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Characterization of sludges — Laboratory chemical conditioning procedure

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National foreword

This British Standard is the UK implementation of EN 14742:2015.

The UK participation in its preparation was entrusted to Technical Committee EH/5, Sludge characterization.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Characterization of sludges - Laboratory chemical conditioning procedure

Caractérisation des boues - Mode opératoire de conditionnement chimique en laboratoire

Charakterisierung von Schlämmen - Laborverfahren zur chemischen Konditionierung

This European Standard was approved by CEN on 28 February 2015.

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Contents

Page

Foreword

This document (EN 14742:2015) has been prepared by Technical Committee CEN/TC 308 “Characterization of sludges”, the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2015, and conflicting national standards shall be withdrawn at the latest by October 2015.

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

This document supersedes CEN/TR 14742:2006.

The main changes with respect to CEN/TR 14742 are listed below:

a) normative references have been updated;

b) terms and definitions have been updated;

c) principle has been updated and supplemented with notes on conditioning agents;

d) procedure has been updated;

e) figures have been updated.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

This method gives a standardized procedure for the conditioning operation when selecting a conditioning product at laboratory scale and also for the production of flocculated thickened sludge for subsequent dewatering tests.

These tests may also provide details regarding energy requirements, provided information on stirring power is available.

1 Scope

The laboratory assessment of sludge dewaterability is sensitive to the operating procedure adopted for the conditioning step. No generalized ranking of products in order of effectiveness can be given since the ranking changes with the sludge type, dosage of conditioning agent, degree of shearing and dewatering device.

The scope of this European Standard applies for sludges and suspensions from:

— storm water handling;

— urban wastewater collecting systems;

— urban wastewater treatment plants;

— industrial wastewater that has been treated similarly to urban wastewater (as defined in Directive 91/271/EEC);

— water supply plants.

This method is applicable to sludge and suspensions of other origin.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 12832, *Characterization of sludges — Utilization and disposal of sludges — Vocabulary*

EN 12880, *Characterization of sludges — Determination of dry residue and water content*

EN 14701-4, *Characterization of sludges — Filtration properties — Part 4: Determination of the drainability of flocculated sludges*

EN 15933, *Sludge, treated biowaste and soil — Determination of pH*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12832 and the following apply.

3.1
chemical conditioning
mixing of a chemical product with the sludge in order to increase its thickenability/dewaterability

4 Principle

Sludge conditioned in accordance with this procedure (Clause 6) can subsequently be used in specific characterization tests in order to determine the most suitable operating conditions for each particular sludge or suspension; these tests determine particularly the required nature of the reagent, the dosage, and the sequence of adding the reagent.

Each test is performed as a batch process.

Variables and/or parameters that can have significant effects on the conditioned sludge are either chemical or physical.

Chemical parameters:

— characteristics of the sludge, e.g. pH, dry residue, loss on ignition;

— characteristics of the conditioning product, e.g. charge density, molecular weight, chemical structure;

— concentration and dosage of the chemical product;

— water used for on-site product preparation when polyelectrolytes are added.

Physical parameters:

— method of preparing the solution and its storage;

— intensity and duration of stirring;

— characteristics of the stirrer, i.e. type, dimensions, position;

— method of injecting the conditioning product into the sludge;

— (separation-) time between the end of mixing and the dewatering procedure itself.

The method of mixing the conditioning product into the sludge is particularly important since inadequate mixing, leading to poor initial dispersion or strong floc shearing, can result in very poor test performances: a large portion of the product may react instantly with particles upon first contact, without dispersing throughout the mixture.

Inorganic coagulants are generally injected into a reactor with a short residence time (1 min to 5 min) in a high shearing condition. For this purpose, the reactor is equipped with a high rotating speed stirrer that enables an instantaneous dispersion of the product and brings the high energy needed (e.g. 80 W/m^3 to 100 W/m^3).

Polyelectrolytes are injected into a reactor with a residence time of a few seconds to a few minutes. As far as mechanical degradation can occur, the shearing rate is 4 to 10 times lower than for coagulants and the energy needed lower (e.g. 40 W/m^3 to 50 W/m^3).

The conditioning procedure may concern the addition of more than one product. In that case, the addition sequence is another parameter that requires careful instructing.

5 Apparatus

5.1 Device with control and measurement of physical parameters (variable stirrer/mixer speed and time, position of mechanical stirrer depending on sludge volume, see Annex B). A magnetic stirrer is not recommended, as it does not ensure homogenous and reproducible mixing. A mechanical stirrer with 4 horizontal perpendicular blades is recommended for proper polymer dispersion and floc maturation. In case the formed flocs are very sensitive to shearing, a tilted 3-blade stirrer (see Annex B) should be used. The characteristics of mechanical stirrer shall be the same in comparative tests. Length of each impeller blade should be between 1/4 and 1/3 of the beaker diameter.

5.2 Beakers, volume 200 ml to 1 000 ml.

5.3 Volumetric pipettes or syringe or automatic polymer injection system at constant flow rate.

5.4 Analytical balance with a precision of at least 0,1 mg.

5.5 pH-meter

5.6 Chronometer, e.g. stopwatch, computer.

5.7 Apparatus for evaluation of flocculation drainability, e.g. drainage cell (see EN 14701-4).

6 Procedure

6.1 Measure the pH of the sludge to be tested according to EN 15933. If necessary, adjust the pH to meet the requirements of the conditioning agent to be used.

6.2 Measure the dry residue of the sludge to be tested according to EN 12880.

NOTE The loss on ignition could be determined for additional information. For details, see EN 15935.

6.3 Put a defined amount of sludge into a beaker. The amount of sludge should be between 25 % and 50 % of the capacity of the beaker to avoid loss of sludge during mixing and sludge height between 0,5 and 1 diameter of the cell to ensure proper mixing. It should be taken into account that the whole flocculated sludge will be transferred in a drainage cell and the quantity of dry matter deposited on the filter cloth should be between 0,25 kg/m^2 and 1,5 kg/m^2.

6.4 Adjust the position of the stirrer to the sludge volume (the stirrer should be positioned from the beaker bottom at 1/3 of the sludge volume).

6.5 Polymer addition and dispersion

Add a precise amount of conditioning agent using a volumetric pipette or syringe before stirring. Ensure proper distribution of the polymer, across the level surface of the sludge.

If the conditioning agent injection is prepared during the stirring of the sludge, add it in the shortest amount of time possible at a controlled and constant flow rate. It is recommended to report the time of the beginning of the injection (TB1) and the time at the end of the injection (TE1).

The solution of conditioning agent should be prepared according to the recommendations of suppliers and should be used the day of preparation within 3 h after preparation.

NOTE Rapid pre-selection of conditioning agents can be made by pouring the sludge back and forth between two beakers in order to check if flocs are formed.

In case of coagulant use, disperse the product at high speed (1 700 rpm) for 2 min and let the microflocs grow at 700 rpm.

In case of polymer use, the mixing speed of the horizontal 4-blade impeller should be kept constant at 700 rpm since polymer dispersion and floc maturation occur simultaneously.

In case of highly shear-sensitive flocs, it is recommended to disperse the flocculent in the sludge using an axial impeller (tilted 3-blade impeller) at high speed (1 700 rpm) for a very short time (2 s max), followed by a low speed (700 rpm) to enable floc growing.

In this case, it is useful to differentiate:

TBB1: Beginning time of polymer dispersion = TE1

TEE1: End of polymer dispersion

TBB2: Beginning of polymer maturation

TEE2: End of polymer maturation

6.6 Floc maturation

Continue stirring until polymer maturation (TEE2) is reached.

Mixing time is the variable parameter for floc growth, depending on the sludge and conditioning agent characteristics. Preliminary tests are recommended to evaluate this parameter by visual observation of floc formation without shearing or by quantitative measurement (torque measurement, particle size). Then, mixing time will be fixed and kept constant for comparative tests.

6.7 Transfer the whole flocculated sludge into a drainage cell to evaluate flocculation drainability according to EN 14701-4 and produce thickened sludge for further specific dewatering test (e.g. CST, specific resistance to filtration) immediately after the conditioning procedure.

An automatic transfer from the flocculation cell in the drainage cell as in the device described in Annex B (Bootest) is recommended to avoid operator's influence on transfer to drainage cell or on floc sampling.

6.8 Repeat steps 6.3 to 6.8 for a lower stirring speed only if flocculation does not take place.

7 Test report

The test report (example in Annex C) shall include at least the following information:

a) reference to this European Standard (EN 14742);

b) all information necessary for complete identification of the sludge sample;

c) details of sample pretreatment, if carried out;

d) details of the experimental device;

e) nature, dosage and preparation procedure of chemical agent;

f) mixing conditions;

g) results of the preparation according to 6.7 (see Annex B);

h) any detail not specified in this European Standard and any other factor which may have affected the results.

8 Precision

Validation data were obtained through an European ILT based on the “Modified Round Robin Tests” procedure developed by CEN/TC 308/WG 1, and reported in doc. CEN/TC 308 N 822.

Data were examined according to ISO 5725-2 statistical prescriptions.

Three types of sludge, i.e. digested sewage sludge, activated sewage sludge, and waterworks sludge, were used for validation trials whose full results are reported in Annex A (informative).

There was only one operating condition (polymer dosing) which was manual during the tests and could have an influence on precision of data results. All sludge types considered, average values of relative *repeatability* standard deviation were 1,8 % and average values of relative *reproducibility* standard deviation was 2,9 %. For repeatability, minimum value was 0,9 % (sludge 2A) while maximum one was 3,5 % (sludge 1A). For reproducibility, minimum value was 1,5 % (sludge 2A) while maximum one was 5,5 % (sludge 1 A).

Validation results showed to be suitable for supporting publication as an EN. The precision data given as averages should be at least reached in the analysis of sludge.

Annex A
(informative)

Results of Validation trials

Because the circulation of samples of real sludge high in organic content is not possible due to problems associated to changes in their physical characteristics during handling and transportation, the “Modified Round Robin Tests” procedure, developed by TG 3 of CEN/TC 308/WG 1, and reported in doc CEN/TC 308 N 822, was followed. With this procedure, the round robin tests are carried out through “circulation of analysts”, i.e. operators coming from the laboratories participating to the exercise meet in a common location, close to the place where samples are collected, and work there on same samples, each using the same apparatus.

Validation trials were carried out at I.F.T.S at Foulayronnes (near Agen (47) – France), on 10th-11th April, 2013 with two Bootest, each allowing tests in duplicates (see Annex B), equipped with filter cloth reference (Reference: Si030904 from Rai-Tillières manufacturer).

Trials involved a total of 15 Operators from the following 15 Laboratories/Institutions in 6 Countries: Acquedetto Pugliese (Italy), Andritz (France), Aquateam (Norway), CUTEC (Germany), DIN (Germany), Dokuz Eylul University (Turkey), IFTS (France), IRSTEA (France), Milan Polytechnic (Italy), Regional Agency Environment Protection Emily Romagne (Italy),UNI (Italy), University of Albi (France), University of Bordeaux (France), University of Liege (Belgium), Veolia (France)

The following 3 different sludge types were tested and flocculated in 3 different conditions:

— Sample 1: Activated sewage raw sludge (DS: 3,64 %, LOI: 65,5 %, pH: 6,71) sampled 9th April at Saint Pierre de Gaubert (47- France) wastewater treatment plant. The flocculation conditions (test carried out on April 10th afternoon) are:

  • 1.A: Cationic Crosslinked polymer EM 640 MBL from SNF Floerger, prepared at 5 g/L and used at a mean of 14,3 kg/ T DS (SD: 0,79 between analysts during tests) and mixed with sludge during 4 s at 700 rpm

  • 1.B: Cationic Crosslinked polymer EM 640 HIB from SNF Floerger, prepared at 5 g/L and used at a mean of 15,0 kg/ T DS (SD: 0,69 between analysts during tests) and mixed with sludge during 4 s at 700 rpm

  • 1.C: Cationic Crosslinked polymer EM 640 TRM from SNF Floerger, prepared at 5 g/L and used at a mean of 15,4 kg/ T DS (SD: 0,45 between analysts during tests) and mixed with sludge during 4 s at 700 rpm

— Sample 2: Digested sewage raw sludge (DS: 3,53 %, LOI: 65,3 %, pH: 7,90) sampled 8th April at Albi (81- France) wastewater treatment plant. The flocculation conditions (tests carried out April 11th) are:

  • 2.A: Cationic crosslinked polymer EM 640 MBL from SNF Floerger, prepared at 5 g/L and used at a mean of 35,6 kg/ T DS (SD: 0,53 between analysts during tests) and mixed with sludge during 3 s at 700 rpm

  • 2.B: Cationic structured polymer EM 640 HIB from SNF Floerger, prepared at 5 g/L and used at a mean of 35,8 kg/ T DS (SD: 0,75 between analysts during tests) and mixed with sludge during 8 s at 700 rpm

  • 2.C: Cationic structured polymer Zetag 9018 from BASF, prepared at 5 g/L and used at a mean of 24,3 kg/ T DS (SD: 0,49 between analysts during tests) and mixed with sludge during 27 s at 700 rpm

— Sample 3: Waterworks sludge (DS: 3,34 %, LOI: 56,2 %, pH: 7,74) sampled 9th April at Nerac (81-France) drinking water supply plant. The flocculation conditions (tests carried out April 11th) are:

  - 3.A: Cationic crosslinked polymer EM 440 MBL from SNF Floerger, prepared at 5 g/L and used at a mean of 25,3 kg/ T DS (SD: 0,63 between analysts during tests) and mixed with sludge during 5 s at 700 rpm

  - 3.B: Cationic structured polymer EM 440 HIB from SNF Floerger, prepared at 5 g/L and used at a mean of 25,0 kg/ T DS (SD: 0,40 between analysts during tests) and mixed with sludge during 10 s at 700 rpm

  - 3.C: Anionic linear polymer AN 934 SH from SNF Floerger, prepared at 5 g/L and used at a mean of 4,0 kg/ T DS (SD: 0,12 between analysts during tests) and mixed with sludge during 11 s at 700 rpm

All analyses for measuring solids concentration and/or dry residue were carried out at the laboratories of IFTS at Foulayronnes, the days after the interlaboratory tests.

Test results have been statistically analysed by INERIS (France) for the evaluation of precision according to ISO 5725-2. In particular, the repeatability standard deviation, and the reproducibility standard deviation were calculated. After compilation of data potential outliers were identified using ISO 5725-2. All outlier-free data were then used for calculation.

1) EM 640 MBL, EM 640 HIB, EM 640 TRM, EM 440 MBL, EM 440 HIB, AN 934 SH, are the trade names of a product supplied by SNF Floerger, France. This information is given for the convenience of users of this European standard and does not constitute an endorsement by CEN of the product named.

Equivalent products may be used if they can be shown to lead to the same results.

2) Zetag 9018 is the trade name of a product supplied by BASF, Germany. This information is given for the convenience of users of this European standard and does not constitute an endorsement by CEN of the product named.

Equivalent products may be used if they can be shown to lead to the same results.

3) Si030904 is the trade name of a product supplied by Rai-Tillières, France. This information is given for the convenience of users of this European standard and does not constitute an endorsement by CEN of the product named.

Equivalent products may be used if they can be shown to lead to the same results.

Results are presented in the following six tables.

Table A.1 — Results for ratio of mass of filtrate to sludge mass (M/M0) at *t* = 10 s

	Activated sludge			Digested sludge			Waterworks sludge		
Sludge conditioning	1A	1B	1C	2A	2B	2C	3A	3B	3C
Total number of participants	15	15	15	15	15	15	15	15	15
Replicates	2	2	2	2	2	2	2	2	2
Number of participants (outliers removed)	15	15	15	15	15	15	15	15	15
Outliers	0	0	0	0	0	0	0	0	0
Average value (%)	23,73	31,53	38,43	13,07	17,47	8,10	10,97	8,60	9,57
Mean repeatability standard deviation (%)	2,90	3,45	3,21	1,69	2,48	2,40	1,43	1,00	1,78
Mean repeatability variation coefficient (%)	9,52	8,62	6,60	12,69	6,97	24,00	9,64	7,79	12,89
Mean reproducibility standard deviation (%)	4,06	3,89	4,22	3,63	2,48	3,29	2,06	2,46	1,78
Mean reproducibility variation coefficient (%)	14,77	9,60	9,26	26,23	11,86	34,78	16,39	27,46	12,16

Table A.2 — Results for ratio of mass of filtrate to sludge mass (M/M0) at *t* = 30 s

	Activated sludge			Digested sludge			Waterworks sludge		
Sludge conditioning	1A	1B	1C	2A	2B	2C	3A	3B	3C
Total number of participants	15	15	15	15	15	15	15	15	15
Replicates	2	2	2	2	2	2	2	2	2
Number of participants (outliers removed)	15	15	15	15	15	15	15	15	15
Outliers	0	0	0	0	0	0	0	0	0
Average value (%)	38,50	45,90	49,73	23,50	29,93	17,67	21,03	21,07	17,77
Mean repeatability standard deviation (%)	3,08	2,21	2,14	1,49	3,25	2,73	3,35	1,32	2,60
Mean repeatability variation coefficient (%)	6,39	3,64	3,11	5,39	9,68	12,37	13,00	4,61	11,95
Mean reproducibility standard deviation (%)	4,69	2,63	241	6,09	3,36	3,37	4,37	2,12	3,00
Mean reproducibility variation coefficient (%)	10,79	4,61	3,78	25,51	8,18	15,60	17,48	9,05	13,36

Table A.3 — Results for ratio of mass of filtrate to sludge mass (M/M0) at *t* = 90 s

	Activated sludge			Digested sludge			Waterworks sludge		
Sludge conditioning	1A	1B	1C	2A	2B	2C	3A	3B	3C
Total number of participants	15	15	15	15	15	15	15	15	15
Replicates	2	2	2	2	2	2	2	2	2
Number of participants (outliers removed)	15	15	15	15	15	15	15	15	15
Outliers	0	0	0	0	0	0	0	0	0
Average value (%)	48,07	52,17	53,97	35,40	41,17	26,80	33,73	31,63	28,93
Mean repeatability standard deviation (%)	1,75	1,17	1,02	2,48	3,48	2,21	2,70	1,91	2,52
Mean repeatability variation coefficient (%)	3,00	1,90	1,49	5,51	6,81	6,87	6,51	4,36	6,88
Mean reproducibility standard deviation (%)	2,98	1,37	1,07	6,28	3,48	5,13	4,34	3,78	3,81
Mean reproducibility variation coefficient (%)	5,64	2,10	1,46	17,04	4,79	18,22	11,57	11,15	11,63

Table A.4 — Results for 50 % (final mass of filtrate) recovery time

	Activated sludge			Digested sludge			Waterworks sludge		
Sludge conditioning	1A	1B	1C	2A	2B	2C	3A	3B	3C
Total number of participants	15	15	15	15	15	15	15	15	15
Replicates	2	2	2	2	2	2	2	2	2
Number of participants (outliers removed)	15	15	15	15	15	15	15	15	15
Outliers	0	0	0	0	0	0	0	0	0
Average value (s)	11,60	7,90	5,70	22,90	15,77	28,87	23,03	26,53	23,87
Mean repeatability standard deviation (s)	2,14	1,17	0,95	2,99	2,17	2,42	3,33	2,45	3,71
Mean repeatability variation coefficient (%)	13,99	12,90	13,31	10,60	11,47	7,79	13,48	7,59	12,81
Mean reproducibility standard deviation (s)	2,89	1,43	1,33	5,92	2,48	4,45	6,13	4,27	3,81
Mean reproducibility variation coefficient (%)	21,25	14,79	20,11	24,12	12,39	14,25	24,57	14,69	11,57

Table A.5 — Results for cake dryness

	Activated sludge			Digested sludge			Waterworks sludge		
Sludge conditioning	1A	1B	1C	2A	2B	2C	3A	3B	3C
Total number of participants	15	15	15	15	15	15	15	15	15
Replicates	2	2	2	2	2	2	2	2	2
Number of participants (outliers removed)	15	15	15	15	15	15	15	15	15
Outliers	0	0	0	0	0	0	0	0	0
Average value (%)	7,92	8,04	8,05	5,81	6,28	5,29	13,45	12,77	12,98
Mean repeatability standard deviation (%)	0,15	0,13	0,17	0,14	0,21	0,30	0,47	0,44	0,42
Mean repeatability variation coefficient (%)	1,72	1,17	1,55	2,13	2,82	4,49	3,02	2,71	2,65
Mean reproducibility standard deviation (%)	0,18	0,26	0,27	0,44	0,29	0,44	1,50	1,59	1,15
Mean reproducibility variation coefficient (%)	1,92	2,98	3,00	7,35	3,95	7,20	10,89	12,18	8,54

Table A.6 — Results for filtrate suspended matters

	Activated sludge			Digested sludge			Waterworks sludge		
Sludge conditioning	1A	1B	1C	2A	2B	2C	3A	3B	3C
Total number of participants	15	15	15	15	15	15	15	15	15
Replicates	2	2	2	2	2	2	2	2	2
Number of participants (outliers removed)	15	15	15	15	15	15	15	15	15
Outliers	0	0	0	0	0	0	0	0	0
Average value (g/L)	0,91	0,61	0,74	0,63	0,40	0,63	3,88	4,82	6,39
Mean repeatability standard deviation (g/L)	0,10	0,05	0,08	0,10	0,04	0,06	0,69	1,19	0,98
Mean repeatability variation coefficient (%)	8,54	6,68	8,12	10,39	8,60	8,43	16,60	15,41	12,48
Mean reproducibility standard deviation (g/L)	0,12	0,07	0,11	0,21	0,12	0,27	1,17	2,53	1,56
Mean reproducibility variation coefficient (%)	10,52	9,13	12,69	30,95	28,44	41,68	27,46	49,55	21,91

Annex B
(informative)

Examples for mixing devices for chemical conditioning procedure

Dimensions in millimetres

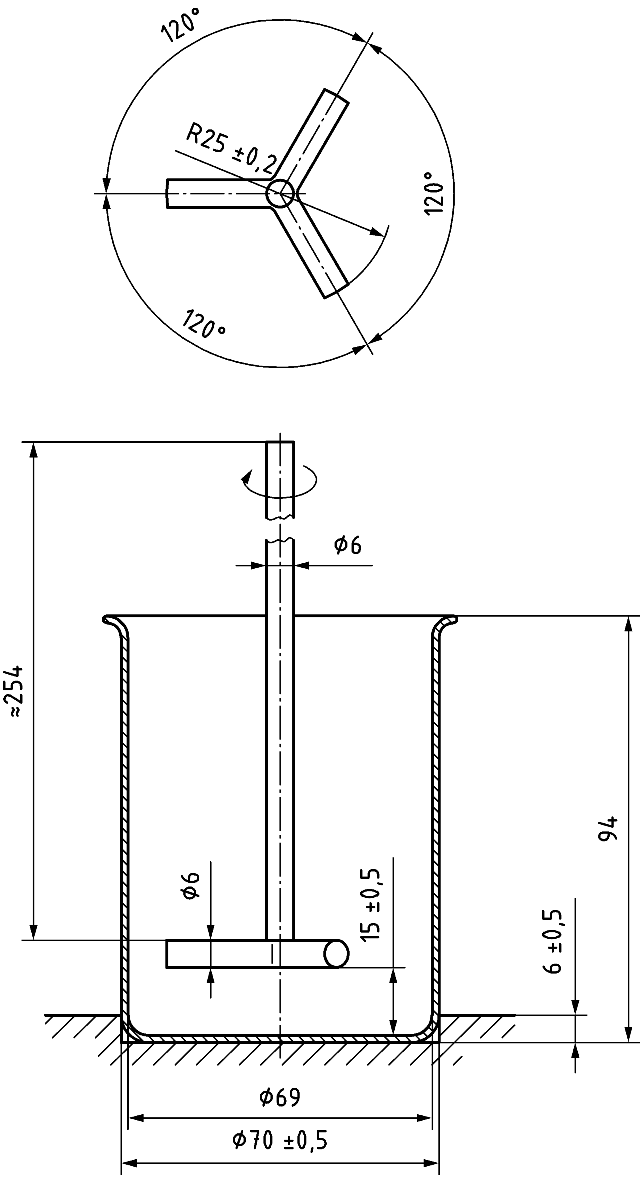
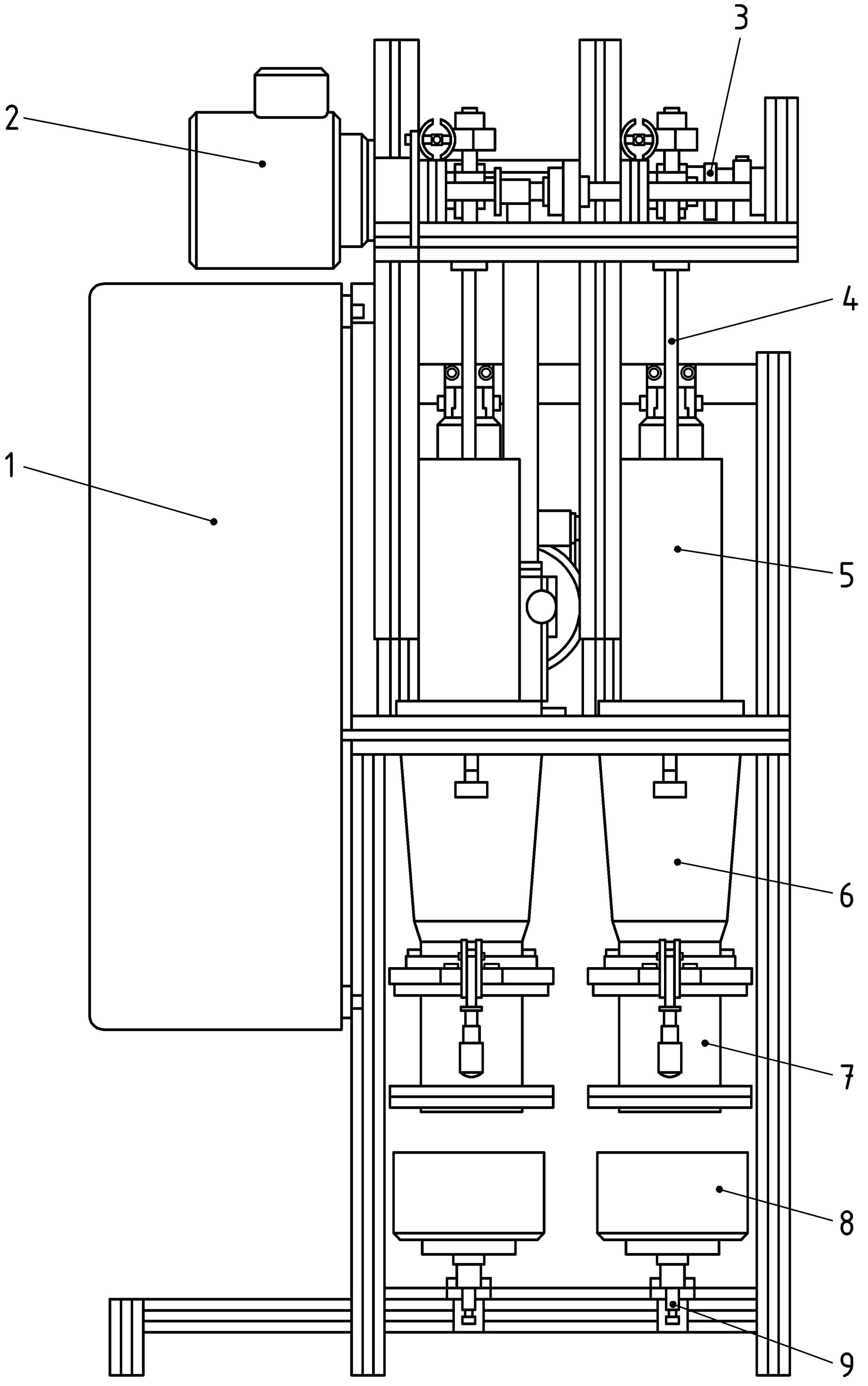


Figure B.1 — Device developed by EEC Concerted Action COST 68



Key

1 control box
2 motor
3 switching gearing
4 rotor
5 flocculation cell
6 drainage cell
7 thickened sludge box
8 filtrate beaker
9 weight sensor

Figure B.2 — Bootest developed by IFTS (Model N°1204)

Annex C
(informative)

Example of table summarizing the operating conditions and the results of the characterization tests

Table C.1 — Example of test results

Sludge sample				
Type of sludge				
Pretreatment				
Dry matter				
Loss of ignition (%)				
pH				
Sludge weight (g)				
Conditioning				
Reference of Product				
Concentration (g/l)				
Dosage (kg/t DS)				
Conditioner injection				
TB1 (s)				
TE1 (s)				
Method				
Mixing device				
Impeller type				
Impeller diameter (mm)				
Impeller position (mm for the bottom)				

Mixing conditions				
	Polymer dispersion			
Mixing speed (rpm)				
TBB1 (s)				
TEE1 (s)				
	Floc growth			
Mixing speed (rpm)				
TBB2 (s)				
TEE2 (s)				
Comments (floc quality)				
Drainage test				
Loaded mass (kg DS/m^2)				
M/M_0 at 10 s				
M/M_0 at 30s				
M/M_0 at 90 s				
50 % filtrate recovery time (s)				
Sludge thickened dryness (%)				
Suspended matters in filtrate (mg/l)				

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