Amenability of Sewage Sludge to Anaerobic Digestion 1977

Methods for the Examination of Waters and Associated Materials

Contents

London Her Majesty's Stationery Office

Warning to users

The analytical procedures given in the booklet should only be carried out by competent trained persons, with adequate supervision when necessary. Local Safety Regulations must be observed. Laboratory procedures should be carried out only in a properly equipped laboratory. Field operations should be conducted with due regard to possible local hazards, and portable safety equipment should be carried. Care should be taken against creating hazards for others. Lone working, whether in the laboratory or field, should be discouraged. Reagents of adequate purity must be used, along with properly maintained apparatus and equipment of correct specification. Specifications for reagents, apparatus and equipment are given in manufacturers' catalogues and various published standards. If contamination is suspected, reagent purity should be checked before use.

There are numerous handbooks on first aid and laboratory safety. One such publication is 'Code of Practice for Chemical Laboratories' issued by the Royal Institute of Chemistry, London. Where the Committee has considered that a special unusual hazard exists, attention has been drawn to this in the text so that additional care might be taken beyond that which should be exercised at all times when carrying out analytical procedures. It cannot be too strongly emphasised that prompt first aid, decontamination, or administration of the correct antidote can save life, but that incorrect treatment can make matters worse. It is suggested that both supervisors and operators be familiar with emergency procedures before starting even a slightly hazardous operation, and that doctors consulted after any accident involving chemical contamination, ingestion, or inhalation, be made familiar with the chemical nature of the injury, as some chemical injuries require specialist treatment not normally encountered by most doctors. Similar warning should be given if a biological or radiochemical injury is suspected. Some very unusual parasites, viruses and other micro-organisms are occasionally encountered in samples and when sampling in the field. In the latter case, all equipment including footwear should be disinfected by appropriate methods if contamination is suspected.

The best safeguard is a thorough consideration of hazards and the consequent safety precautions and remedies well in advance. Without intending to give a complete checklist, points that experience has shown are often forgotten include: laboratory tidiness, stray radiation leaks (including ultra violet), use of the correct protective clothing or goggles, removal of toxic fumes and wastes, containment in the event of breakages, access to taps, escape routes, and the accessibility of the correct and properly maintained first aid, fire-fighting, and rescue equipment. If in doubt it is safer to assume that a hazard may exist and take reasonable precautions than to assume that no hazard exists until proved otherwise.

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About this series

This booklet is one of a series intended to provide recommended methods for the determination of water quality. In the past, the Department of the Environment and its predecessors, in collaboration with various learned societies, has issued volumes of methods for the analysis of water and sewage culminating in '*Analysis of Raw, Potable and Waste Waters*'. These volumes, inevitably, took some years to prepare, so that they were often partially out of date before they appeared in print. The present series will be published as individual methods, thus allowing for the replacement or addition of methods as quickly as possible without need of waiting for the next edition. The rate of publication will also be related to the urgency of requirement for that particular method, tentative methods being issued when necessary. The aim is to provide as complete and up to date a collection of methods and reviews as is practicable, which will, as far as possible, take into account the analytical facilities available in different parts of the Kingdom, and the quality criteria of interest to those responsible for the various aspects of the water cycle. Because both needs and equipment vary widely, where necessary, a selection of methods may be recommended for a single determinand. It will be the responsibility of the users—the senior analytical chemist, biologist, bacteriologist etc, to decide which of these methods to use for the determination in hand. Whilst attention of the user is drawn to any special known hazards which may occur with the use of any particular method, responsibility for proper supervision and the provision of safe working conditions must remain with the user.

The preparation of this series and its continuous revision is the responsibility of the Standing Committee of Analysts (to review Standard Methods for Quality Control of the Water Cycle). The Standing Committee of Analysts is one of the joint technical committees of the Department of the Environment and the National Water Council. It has nine Working Groups, each responsible for one section or aspect of water cycle quality analysis. They are as follows:

1.0 General principles of sampling and accuracy of results
2.0 Instrumentation and on-line analysis
3.0 Empirical and physical methods
4.0 Metals and metalloids
5.0 General non-metallic substances
6.0 Organic impurities
7.0 Biological methods
8.0 Sludge and other solids analysis
9.0 Radiochemical methods.

The actual methods etc are produced by smaller panels of experts in the appropriate field, under the overall supervision of the appropriate working group and the main committee. The names of those associated with this method are listed inside the back cover.

Publication of new or revised methods will be notified to the technical press, whilst a list of Methods in Print is given in the current HMSO Sectional Publication List No 5, and the current status of publication and revision will be given in the biennial reports of the Standing Committee of Analysts.

TA DICK
Chairman

LR PITTWELL
Secretary

4 *November* 1976

Amenability of Sewage Sludge to Anaerobic Digestion (1977 version)

1 Introduction

Anaerobic digestion processes for the treatment of sewage sludge depend on the activity of methane-producing bacteria which are particularly susceptible to inhibition by toxic substances present in the sludge. It is recommended that small-scale batch digestion tests, which indicate the amenability of sewage sludge to anaerobic digestion (a qualitative measure of the toxicity of the sludge), be carried out as an essential part of the laboratory control of the anaerobic digestion process. The recommended batch digestion test contains two procedures; (i) a full test which is intended primarily for investigational purposes such as digester malfunction, weekly composite raw sludges and trade effluent testing, and (ii) a rapid test which is intended primarily for the routine daily screening of the raw sewage sludge to determine if it is suitable for feeding to the digester.

2 Performance Characteristics of the Method

2.1	Parameter determined	Amenability and/or toxicity of sewage sludges to anaerobic digestion (indicative of the absence or presence of substances which inhibit anaerobic digestion).
2.2	Types of sample	Raw sewage sludges (for trade effluents see Section 10).
2.3	Nature of test	Qualitative.
2.4	Basis of the method	Comparison of the volume of gas produced by incubation of mixtures of the sample sludge and a digesting seed sludge with that produced by incubation of similar mixtures of a reference raw sludge and the same digesting seed sludge.
2.5	Interferences	Chemically unstable or biodegradable toxic substances may give false results (see Section 4).
2.6	Time required for the test	(i) *Full test*. Total time of up to 6½ days; Operator time of 4 hours per sample. (ii) *Rapid test*. Total time of up to 1 day; Operator time of 1 hour per sample.

3 Principle

The full test and the rapid test are both based on the same principle. The full test is based on the method developed at Water Research Centre (Stevenage Laboratory)[1] and the rapid test is based on the method developed at Norwich Sewage Works [2].

The sample sludge and the reference raw sludge must contain the same amount of digestible material. In the full test the total solids content and the volatile solids content of both sludges are determined and the volatile solids content of the reference raw sludge is adjusted by dilution or removal of supernatant liquor to that of the sample sludge prior to incubation. In the rapid test the total solids contents of the two sludges are estimated visually and the total solids content of the reference raw sludge is visually adjusted to approximately that of the sample sludge prior to incubation. During the incubation period the total solids contents of the sample sludge and the adjusted reference raw sludge are determined and a correction applied at the end of the test.

A factor is calculated by comparing the volumes of gas obtained by incubating a series of mixtures of the sample sludge with an actively digesting seed sludge with those obtained by incubating a similar series of mixtures of a reference raw sludge with the same actively digesting seed sludge. The factor calculated in this manner is called the Amenability Factor.

4 Interferences

No specific tests of interfering substances have been made. Toxic substances which are chemically unstable or biodegradable may give false results because they could indicate toxicity in the batch tests but may not be toxic in a full size digester. Examples of such possible interfering substances are free chlorine, chromates and high concentrations of propionic acid.

Care must be taken to carry out the tests in a part of the laboratory where chlorinated hydrocarbon solvents are not in use, since minute traces of chlorinated hydrocarbon vapours (eg chloroform) can seriously inhibit gas production.

5 Hazards

Samples of sludge should be transported in wide necked non-rigid polyethylene containers fitted with loose fitting caps or stoppers. Particular care should be taken when transporting sludges which are in a state of active digestion, due to fire and explosion hazards from generated gases. During the test ensure that all lines carrying gas are free from obstruction to avoid pressurising glass vessels. The tests should be carried out in a well ventilated laboratory to avoid the possibility of forming explosive mixtures of methane and air.

6 Reagents

6.1 Reference raw sludge

A reliable supply of non-toxic raw sludge of total solids content greater than 4% is required. A suitable source of this is a small rural sewage works which receives little or no trade effluent. Such sludges may contain high concentrations of anionic synthetic detergents. Care should be taken to collect the raw sludge on days when it is known that the detergent concentration is low or, alternatively, the detergent should be partially neutralised by the addition of long-chain aliphatic amines[3]. It is also desirable (but not always practicable) that the reference raw sludge should have a higher total solids content than the sample sludges. The reference raw sludge should be obtained freshly each week and stored at a temperature between 0 and 4°C.

6.2 Digesting seed sludge

A digesting seed sludge of uniform activity is required. This is obtained from a continuous breeder digester operated in the laboratory at 32±1°C. This digester must be fed daily with a suitable volume of the reference raw sludge so as to give a retention time in the digester of about 25 days and it follows that the capacity of the digester needs to be at least 25 times the volume of digesting seed sludge required per day.

It is recommended that a second identical laboratory continuous breeder digester is operated in parallel in case the first malfunctions or is accidentally damaged.

7 Apparatus

7.1 Batch digestion (incubation) apparatus

This apparatus is constructed in the laboratory. A suitable apparatus of the type used at the Water Research Centre (Stevenage Laboratory) is shown diagrammatically in Figure 1. The following approximate dimensions are recommended:

Full test	Incubation bottles	500-ml capacity
	Gas collecting tubes	110 cm length, 5 cm internal diameter, 1500 ml useable capacity
Rapid test	Incubation bottles	150-ml capacity
	Gas collecting tubes	150 cm length, 2 cm internal diameter 350 ml useable capacity

The gas collecting tubes should be calibrated in volume (ml) or height (mm) with the zero mark at the top of the tube. Self-adhesive graduations are suitable. The gas is collected over water acidified with hydrochloric acid to a pH value of 4·0.

7.2 Water bath

A water bath with adequate circulation to maintain a temperature of 32±1°C in all parts of the bath is required.

7.3 Vacuum pump

A water ejector vacuum pump is used to evacuate methane from the apparatus at the end of the test.

7.4 Glassware

Measuring cylinders are preferred to pipettes for measuring out volumes of sludge. With the thicker sludges care should be taken to ensure that the measured volume is completely dispensed.

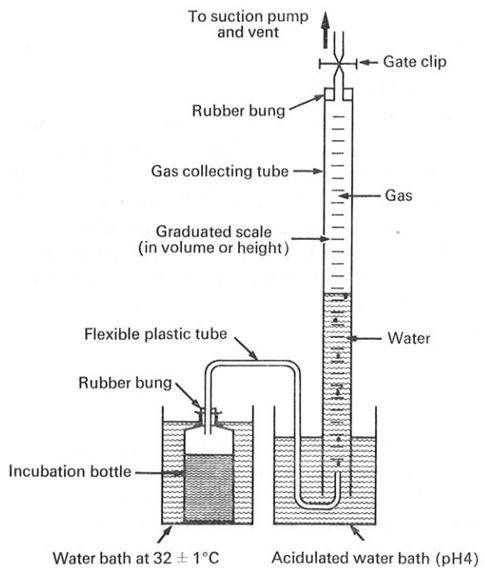

Figure 1 Batch digestion apparatus (side view—not to scale)

8 Test Procedure

8.1 Full Test

Step	Experimental Procedure	Notes
	Standardization of sludges	
8.1.1	Determine the total solids content and the volatile solids content of the sample sludge and the reference raw sludge by suitable methods (see appropriate methods in this series).	
8.1.2	Adjust the volatile solids content of the reference raw sludge to that of the sample sludge by the addition of the appropriate volume of water to, or by the removal of an appropriate volume of supernatant liquor from, the reference raw sludge (notes a and b). Mix the adjusted sludge.	(a) It is desirable that the reference raw sludge has the higher volatile solids content so that an addition of water is made. (b) If it is necessary to remove supernatant liquor, a centrifuge is useful.
	Incubation of sludge mixtures	
8.1.3	Warm the water bath to 32±1°C.	
8.1.4	Label six incubation bottles, 1a to 3b, as in Table 1, and transfer to them the volumes of sample and reference raw sludges shown in this table (note c). Place the incubation bottles in the warm bath for 15–20 min to allow the sludges to warm up to 32±1°C.	(c) To dispense representative sub-samples of sludges, shake the bulk sample vigorously in a closed bottle and then rapidly measure the required volume into a measuring cylinder.
8.1.5	Transfer the volumes of digesting seed sludge shown in Table 1 to the appropriate incubation bottles (note c). Connect the incubation bottles to the gas-collecting tubes, gently shake or swirl the bottles and wait about 15 min for the temperatures to equilibrate (note d). Adjust the water level in the gas-collecting tubes to the zero mark by suction. Close the gate-clip and note the time, this is the start of the incubation time (note e).	(d) Some gas will be displaced into the collecting tube indicating a gas-tight seal.
8.1.6	After the commencement of incubation and for up to 5 days thereafter, gently shake or swirl the incubation bottles twice daily (at the beginning and end of the working day), wait for about 2 min and read the volume or height of gas produced from each incubation bottle.	(e) The total time for a full test may be up to $6\frac{1}{2}$ days made up of approximately $1\frac{1}{2}$ days determining the total and volatile solids content of the sample sludge and preparing the apparatus followed by up to 5 days incubation.
	Calculation of results	
8.1.7	On one piece of graph paper plot the cumulative gas volumes against incubation time for each incubation mixture.	
8.1.8	Select a time when the rate of gas production is constant (note f) for each of the mixtures (usually between 24 and 72 hours) and from the graph read the volumes of gas produced V_{1a}, V_{1b}, V_{2a}, V_{2b}, V_{3a} and V_{3b} at the selected time.	(f) This is the time when the concentration of substrate is not rate limiting.
8.1.9	Calculate the amenability factors A_1, A_2, A_3 from:	

$$A_1 = \frac{V_{1b} \times 100}{V_{1a}}$$

$$A_2 = \frac{V_{2b} \times 100}{V_{2a}}$$

$$A_3 = \frac{V_{3b} \times 100}{V_{3a}}$$

Table 1

Incubator bottle number	Volume of reference raw sludge (ml)	Volume of sample sludge (ml)	Volume of digesting seed sludge (ml)	Total Volume (ml)	Ratio of volumes of raw sludge to digesting sludge
1a	35±1	Nil	350±2	385	1:10
1b	Nil	35±1	350±2	385	1:10
2a	90±1	Nil	270±2	360	1:3
2b	Nil	90±1	270±2	360	1:3
3a	120±1	Nil	240±2	360	1:2
3b	Nil	120±1	240±2	360	1:2
4a*	180±2	Nil	180±2	360	1:1
4b*	Nil	180±2	180±2	360	1:1

*As the proportion of digesting sludge decreases, the test becomes more sensitive to inhibition and there is a greater risk of overloading. Sometimes when the toxicity is low, tests 4a and 4b may give useful information on toxicity.

8.2 **Rapid Test**

Step	Experimental Procedure	Notes
	Standardization of sludges	
8.2.1	Visually compare the consistency of the sample sludge and the reference raw sludge. If there is any obvious difference in the consistency adjust a portion of the reference raw sludge to the same approximate consistency as the sample sludge by addition of the appropriate volumes of water or removal of the appropriate volume of supernatant liquor (notes g and h). Mix the adjusted sludge.	(g) It is desirable that the reference raw sludge has the higher total solids content so that an addition of water is made. (h) If it is necessary to remove supernatant liquor, a centrifuge is useful.
8.2.2	Determine the total solids content of the sample sludge and the adjusted reference raw sludge by a suitable method (see appropriate method in this series). (note j.). Let the total solids contents be S_s and S_r respectively.	(j) These determinations are carried out in parallel with the incubation procedure.
	Incubation Procedure	
8.2.3	Warm the water bath to 32±1°C.	
8.2.4	Transfer to each of two labelled (1a and 1b) incubation bottles 40±1ml of the reference raw sludge and to each of a further two labelled (2a and 2b) incubation bottles 40±1ml of the sample sludge (note k). Place the incubation bottles in the water bath for 15–20 min to allow the sludges to warm up to 32±1°C.	(k) To dispense representative sub-samples of sludges, shake the bulk sample vigorously in a closed bottle and then rapidly measure the required volume into a measuring cylinder.
8.2.5	Add 80±1ml of digesting seed sludge to each incubation bottle (note k). Connect the incubation bottles to the gas collecting tubes, gently shake or swirl the bottles and wait about 15 min for temperatures to equilibrate (note l). Adjust the water level in the gas collecting tubes to the zero mark by applying suction. Close the gate-clip and note the time.	(l) Some gas will be displaced into the collecting tube indicating a gas-tight seal.
8.2.6	After 22 hours gently shake or swirl the incubation bottles, wait about 2 min and read the volume or height of gas produced for each incubation bottle and let the volume be V_{1a}, V_{1b}, V_{2a} and V_{2b}.	

Step	Experimental Procedure	Notes
	Calculation of Results	
8.2.7	Calculate the average volumes of gas produced from the reference and sample sludges V_1 and V_2 respectively: $V_1=\frac{V_{1a}+V_{1b}}{2}$ $V_2=\frac{V_{2a}+V_{2b}}{2}$	
8.2.8	Calculate the apparent amenability factor A_a from $A_a=\frac{V_2\times 100}{V_1}$	
8.2.9	Calculate the corrected amenability factor A_c from $A_c=A_a\times\frac{S_r}{S_s}$	
8.2.10	Calculate the actual amenability factor, A, from $A=\frac{A_a+A_c}{2}$ (note m)	(m) If the sludges have been correctly matched in solid content A_a and A_c should be of approximately equal value.

9 Interpretation of Results

9.1 **Full Test**

For a non-toxic sample sludge the amenability factors A_1, A_2 and A_3 will be close to 100. If the sludge is slightly toxic A_3 will drop significantly below 100. If the sludge is highly toxic both A_2 and A_3 will be below 100 with A_3 being lower than A_2. These amenability factors should be correlated with the performance of the full size digester to obtain an indication of the values of A_2 and A_3 that correspond to sludge toxicities likely to affect the full size digester. This correlation will vary from plant to plant.

Amenability factor A_1, provides an internal standardization of the test. If the reference sludge has been standardized correctly (see steps 8.1.1 and 8.1.2) A_1 will be close to 100. If A_1 deviates appreciably from 100 then the sample is either highly toxic or its volatile solids content produce an atypical gas yield. If the latter is the case, then standardization of the raw sludges on the basis of the volatile solids content is inappropriate and the test should be repeated using the relative rates of gas production from incubators 1a and 1b as the basis for standardization.

9.2 **Rapid Test**

For a non-toxic sample sludge the amenability factor, A, will be close to 100. Values significantly below 100 indicate the degree of toxicity of the sludge although it is advisable to check the volatile solids content of the sample sludge to confirm that it is at the normal level. The values of A should be correlated with the performance of the full size digester.

9.3 **General Comments**

Amenability factors significantly greater than 100 indicate a suspect reference raw sludge. The usual amount of gas produced from the incubators containing the reference raw sludge will be found by experience. Any appreciable deviation from this amount which cannot be explained by variations in the total solids content may indicate an ailing breeder digester. A regular check of the pH, total alkalinity and volatile solids content of the breeder digester should be made.

These short term batch tests may indicate a dangerous level of toxicity to which the full size digester might become acclimatized but on the other hand the short term batch tests may be unaffected by a concentration of a substance which over a longer period proves highly toxic in the full size digester.

10 Trade Effluent Testing

An indication of the effect of receiving a trade effluent at a sewage works on the amenability of the raw sludge to digestion may be obtained by using this test. The sample sludge is obtained by mixing the trade effluent with the reference raw sludge based on a procedure described by Wimlett[4]. It may be necessary to adjust the pH value of the trade effluent so that it falls in the range 6·5—7·5. This may be effected by adding minimum volumes of sodium hydroxide or hydrochloric acid as appropriate.

The reference raw sludge is thoroughly mixed with an appropriate volume of trade effluent calculated as follows:

$$\begin{pmatrix}\text{volume of trade effluent required}\\ \text{per 100 ml of reference raw sludge}\end{pmatrix} = \frac{100 \times \text{Trade effluent flow } (m^3/d)}{\text{Raw sludge produced } (m_3/d)}$$

The mixture is allowed to settle and a volume of supernatant liquor equal to the amount of trade effluent added is withdrawn and discarded. The remaining sludge is used as the sample sludge in the test.

Either the full test or the rapid test may be used for trade effluent testing. The full test would usually be used to establish whether the trade effluent produces a toxic sludge prior to accepting the trade effluent at the works.

The rapid test would usually be used to screen trade effluents to ascertain whether they were responsible for or likely to cause a full size digester malfunction.

11 References

(1) *Analysis of Raw, Potable and Waste Waters*, HMSO, 1972, p. 268-272.

(2) Standing Committee of Analysts File WS/646/68, *Committee Paper* SCA/8.4/2.

(3) Swanwick JD and Shurben DG, *Wat. Pollut. Cont.* 1969, **2**, 190.

(4) Wimlett BG, *Private Communication*, SCA File WS/646/68 *Committee Paper* SCA/8.4/4.

Address for Correspondence

However thoroughly a method may be tested, there is always the possibility of a user discovering a hitherto unknown problem. Users with information on this method are requested to write to:

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The Standing Committee of Analysts
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