspended solids in the activated sludge and the amount of test compound added are specified to simulate real conditions of waste water treatment plants. Thus, the initial concentration of the test compound is usually as low as possible within the sensitivity range of the analytical methods available.

The concentration of the test compound is measured using substance-specific analytical methods at the beginning, during and at the end of the test (normally 24 h). The measured values are used as the basis for calculating the degree of adsorption and, optionally, the distribution coefficient and the mass balance. The measurement of elimination in the test vessels generally allows no direct differentiation between adsorption and other elimination mechanisms such as complex formation, flocculation, precipitation, sedimentation or biodegradation. More information can be obtained by using an abiotic elimination control without activated sludge, sterilized sludge and/or by determining biodegradation of the test compound with appropriate tests.

4 Reagents and materials

Use only reagents of recognized analytical grade.

4.1 Water

Use distilled or de-ionized water containing less than 1 mg of dissolved organic carbon (DOC) per litre.

4.2 Test medium

4.2.1 Preparation of solution A

Anhydrous potassium dihydrogen phosphate (KH_2PO_4)	8,5 g
Anhydrous dipotassium hydrogen phosphate (K_2HPO_4)	21,75 g
Disodium hydrogen phosphate dihydrate ($Na_2HPO_4 \cdot 2H_2O$)	33,4 g
Dissolve in water (4.1) and make up to	1 000 ml

It is recommended that this buffer solution be checked by measuring its pH. If it is not about 7,4 ± 0,5, prepare a new solution.

4.2.2 Preparation of solution B

Dissolve 12,3 g of magnesium sulfate heptahydrate ($MgSO_4 \cdot 7H_2O$) in water (4.1) and make up to 1 000 ml.

4.2.3 Preparation of solution C

Dissolve 29,4 g of calcium chloride dihydrate ($CaCl_2 \cdot 2H_2O$) in water (4.1) and make up to 1 000 ml.

4.2.4 Preparation of solution D

Dissolve 22,4 g of sodium hydrogen carbonate ($NaHCO_3$) in water (4.1) and make up to 1 000 ml.

4.2.5 Preparation of test medium

The test medium is prepared at the beginning of each test by adding the correct amounts of stock solutions A to D to the test compound as described in Clause 7. It is important to follow the instructions in Clause 7 to avoid precipitation of the salts. The hardness of the test medium is adjusted to a value which will depend on the usual water hardness in the region concerned or on the purpose of the test. Mixing 10 ml of each of solutions A to D and making up to 1 000 ml with water (4.1) will give a hardness of 2,5 mmol/l (80 mg/l of Ca^{2+}, 12 mg/l of Mg^{2+}) and a hydrogen carbonate (HCO_3^-) concentration of 162 mg/l, which is typical for many waste waters. If required, another hardness may be used. In this case, change the amounts of solutions B and C added, bearing in mind that an extra 1 ml of solution B corresponds to an increase in Mg^{2+} concentration of 0,05 mmol/l and an extra 1 ml of solution C to an increase in Ca^{2+} concentration of 0,2 mmol/l. Indicate the hardness used and the Ca/Mg ratio clearly in the test report.

If a test compound influences the pH of the mixture significantly at the chosen concentration (e.g. if the pH is outside 6,0 to 9,0), an increase in the buffer capacity of the test medium may be required. In such cases, add more of solution A, e.g. 100 ml instead of 10 ml.

Solutions A to D may be stored for up to 6 months in the dark at room temperature.

4.3 Preparation of stock solutions of test compound and reference material

Dissolve the test compound in water (4.1) or in test medium (4.2) at a suitable concentration. Suitable means a concentration which simulates real environmental conditions (e.g. of waste water), but which is high enough to allow a sufficiently accurate quantitative determination of the test compound remaining at the end of the test to be made, using the intended analytical procedure, even after elimination of about 90 % by adsorption. In the case of substances which are toxic to activated sludge and might for this reason influence the adsorptive capacity of the sludge and hence the test result (see Clause 1), the concentration shall be low enough to avoid this effect. The concentration may also be governed by the intended purpose of the test, for example the simulation of an exposure scenario at a given environmental concentration. If there is no special request or other information, a concentration of 1 mg/l to 5 mg/l is appropriate for substance-specific analysis and 40 mg of DOC/litre in the case of DOC analysis (see Clause 7). Prepare the stock solution freshly before use or store it, depending on the stability of the test compound, in the dark at about 4 °C.

It is generally not necessary to test slightly water-soluble test compounds at levels above their solubility in water, as they are mechanically removed in waste water treatment plants, e.g. by sedimentation. In such cases, therefore, the concentration of the solution has to be below the solubility in water under the test conditions, but high enough to allow a sufficiently accurate determination at the end of the test, even after elimination of about 90 % by adsorption. If substances pass into waste water treatment plants in the form of stable emulsions or dispersions and hence enter the environment in this form, they can be tested in this form if available.

To decide whether a test compound is sufficiently water-soluble, it is recommended that a sample be taken from the freshly prepared stock solution and the total organic carbon (TOC) determined directly and, after