PD CEN/TR 15463:2007

# Characterization of sludges — Physical consistency — Thixotropic behaviour and piling behaviour

ICS 13.030.20



British Standards

# National foreword

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TECHNICAL REPORT

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**CEN/TR 15463**

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English Version

# Characterization of sludges - Physical consistency - Thixotropic behaviour and piling behaviour

Caractérisation des boues - Consistance physique - Comportement thixotrope et comportement au tassement

Charakterisierung von Schlämmen - Physikalische Beschaffenheit - Thixotropes und Schüttverhalten

This Technical Report was approved by CEN on 4 August 2006. It has been drawn up by the Technical Committee CEN/TC 308.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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## Foreword

This document (CEN/TR 15463:2007) has been prepared by Technical Committee CEN/TC 308 “Characterization of sludges”, the secretariat of which is held by AFNOR.

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 CEN-Report “Physical Consistency” derives from the Desk Studies “Physical Properties – Flowability” (HORIZONTAL Report No. 21 [60]) and “Physical Properties – Solidity, Thixotropic Behaviour and Piling Behaviour” (HORIZONTAL Report No. 22 [61]) of the *Horizontal* Project. The “*Horizontal*” project has the objective to develop horizontal and harmonised European Standards in the fields of sludge, bio-waste and soil to facilitate regulation of these major streams in the multiple decisions related to different uses and disposal governed by EU Directives. The Horizontal Project includes the Work Package 7 “*Mechanical properties*” consisting in the development of Desk Studies on physical consistency, because it is recognized that this property is very important for the characterization of sludge, since it affects almost all treatment, utilization and disposal operations, such as storage, pumping, transportation, handling, land-spreading, dewatering, drying, landfilling. The importance of the physical consistency is also true for the characterization of bio-waste and soil. Also handling and utilization of many other materials, such as cement and asphalt are strictly depending on their physical consistency. The needs for control of operations and also material characteristics are described.

The first action carried out is consisted in searching for existing standards to be possibly used or adapted for utilisation in the specific field of consistency evaluation. The complete list of standards is reported in Annex 1 of the HORIZONTAL Reports No. 21 [60] and No. 22 [61], from which it can be seen that more than 250 standards and non-standardised methods are potentially applicable to consistency evaluation. On the basis of the selected list of standards and non-standardised methods for further consideration the methods for the determination of flowability, solidity, thixotropic behaviour and piling behaviour of sludge, bio-waste and soil have been divided into several groups, according to the instruments used for measuring:

— Flowability: Capillary viscometers, Penetrometers, Rotational viscometers and Flow apparatus.

— Solidity: Shearing apparatus, Vane testing apparatus and Penetrometers.

— Thixotropic behaviour: It should be investigated a combination of methods for determination of the solidity like penetration, etc. and an energy-input in terms of "flow" apparatus to simulate the shear stress.

— Piling behaviour: Slump test apparatus, Compacting apparatus, Cubic Piling Box (CPB) and "Turned Box".

For each group was evaluated the laboratory or field test feasibility. Apparatuses of the measuring procedures and existing applications to different materials were described. On this basis the applicability of the described methods to the materials of investigation was evaluated and documented in the lists of analysed standards.

The recommended methods are for flowability the coaxial cylinder viscometer as laboratory apparatus, while field apparatus are flow cone, magnesium penetration cone and extrusion tube viscometer. The recommended methods are for solidity the “Laboratory vane shear apparatus” and “Vicat needle” as laboratory reference and the pocket penetrometers for field test. The penetrometers in general could be used for both laboratory reference method and field test. Also for determination of the thixotropic behaviour the penetrometer is together with an energy-input in terms of a vibrating table or a hammer a suitable instrument. For measuring the piling behaviour the Cubic Piling Box (CPB) and the Oedometer are the recommended methods, whereby the CPB could be used in both laboratory and field while the Oedometer could be used only in the laboratory. All methods should be tested and optimized to adapt design and part dimensions to the materials in a future experimental activity.

For the research needs first the basics of methods are explicated and the applicability of methods to the materials is clarified. The questions to be answered (precision, repeatability, reliability, etc.), the route, how to answer them and finally the steps to be taken are important for following procedures.

In the Horizontal Report No. 21 a total of 6 proposals for draft standards are given, whereby one laboratory method and five field tests exist. In the Horizontal-Report No. 22 a total of 11 proposals for draft standards are given, consisting of six laboratory methods and five field tests.

# 1 Introduction

## 1.1 The Horizontal project and the Work Package 7

The revision of the Sewage Sludge Directive 86/278/EEC, the upcoming Composting Directive on the biological treatment of biodegradable waste and the Soil Monitoring Directive call for standards on sampling, hygienic and biological parameters, methods for inorganic and organic contaminants, and for mechanical properties of these materials.

In addition, when materials cannot be utilized, landfilling becomes important, in which case leaching becomes an issue as stipulated by the Council Directive 1999/31/EC on the landfill of waste. More recently, a Council Decision establishing criteria and procedures for the acceptance of waste at landfills, pursuant to Article 16 and Annex II of mentioned Directive on the landfill of waste was issued (16/12/02) with physical consistency being one basic parameter of interest.

The "*Horizontal*" project has the objective to develop horizontal and harmonised European Standards in the fields of sludge, bio-waste and soil to facilitate regulation of these major streams in the multiple decisions related to different uses and disposal governed by EU Directives.

Part of the work to be carried out will focus on *co-normative* work with an emphasis on horizontal standardization starting from existing standards developed for the same parameter in the fields of sludge, bio-waste and soil. Another part of the work will focus on *pre-normative* research required to develop standards lacking at this point and needed in the next revision of the regulations in these fields.

The work within the HORIZONTAL Project was coordinated in the Work Package 7 "*Mechanical properties*" and done in cooperation of the involved teams. It consists in the development of the Desk Studies on physical consistency mentioned above, because it is recognized that this property is very important for the characterization of sludge e.g., since it affects almost all treatment, utilization and disposal operations, such as storage, pumping, transportation, handling, land-spreading, dewatering, drying, landfilling. In fact, the selection of the most suitable equipment and procedure for land application, storage and transportation of sludge e.g. is strongly connected to its consistency. Similarly, compacting sludge in a landfill or forming a pile in composting is depending on sludge shear strength rather than on its solids concentration. In particular, with reference to the regulations requirements, according to the Sludge Directive 278/86, agricultural reused sludge should have agronomic interest, be healthy and easily usable, i.e. easily stored, transported, handled, and spread.

In Council Directive 1999/31/EC (Landfill Directive), Article 2 (q) gives a definition of "liquid waste", and Article 5 (3.a) does not allow a liquid waste to be landfilled, but a standardized method for this evaluation has to be developed yet. Further, Annex II (2. General principles) requires that "The composition, … and general properties of a waste to be landfilled must be known as precisely as possible", and Annex I (6. Stability) is referring to "... ensure stability of the mass of waste ... particularly in respect of avoidance of slippage", so the shear strength and piling behaviour should be known. Article 2 (h) says, that "treatment means ... processes ... in order to … facilitate its handling". Finally, Article 11 (1.b) asks for: " – visual inspection of the waste at the entrance and at the point of deposit and, as appropriate, verification of conformity with the description provided in the document submitted by the holder", so simple and easy tests to be carried out on the field and followed by the operators should be defined. Further, the Council Directive establishing criteria and procedures for the acceptance of waste at landfills, pursuant to Article 16 and Annex II of mentioned Directive on waste landfilling included "consistency" among the basic parameters to be evaluated for waste characterization before landfilling; for specific cases it is also demanded, that EU Member States must set criteria to ensure a sufficient physical stability and bearing capacity of waste. It is also to be pointed out that in many analytical methods for sludge characterization (e.g. pH, dry matter, leachability, etc.) different procedures are indicated depending on whether the sample to be examined is liquid or not, is solid or not, but no procedures are given for evaluating such properties. The importance of the physical consistency is also true for the characterization of bio-waste and soil.

## 1.2 Desk study subject

The Task Group 3 (TG3) of CEN/TC308/WG1 defined 3 physical states for sludge (CEN/TC308/WG1/TG3, 2000):

a) ***Liquid***: sludge flowing under the effect of gravity or pressure below a certain threshold.

b) ***Paste-like***: sludge capable of continuous flow under the effect of pressure above a certain threshold and having a shear resistance below a certain threshold.

c) ***Solid***: sludge having a shear resistance above a certain threshold.

This firstly involves the necessity to set up methods to measure values in the range of the boundary area between liquid and paste-like behaviours (limit of *flowability*) and that between solid and paste-like (limit of *solidity*). Further, the *thixotropic* behaviour of solid materials (from “the solid to the liquid state and vice versa”) should be evaluated, together with the *piling* behaviour referred both to “compaction and physical stability”. Also the CEN/TC292/WG2, in the method EN 12457 for the characterisation of waste included in Annex B (Informative) the description of a test for determining whether waste is in the liquid state (CEN/TC292/WG2, 2002).

Although the methods to be developed are partly known and used in other technology fields, e.g. soil mechanics, materials for construction works (concrete, suspensions), etc., widely accepted methodologies for the evaluation of above properties, able to give comparable and reliable results, are not available yet. It therefore follows the necessity to define *simple* and *reliable* measurement procedures to be applied in the *field*, together with those to be used as reference in *laboratory*. Standardisation procedures for the material examination will consist of

- Sampling, transport, preservation, storage
- Pre-treatment
- Measurement and evaluation of results.

In the report “Globally Harmonized system of Classification and Labelling of Chemicals (GHS)” other definitions of liquid and solid are given [59]:

***Liquid*** means a substance or mixture which at 50 °C has a vapour pressure of not more than 300 kPa (3 bar), which is not completely gaseous at 20 °C and at a standard pressure of 101.3 kPa, and which has a melting point or initial melting point of 20 °C or less at a standard pressure of 101.3 kPa. A viscous substance or mixture for which a specific melting point cannot be determined shall be subjected to the ASTM D 4359-90 test [56]; or to the test for determining fluidity (penetrometer test) prescribed in section 2.3.4 of Annex A of the European Agreement concerning the International Carriage of Dangerous Goods by Road (ADR) [55];

***Solid*** means a substance or mixture which does not meet the definitions of liquid or gas.

## 1.3 Evaluation of needs for control of operations and material characteristics

### 1.3.1 Evaluation of needs for control of operation

The purpose of using characterisation standards is to control and ascertain the material amenability to handling and different operations. Materials considered are

- Sewage sludge
- Waterworks sludge
- Bio-waste and

— Soil

Materials, which cannot be utilised, are subjected as waste to the Landfill Directive (Council Directive 1999/31/EC), respectively the ordinances of the member states. The member states had to translate this directive into national law. In Germany e.g., there is the landfill ordinance [58], which became operative on 24.07.2002. Furthermore there does exist the waste disposal ordinance, it is a kind of adjustment respectively update of the German “TA Siedlungsabfall”. By this regulation among other things the limit value of ≥ 25 kN/m² for vane shear strength – termed also in the HORIZONTAL Report No.22 [61] - was set. For this regulation it is not important, from where the materials come from. It is valid for different respectively all kinds of wastes.

For handling and operating these materials many parameters should have to be known; they include homogeneity, particles sizes and shape, solids (total, suspended, volatile) that, if available, could define the range of variation of variable considerations (i.e. viscosity, etc.).

The parameters flowability is an overall parameter taking into account all above mentioned material properties or characteristics. In particular, the flowability evaluation for sludges, including wastewater, waterworks and similar sludges, is of fundamental importance in many operations such as pumping, transportation, storage, dewatering, stabilisation, spreading, etc. also considering the possible formation from a gel to a liquid (sol) and vice versa. Similarly, for bio-waste, including the shredded organic fraction of municipal solid waste (OFMSW), in operations such as handling, digestion, reuse, etc. the measure of the parameter flowability have to be considered. Finally, for fine-grained soils, the water content (and therefore consistency and flowability) has always been considered an important indication of their mechanical properties. Moreover in case of soil slurries it is very important to verify flowability as a measurement of their workability and time of setting.

The solidity is also a parameter, which concerns all the material properties or characteristics mentioned above. The determination of this parameter is getting more important for handling of solid materials like dewatered sludge, other bio-waste – e.g. in terms of pieces (compost) – and soil, where the grain size distribution and water content have to be considered, during operations like pumping, transportation, storage, etc.

The measurement of thixotropic behaviour for solid materials is relevant especially for dewatered sludge like sewage, waterworks and related sludge. By dewatering and storage the sludge becomes solid. During operations such as transportation the sludge gets in a liquid state due to the vibration of a truck.

The piling behaviour evaluation is also for dewatered sludge, particular bio-waste and soil of importance. The determination of the piling angle is a useful instrument to characterise the storage properties and calculate the space, which is needed for e.g. storage and transportation. Together with the thixotropic behaviour the piling behaviour refers to the compaction and stability.

However, the development of reliable measurement procedures of all parameters is not a simple matter, because measurements are influenced by below described properties or characteristics. This means that those factors must be considered with great attention and methods defined by avoiding any negative interference with them during measuring procedures. For this reason, it is first essential to select, if any, the most adapted standards or non-standardised methods applicable to sludge, bio-waste and soil or to develop a new one, and then to carry out parallel tests to evaluate how they are affected by the other specific characteristics. In addition, these aspects require to be investigated for both laboratory methods, to be adopted as a reference, and simple tests to be applied in the field.

### 1.3.2 Material characteristics

#### 1.3.2.1 Sewage sludge

Sewage sludge can be produced from several processes (primary sedimentation, activated sludge process, aerobic or anaerobic digestion etc.). Their solid content cover a wide range from 1 % to 30%, while different total volatile solids percentage on dry matter can vary from 75% to 45%. The presence of coarse particles is strongly related to the sieve adopted in head-works or external material used in some processes (anaerobic co-digestion, etc.). Sewage sludge covers a wide range of physical state from liquid to solid. Bibliography does not offer a characterization of particle size distribution of sewage sludge, a wide range of these characteristics is forecasting in relation to the process adopted (opening of sieves etc.) and different type of sewage sludge treated. Some indications are found for sewage sludge (see Table 1 [1]).

**Table 1 — Particle size distribution of sewage sludges**

| Material | Process | TS basis<br>% cumulative retained w.w. | | | TVS basis<br>% cumulative retained w.w. | | |
|---|---|---|---|---|---|---|---|
| | | 5 mm | 2 mm | 0,84 mm | 5 mm | 2 mm | 0,84 mm |
| Sewage sludges WTS | Aerobic process | 0 | 3,7 | 9,4 | 0 | 4,7 | 8,4 |
| Mixed primary sludges ADS | Mesophilic anaerobic digestion | 0 | 10,5 | 18,5 | 0 | 15,5 | 30,5 |

Each kind of sludge was analysed for its particle size distribution by wet sieving, using three sieves with openings of 5,2 mm and 0,82 mm. According to these data the samples can be divided into four conventional classes: coarse (>5 mm), medium (from 5 mm to 2 mm), medium-fine (from 2 mm to 0,84 mm) and fine (<0.84 mm). It can be noted that the sewage sludges have no coarse particles but a different percentage of medium and fine particles.

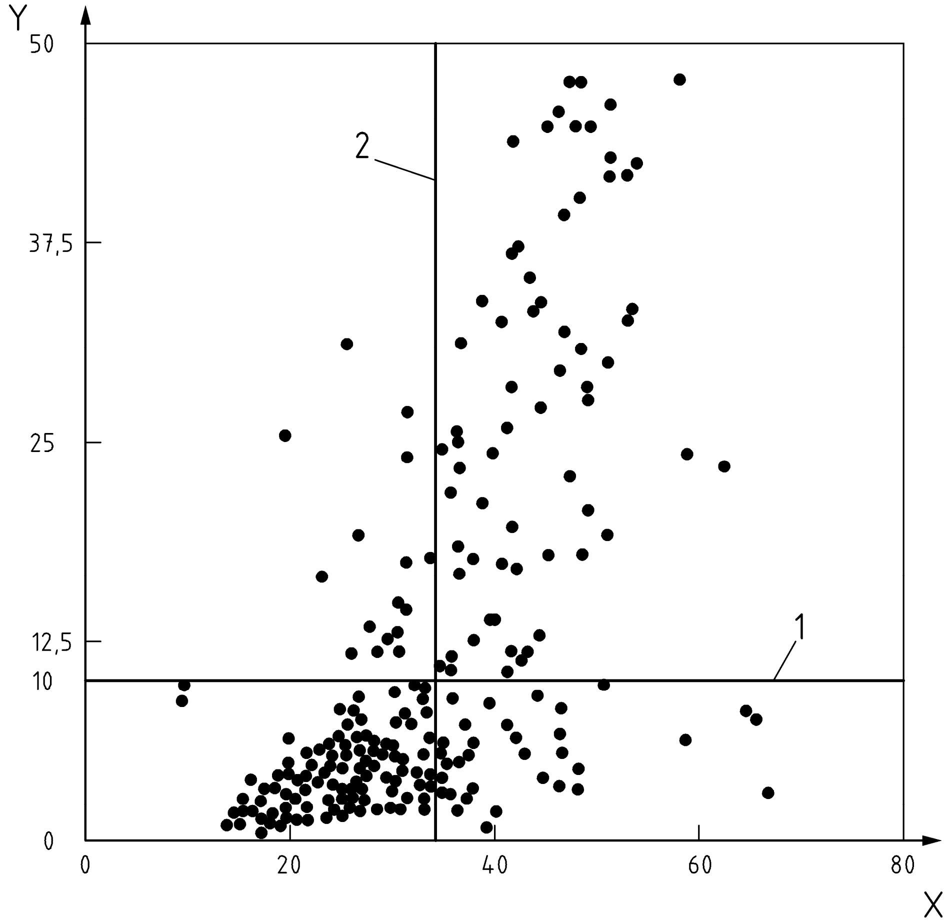
The most diffuse sludge characterization is that rheological, beside CST – capillary suction time, R specific resistance to filtration etc.. Rheological parameters (yield stress, viscosity and thixotropy) were originally applied to calculation of the head losses in sludge pumping operations, recently it has been shown that they can affect filtration, thickening [2], pumping [3, 4] and constitute useful on line control parameters for sludge conditioning and dewatering [5, 6, 7].

Rheological measurements of sewage sludges have been performed using commercial rotational viscometer. The rheological properties normally determined by using the Bingham plastic model are the yield stress (YS) (that is the stress required to start the material flowing) the plastic viscosity (that is the internal resistance to flow under defined shear rate). The Thixotropy, determined by the hysteresis area, is only sometimes observed (Table 2) [8].

**Table 2 — Rheological properties of sewage sludges**

| Sludge | Process | TS range<br>% | TVS/TS | YS<br>Pa | Plastic viscosity<br>mPa · s |
|---|---|---|---|---|---|
| Mixed | | 3,0-14 | 0,52 | 1,90-185 | 30-630 |
| Primary | | 7,0-16 | 0,43 | 1,0-49 | 20-320 |
| Activated | | 3,0-9 | 0,73 | 5,0-214 | 70-1110 |
| Mixed Sludges | Aerobically stabilized | 4,0-10 | 0,53 | 0,07-58 | 10-410 |
| Mixed Sludges | Anaerobically digested | 4,0-9 | 0,59 | 1,0-112 | 20-390 |
| Mixed sludges | Mesophilic anaerobic digestion | 3,5-5,0 | 0,5-0,6 | 0,4-1,6 | 8,0-24 |
| Mixed sludges | Thermophilic anaerobic digestion | 3,5-5,0 | 0,5-0,6 | 0,1-0,5 | 11,0-17 |

Mechanical properties of sewage sludge in solid state were studied with the aim to define the feasibility of landfill disposal; a correlation between shear strength and dry solid matter for sewage sludge was done using a Vane apparatus (Figure 1) [9], sludges are suitable for landfilling if their shear strength is of 10 kN/m² at least (limit value in Italy).




Number of values taken into consideration: 292

**Key**

| | | | |
|---|---|---|---|
| X | dry solid matter (%) | 1 | minimum strength 10 kN/m³ |
| Y | shearing strength (kN/m³) | 2 | 35 % |

**Figure 1 — Collation between shearing strength and dry solid method [9]**

### 1.3.2.2 Waterworks sludge

In DVGW W221-1 [10] sludges are defined as solid-water suspensions capable of flowing after sedimentation, flotation or thickening. Dewatered sludges are sludges, which were dewatered by natural or mechanical treatments until they are no longer able to flow.

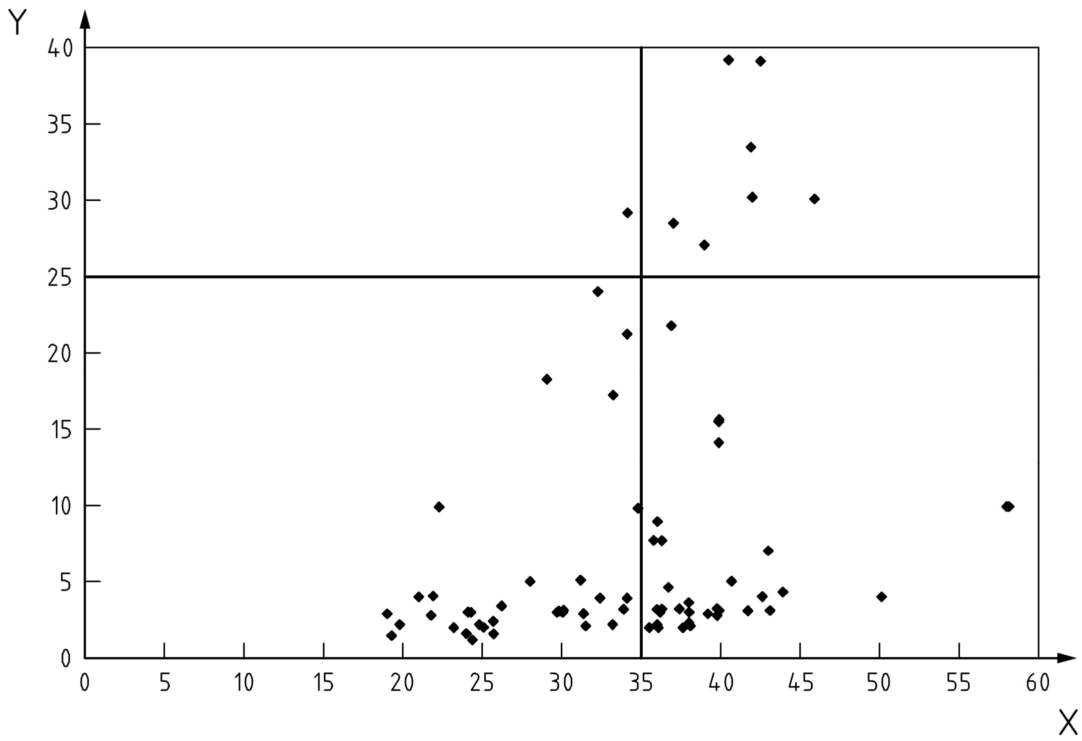
Sludges from water treatment contain several phases differing by their physical state and/or chemical nature. The space distribution of these phases, as well as the physical-chemical interactions between them, gives to sludges their cohesion. A too low cohesion of sludges and/or its high fluctuation in the time commonly generates handling (shovelling- and spreading- ability) and storing difficulties.

An orderly utilisation and disposal of waterworks sludges need the control of the mechanical properties in order to ensure a quality that is demanded for storage, transport and handling. The mechanical measurements should be seen and done in connection with other measurements that have been or will be standardised. There should be mentioned methods for chemical and physical parameters (chemical elements, dry solids, loss on ignition, pH-value) and operational methods (capillary suction time CST, specific filtration resistance). The different composition of waterworks sludges with inorganic (Fe, Ca, Al etc.) and organic (Algae, humid substances, powdered activated carbon etc.) substances depending on the source of raw water and water treatment processes should be considered. An inquiry from *Wichmann et al (2002)* [17] showed, that the waterworks sludges of different types in Germany amounted in 1998 to ca. 181.000 t DS (Lime sludge 40%, Iron sludge 14%, Fe-/Al-Flocculation sludge 13% and other 33%). The composition of the sludge can be determined after [11] (10 parameters) in Table 3.

**Table 3 — Determination of the composition of sludge**

| Parameter | Fraction [g/kg DS] (Fictive value) | Conversion in: | Fraction [g/kg DS] |
|---|---|---|---|
| Acid insoluble (= $HCl_{ins.}$) | 40 | Insoluble components (e.g. sand, activated carbon) | 40 |
| TOC | 30 | Total organ. content (Factor: 2) | 60 |
| Mn | 20 | $MnO_2$ (Factor: 1.58) | 31,6 |
| Mg | 5 | $Mn(OH)_2$ (Factor: 2.,4) | 12,0 |
| Al | 20 | $Al_2O_3$ x $H_2O$ (Factor: 2.22) | 44,4 |
| $SO_4$ | 5 | $CaSO_4$ (Factor: 1.42) | 7,1 (Ca: 2,1) |
| $CO_3$ (= TIC) | 80 | $CaSO_3$ (Factor: 1.67) | 138,6 (Ca: 57,6) |
| Ca –total- | 65 | | - |
| Residual-Ca | 5.3 | $Ca_3(PO_4)_2$ (Factor: 2.58) | 13,7 ($PO_4$: 8,4) |
| $PO_4$ –total- | 10 | | - |
| Residual-$PO_4$ | 1,6 | $Fe(PO_4)$ (Factor: 1.59) | 2,5 (Fe: 0,94) |
| Fe –total- | 415,7 | | - |
| Residual-Fe | 414.8 | $Fe_2O_3$ x 1,5 $H_2O$ (Factor: 1.67) | 692,6 |
| Total | | | 1042,5 |

The range of 0,2 % to 80 % dry solids contents of waterworks sludge to be utilized is quiet wide, so that several different mechanical properties have to be measured. Possible measuring methods are coming mainly from the soil mechanical or rheological working fields. There are only few data on mechanical properties of waterworks sludge published. In Figure 2 after *Mc Tigue et al. (1990)* [12] e.g. the result of 72 measurement data of different waterworks sludge types and dewatering processes are shown. A laboratory vane shear apparatus was used. The vertical line marks the dry solids concentration of 35 % that was given from *LAGA (1979)* [13] as a minimum value for disposal of wastes in landfills. Sludge with more than 35 % DS were than be considered to be qualified for landfilling. The horizontal line marks the minimum value of 25 $kN/m^2$ concerning the vane shear strength that is now demanded in new regulations in the *TA Siedlungsabfall (1993)* [14]. Approximately 90 % of the waterworks sludges tested after mechanical dewatering could not fulfil the required minimum value.




**Key**

X dry solid [% w/w]
Y vane shear strength [kN/m²]

**Figure 2 — Comparison dry solid contents vs. laboratory vane shear strength [12]**

**1.3.2.3** Bio-waste

Organic fraction of municipal solid waste (OFMSW) is utilized for anaerobic and composting treatment. Anaerobic digestion or co-digestion with sewage sludges is a well-known process where rheological parameters have been studied to control process.

**Table 4 — Particle size distribution of OFMSW**

| Material | Process | TS basis % cumulative retained w.w | | | TSV basis % cumulative retained w.w | | |
|---|---|---|---|---|---|---|---|
| | | 5 mm | 2 mm | 0,84 mm | 5 mm | 2 mm | 0,84 mm |
| Fresh mechanically sorted (F) OFMSW | Mesophilic anaerobic digestion | 8,2 | 11,1 | 18,3 | 6,7 | 21 | 23,5 |
| Pre-composted mechanically sorted (P) OFMSW | Mesophilic anaerobic digestion | | | 19,7 | | | 26,1 |
| Blend of source sorted and mechanically sorted OFMSW | Mesophilic anaerobic digestion | 6,6 | 11,5 | 18,3 | 12,7 | 16,8 | 24,2 |

The OFMSW was analysed for its particle size distribution by wet sieving, using three sieves with openings of 5, 2 mm and 0,82 mm. According to these data the samples can be divided into four conventional classes: coarse (>5 mm), medium (from 5 mm to 2 mm), medium-fine (from 2 mm to 0,84 mm) and fine (<0,84 mm).

The fresh mechanical sorted OFMSW show up to 18 % and 24% of particles greater than fines for TS and TVS, respectively. The coarse particles are mainly extraneous materials (plastics, baggage, etc.), which have no influence on digester fluid-dynamic [21, 22].

Rheological measurements of OFMSW aerobically and anaerobically treated have been performed using commercial rotational viscometer. The rheological properties normally determined by using the Bingham plastic model are the yield stress (YS) (that is the stress required to start the material flowing) the plastic viscosity (that is the internal resistance to flow under defined shear rate). The Thixotropy, determined by the hysteresis area, is only sometimes observed (Table 5) [21, 22].

**Table 5 — Rheological properties of OFMSW aerobically or anaerobically treated**

| Sludge | Process | TS range<br>% | TVS/TS | YS<br>Pa | Plastic viscosity<br>mPa · s | Thixo-tropy<br>Pa/s |
|---|---|---|---|---|---|---|
| OFMSW mechanically sorted and pre-composted | Thermophilic anaerobic digestion | 4,8-32,7 | 0,43-0,49 | 0,4-63 | 9-840 | |
| Fresh OFMSW mechanically sorted | Thermophilic anaerobic digestion | 6,8-25,2 | 0,47-0,50 | 0,1-102 | 17-1660 | |
| OFMSW mechanically sorted enriched with source sorted fraction | Thermophilic anaerobic digestion | 5,8-35,1 | 0,46-0,56 | 0,2-61 | 6-560 | |
| OFMSW | Anaerobically digested under mesophilic and semi-dry conditions | 4,4-18 | 0,52 | 0,25-1,2 | 8,0-54 | 12-125 |
| OFMSW | Anaerobically digested under thermophilic and semi-dry conditions | 5,0-32,7 | 0,48 | 0,26-37,7 | 10-420 | 36-161 |

Bio-waste includes also compost, which is derived from aerobic decomposition of recycled plant waste, bio-solids, fish or other organic material. It is a mixture of decaying organic matter, as from leaves and manure, used to improve soil structure and provide nutrients.

The sizes of compost pieces can vary within a wide range according to the type of compost e.g. organic municipal waste. From there the sampling and especially the pre-treatment are of great importance. For reliable measurements the grain size should be uniform. If needed, the material has to be also compacted in order to avoid voids of higher dimensions, which could influence the results.

#### 1.3.2.4 Soil

Soils are particulate systems and can cover a wide range of physical state from liquid to solid, depending mainly on the size and mineralogy of the particles and on the water content. In particular, soils are divided into coarse-grained soils and fine-grained soils. The surface of the particles has a negative electrical charge, whose intensity depends on the soil mineral. These surface forces exist in addition to the volume forces of the particles and in fine-grained soils they play a dominant role in the mechanical and rheological behaviour. The surface forces strongly depend on the water content and more in general on the chemical composition of the interstitial fluid.

For this reason, it is very important to set up standards that can define different physical states considering not only the water content but also the chemical composition of the pore fluid.

The classification of a granular soil is completely defined by the grain distribution curve, the shape of particle and its specific volume. Whereas the procedure for particle size analysis is well defined and easy to perform (by a simple sieve analysis), a procedure for characterising the particle shape is not available and it should be defined as the particle shape strongly influences the mechanical behaviour of these type of soils.

The behaviour of cohesive soils depends on its mineral composition, the water content, the degree of saturation and its structure. A fine-grained soil can be in a liquid, plastic, semi-solid or solid state, depending on its water content and this physical state is called consistency. The upper and lower limits of water content within which a clay element exhibits plastic behaviour are defined as liquid limit and plastic limit. The procedure is standardised *(ASTM D4318)* but it is recognised to be strongly affected by the operator experience. Typical values of liquid limit for natural fine-grained soils range from 40% up to 90% of water content and the plastic limit from 10% to 50%. Many correlations have been proposed relating the mechanical characteristics of cohesive soils and the consistency limit; each of them is valid for the specific type of soil for which it was verified. Table 6 gives an overview of several grain distributions of different standards. The last one (British Standard) is also valid for Germany e.g. and other European countries.

**Table 6 — Grain size of soil according to different classification systems**

Grain size (mm)

1000 100 10 1 0,1 0,01 0,001

ASTM (D422; D653)

| Boulders | Cobbles | Gravel | Sand | | | Silt | Clay | Colloids |
|---|---|---|---|---|---|---|---|---|
| | | | Coarsa | Medium | Fine | | | |

300 75 4,75 (4) 2 (10) 0,425 (40) 0,075 (200) 0,005 0,001

AASHTO (T88)

| Boulders | Gravel | Sand | | Silt | Clay | Colloids |
|---|---|---|---|---|---|---|
| | | Coarsa | Fine | | | |

75 2 0,425 0,075 0,005 0,001

USCS

| Boulders | Cobbles | Gravel | | Sand | | | Fines (Silt, Clay) |
|---|---|---|---|---|---|---|---|
| | | Coarsa | Fine | Coarsa | Medium | Fine | |

300 75 19 4,75 2 0,425 0,075

Bridch Std. and M.I.T.

| Boulders | Cobbles | Gravel | | | Sand | | | Silt | | | Clay |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Coarsa | Medium | Fine | Coarsa | Medium | Fine | Coarsa | Medium | Fine | |

300 60 20 6 2 0,6 0,2 0,06 0,02 0,006 0,002

## 1.4 Search for existing standards and methods

The first action carried out consisted in searching for existing standards and non-standardised methods to be possibly used or adapted for utilisation in the specific field of *consistency* evaluation.

To this purpose, the following *standardisation organisations* were contacted:

- **ISO** at www.iso.ch
- **ASTM** at www.astm.org
- **CEN** at www.cen.eu
- **UNI** at www.uni.com
- **DIN** at www.beuth.de
- **AFNOR** at www.afnor.fr/portail.asp
- **BSI** at www.bsi.org.uk
- **ASAE** at http://webstore.ansi.org/ansidocstore/default.asp

Other information was obtained through personal contacts with experts in the field. In addition, to obtain selected information, the following keywords were used for research in each web site (among other things): *consistency, viscosity, flowability, shearing, sludge, soil, physical properties, flow properties, suspensions, and compactibility*. The different materials resulted from the research were resins, plastic, lubricant, cement, asphalts/bitumen, etc.

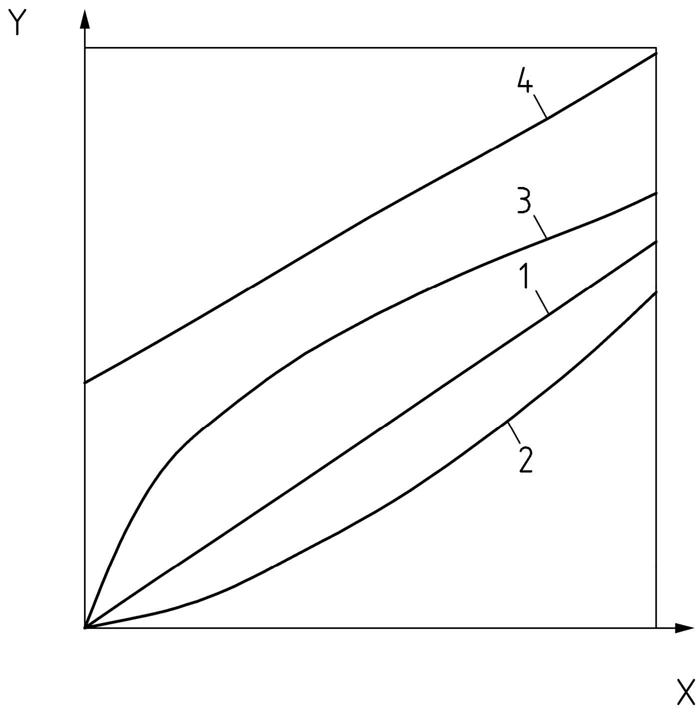
The complete list of standards is reported in Annex 1 of the HORIZONTAL Reports No. 21 [60] and No. 22 [61], from which it can be seen that more than 250 standards and non-standardised methods are potentially applicable to consistency evaluation.

## 1.5 Basic information

### 1.5.1 Flowability

Flowability and rheology

*Flowability* is the state in which a material is able to *"flo*w", i.e. it behaves as a liquid. This characteristics is, therefore, strictly connected to rheological properties, as rheology is the science of deformation studying the relationship between shear stress (internal stress) and shear rate (velocity of deformation) which can be depicted in a rheogram. The rheological behaviour of very thin sludge is Newtonian, like water, i.e. the viscosity is independent of the flow rate (shear rate) and no initial resistance is shown if a force is applied at rest (yield stress). This is shown in Figure 3, as T=μ D, where T is the shear stress, D the shear rate and μ the viscosity. Instead, the behaviour of more concentrated suspensions is described as non-Newtonian: they may exhibit a yield stress and the viscosity may vary with the shear rate. As shown in Figure 3, many models are applicable: a general equation is $T=T_0+\mu\ D^n$, where $\tau_0$ is the yield stress, μ the plastic viscosity or fluid consistency index, and n the fluid behaviour index. The Bingham plastic model, with n=1, so no curvature of the rheogram is exhibited, should seem to be preferable, as it allows to define a unique viscosity-type coefficient, plastic viscosity, measured by the slope of the line of shear stress vs. shear rate [21, 22].




**Key**

X shear rate
Y shear stress
1 Newtonian
2 Dilatant
3 Ostwald pseudoplastic
4 Bingham plastic

**Figure 3 — Rheogram types**

Rheology and sludge management

The use of rheological parameters for sludge flow, in particular for suitability to pumping, is well known, but the existence of some correlations between the rheological properties and other material characteristics have also been evidenced.

To this purpose, the usefulness of rheological measurements in sludge management in relation to treatments (biological, physical-chemical), utilisation/disposal (agricultural use, landfilling), and handling (pumping, storage, transportation) has been extensively discussed in CEN/TC308/WG2-N25 (1995). Table 7 shows the applicability of different treatment/disposal options on sludge having different physical state [22].

**Table 7 — Importance of the sludge physical states in its management (L=Low; M=Medium; H=High)**

| Operation | Liquid | Paste-like | Solid |
|---|---|---|---|
| Stabilization | H | M | L |
| Dewatering | H | M/L | L |
| Storage/Transportation | H | H | H |
| Agricultural use | H | H | H |
| Landfilling | L | M/H | H |
| Incineration | L | M/H | H |

Rheological measurements

A lot of viscometers are available. They fall into two general categories: 1) tube type (often referred to as capillary viscometer) and 2) rotating type.

The use of a tube viscometer is a good approach to define completely the sludge flow characteristics, but a quite complicate procedure is needed and only a poor control of operative parameters is possible. Moreover, the tube diameter should be large enough to prevent any clogging phenomenon, which means that the tube has to be quite long in order to obtain measurable head losses.

Rotational viscometers are generally considered to be more useful. They may have (i) coaxial cylinders with moving inner or outer cylinder, (ii) rotating blades and (iii) cone-plate geometry. The drawback of coaxial cylinder types is that cylinders have to be very close together when studying low concentrated and/or not very viscous sludge. Consequently, there is a risk of obstructions by grains of sand, fibres and other solid materials. Other drawbacks are the separation of particles due to the gravitational and centrifugal fields and the phenomenon of slip occurring at the cylinder/liquid interface, which can be overcome by artificially increasing the roughness of the moving cylinder. In the case of viscometers with blades, or vane apparatus, the velocity gradient is less well defined, so only a mean value based on the mechanical energy dissipated in the medium, calculated from measuring the drive torque of the mover, can be obtained. The cone-plate geometry rheometers can be excluded on the basis of both the large size of sludge particles relative to the gap and the poor liquid sludge consistency.

It should also be pointed out that different conclusion can be drawn from using different equipment and procedures, so standardised procedures are required to allow comparable and reliable results to be obtained. Moreover, the development of simple and reliable methods to be applied in the field is necessary.

### 1.5.2 Solidity

Solidity is the state in which a substance has no tendency to flow under moderate stress, resists forces (such as compression) that tend to deform it, and retains a definite size and shape. It describes the consistency of a solid respectively the limit solid/ paste-like.

For determination of solidity besides the resistance to pressure, the flexural resistance and tensile strength the “undrained shear strength $c_u$” (compare Eq. 2) is an important parameter. Shear may be defined as the tendency of one part of e.g. a soil mass to slide with respect to the other. This tendency occurs on all planes throughout the soil mass. The singular plane of interest, however, is the plane of potential failure, called the plane of rupture. Shear strength, as measured along this plane of interest, is the ability of the mass to resist the occurrence of a shear failure between the soil above and below the plane. Masses, e.g. soils have the ability to develop strength in shear. Different material groups develop this strength in different ways.

Generally the cohesion (c), as defined by the Geotechnics, has to be determined on basis of the evolution of the shear strength ($\tau$) as a function of the applied axial strength ($\sigma$) using the Coulomb rule:

$$\tau = c + \sigma \cdot \tan\varphi \quad (1)$$

where $\phi$ is the angle of shearing resistance or friction angle. For sludges it has been observed that $\phi$ is near zero (*Costet and Sanglerat, 1975* [24]). For this reason it is reasonable to equal $\tau$ and $c$ (see Eq. 2). Furthermore, several authors (*Gazbar, 1993* [25]; *Tisot et al., 1997* [26]) considered that the yield stress (the stress above which the sludge is deformed) is equal to.

$$\tau_f = c_u \quad (2)$$

where $c_u$ is the undrained cohesion or the undrained shear strength.

The ability of a mass to support vertical loads and to resist the sliding effect of lateral loads is governed to a large extent by the shear strength of the material. It is therefore important to determinate accurately the shear strength of mass situated beneath and in close proximity to the proposed construction. There are several field and laboratory tests by which shear strength can be determined with reasonable accuracy: Triaxial compression tests, vane shear tests or direct shear tests. Besides the determination of the "bearing capacity" the "unconfined compression strength", which is determined as twice the undrained shear strength (Eq. 3), and the "California bearing ratio" are important parameters.

$$q_u = 2 \cdot c_u \quad (3)$$

Penetration apparatuses are also being suitable for measuring the consistency respectively shearing strength, which is determined in this case with the aid of a constant depending on the dimensions of the cone tip (see 4.2.1, Eq. 7).

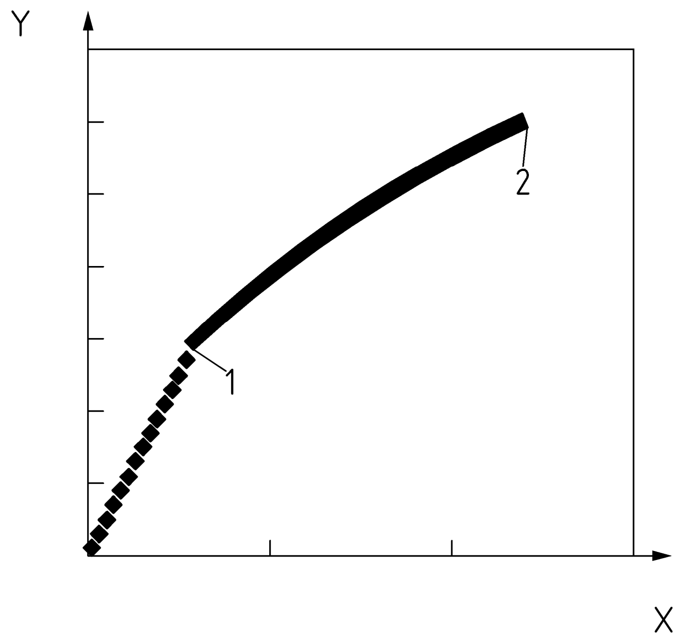
In general the tension or compression tests consist in measuring the strain in relationship to the applied stress. From a conventional stress-strain curve (Figure 4) it can be identified four different ways, in which the material behaves, depending on the induced strain [27]:

— Elastic behaviour: when the load acting on the material is removed, the sample returns to its original shape, (the stress is proportional to the strain),

— Yielding: a slight increase in stress above the elastic limit induces a breakdown of the material and cause it to deform permanently,

— Strain hardening: while the material is sheared, the deformation is permanent and there is no proportionality between the stress and the stress,

— Necking: a crack occurs in a localized region.




**Key**

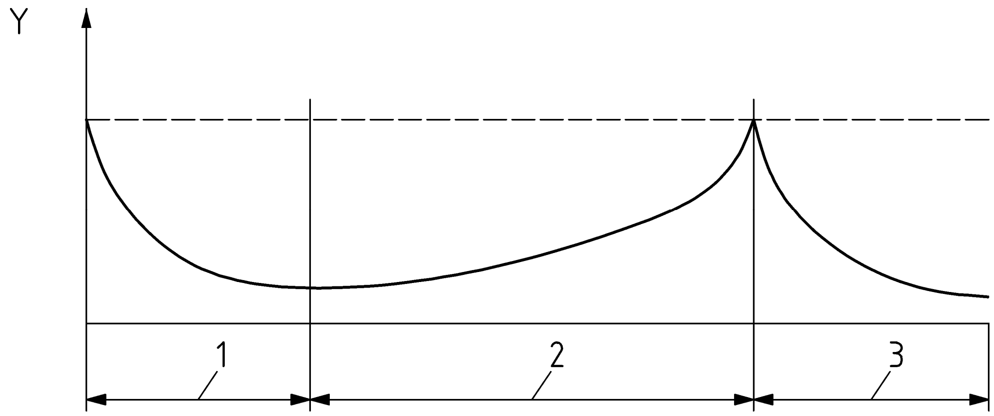
X stress [Pa]
Y strain
1 yielding point
2 failure

**Figure 4 — Schematic stress-strain curve of solid materials [27]**

### 1.5.3 Thixotropic behaviour of solid materials

Thixotropy or thixotropic behaviour is available, when the viscosity of a substance (shear rate $\dot{\gamma}$ = const.) decreases with the time, but after a defined period of rest the initial value of the viscosity is reached again

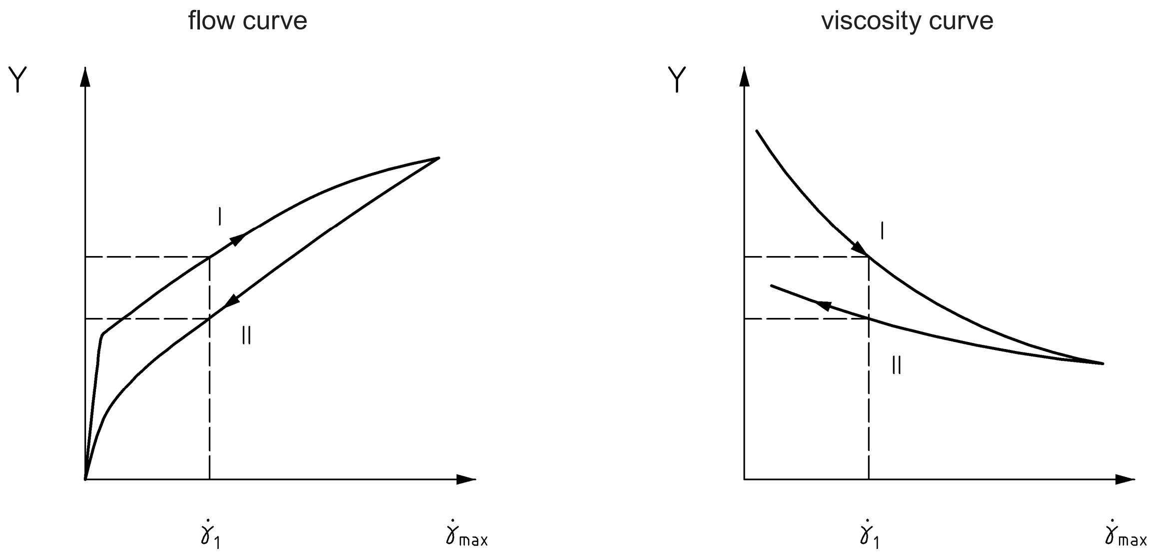
(Figure 5, [28]). The reasons for this behaviour are special formed chem./phys. bonding forces, which influence the relocatability against each other of those components and which are destroyed by the shear strain and during the non-operated time are complete reversible regenerated. Distinctive thixotropic properties are founded e.g. for solid materials with polar surface in covalent matters.




**Key**

Y viscosity
1 shear time
2 period of rest
3 shear time

**Figure 5 — Thixotropy in viscosity-time-graph ([28] modified)**




**Key**
Y shear stress

**Key**
Y viscosity

velocity gradient

**Figure 6 — Thixotropy in flow curve graph and viscosity curve ([29] modified)**

If the increasing curve in the flow curve graph (Figure 6, left, I) is located above the decreasing curve (II), this behaviour is called thixotropic. The received Hysteresis area is a degree of intensity of the constructiveness forces (e.g. structure of gel). In general this area is termed "A" and has the unit "power" related to the sheared volume. If the power is multiplied by the shear time (I+II) the "work" is resulted, which is necessary in order to destroy the thixotropic structure of the bonding forces in the substance volume [29]:

$$A = \tau \cdot \dot{\gamma} \qquad \left[ \mathrm{Pa} \cdot \frac{1}{\mathrm{s}} \right]$$

$$A = \frac{\mathrm{N}}{\mathrm{m}^2} \cdot \frac{1}{\mathrm{s}} = \frac{\mathrm{N} \cdot \mathrm{m}}{\mathrm{s}} \cdot \frac{1}{\mathrm{m}^3}$$

$$A = \frac{work}{sheartime} \cdot \frac{1}{volume} = \frac{power}{volume}$$

Beside the time-depended reduction of the viscosity (with a constant shear rate) there does exit a decrease of viscosity due to intensity of the shear rate (Figure 6, right).

If a material is anti-thixotropic, the position of the increasing curve in the flow curve graph is below the decreasing curve. This aspect is called "rheopexy" or also negative thixotropy. In practise it means the material is getting solid during the time of shear stress [28]. However, in this report mostly materials with a thixotropic behaviour are considered. In particular the value “shear strength”, which is mostly determined for the parameter “solidity”, is observed, whether it falls below the given threshold - because of shear stress - or not.

Waterwork and sewage sludges often show thixotropic behaviour. During shear stress the sludge change from the solid to the liquid state, this may hinder the utilization of the sludge during transport and handling. It should be ensured that dewatered sludge are not fluidized during transport and handling. Therefore it should be measured whether the material is behaving thixotropic and if so under which conditions.

### 1.5.4 Piling behaviour

The materials of interest usually have to be stored during transportation or storage in e.g. piles or containers. To avoid problems with succeeding handling operations the pieces of these materials should undergo no major changes in form and consistence. From there it is of great importance to investigate the piling behaviour, which is displayed by the materials.

The "compactibility" or "compressibility" is a first supporting parameter for determination of the piling behaviour. These parameters could give information about how much a stratum of piled material can settle due to the loading of the material placed above.

The measurement of the "piling angle (angle of rest)" or "slope angle" is also a very helpful parameter to determinate the piling behaviour [47]. It is a new method, which is still in a development stage, but due to the easy measurement procedure and variability (compare 3.3 ff.) a promising tool for the investigation of all materials of interest. The instrument for measuring these parameters consists of a simple box with turnable sidewalls. After filling the box - where necessary compacting the material – and the sidewalls were turned over, the natural slope angle of the remaining material can be measured on each side of the sample remaining on the box base. Because of the average of four values the result are mostly reliable. A further important aspect is that the friction between the sidewalls and the material to be tested does not affect the final result.

# 2 Existing standards or draft standards

The research of existing and draft standards to be utilized as laboratory and field methods for standardisation has been carried out in cooperation of the teams involved in WP7 by consulting the web sites of standardization boards (cp. section 1.4). Besides these standards some non-standardised methods, which are also used in practice, were acquired. The keywords used for the research have included the possible field and relevant properties for which the standards may be applicable. The expert's of WP7 has submitted titles to a preliminary examination and the selected standards (considered for further discussion) have been acquired. They can be divided generally into the four parameter groups flowability, solidity, thixotropic behaviour and piling behaviour.

## 2.1 Flowability

Keywords beside the item flowability:

- Bitumen
- Cement/concrete
- Consistency
- Plasticity
- Plastics
- Sludge (sewage)
- Soil
- Slurry
- Thixotropy
- Transport
- Viscosity
- Waste

In this group 129 standards and non-standardised methods have been found and 90 standards and non-standardised methods have been considered for further discussion.

## 2.2 Solidity

Keywords beside the item solidity:

- Cement
- Concrete
- Cone
- Consistency

— Mechanical properties

— Needle

— Penetrometer

— Plasticity

— Road materials

— Shearing strength

— Sludge (sewage)

— Soil

— Soil properties

— Vane

— Waste (solid)

In this group 68 standards and non-standardised methods have been found and 32 standards and non-standardised methods have been considered for further discussion.

## 2.3 Thixotropic behaviour

Keywords beside the item thixotropic behaviour:

— Cementitious & concrete materials

— Concrete

— Consistency

— Fluidity

— Penetration ball

— Road material

— Sludge (sewage)

In this group 15 standards have been found and 9 standards have been considered for further discussion.

## 2.4 Piling behaviour

Keywords beside the item piling behaviour:

— Cement

— Cementitious & concrete materials

— Concrete

- Consistency
- Flowability
- Oedometer
- Piling box
- Plasticity
- Soil
- Waste (solid)

In this group 13 standards and non-standardised methods have been found and 8 standards and non-standardised methods have been considered for further discussion.

The list of the collected titles is reported in Annex 1 of the HORIZONTAL Reports No. 21 [60] and No. 22 [61]. The standards and non-standardised methods considered for further discussion are presented in Clause 3.

# 3 Evaluation of drafting a Horizontal standard

On the basis of the selected list of standards and non-standardised methods for further consideration the methods for the determination of solidity, thixotropic behaviour and piling behaviour of sludge, bio-waste and soil have been divided into several groups, according to the instruments used for measuring:

## 3.1 Flowability

- capillary viscometers
- penetrometer
- rotational viscometers
- flow apparatus

### 3.1.1 Capillary viscometers

The use of capillary viscometers is suggested in a lot of standard methods for different materials; they are generally laboratory tests since they include complex apparatus that enables the field application of the test, furthermore they can be applied only to Newtonian fluid.

#### 3.1.1.1 Laboratory or field test feasibility

All capillary viscometer tests are laboratory tests; this condition derives by the fact that the tests analysed require the use of a thermostatic condition of material, for this reason capillary viscometers are unusable in field condition.

#### 3.1.1.2 Apparatus

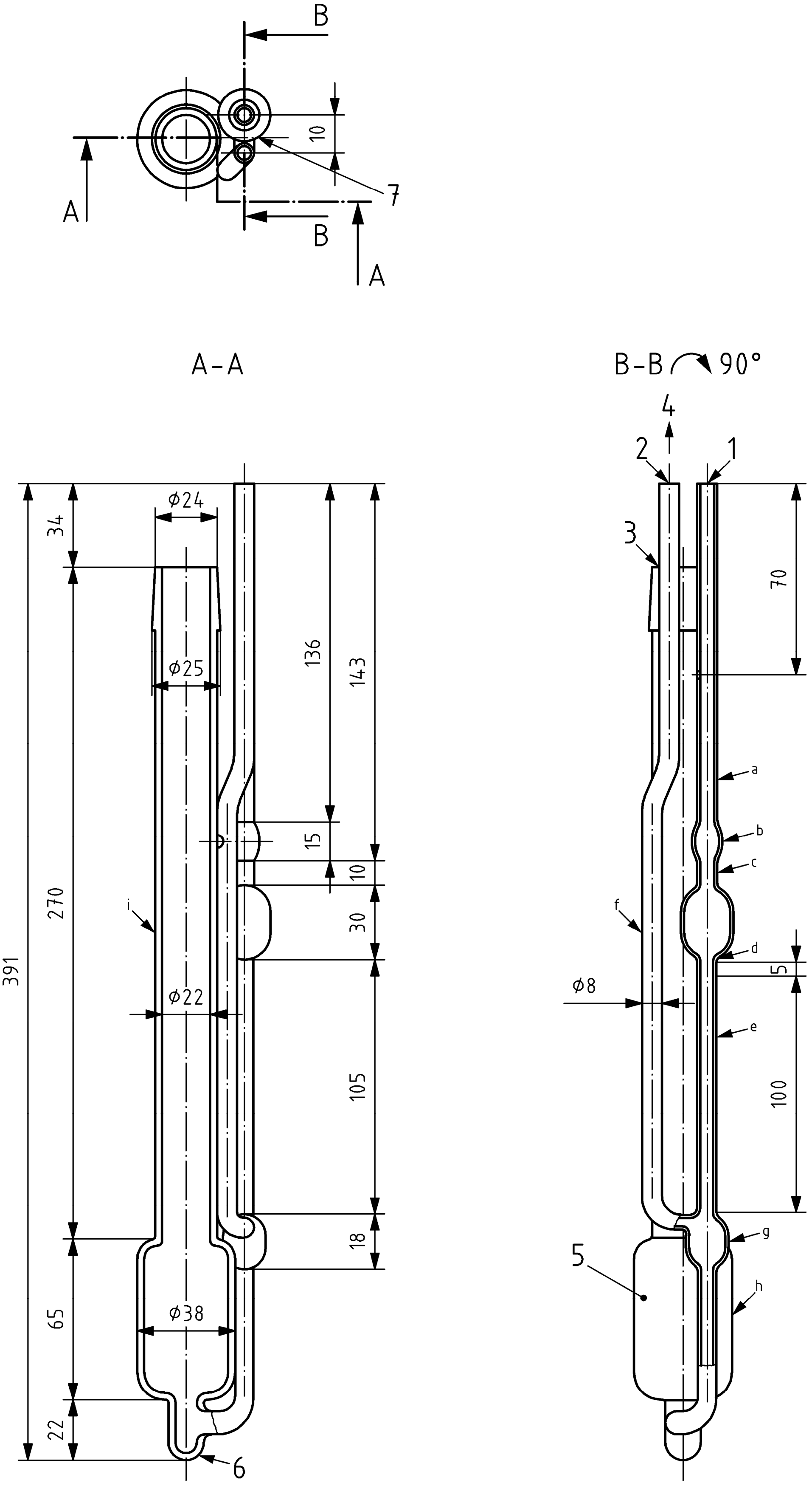
The tests examined use five types of viscometers:

- Gravity flow capillary viscometers (see Figure 7);
- Vacuum capillary viscometers;

- Reverse flow capillary viscometers;
- Differential pressure stainless steel capillary viscometers;
- Screw extrusion capillary rheometers;

Each of them has different geometric dimensions to perform the measurement of rheological parameter in the range of interest; they require a temperature control bath for the material and the apparatus employed.

Dimensions in millimetres



**Key**

1 tube 1
2 tube 2
3 tube 3
4 open to atmosphere
5 reservoir
6 bottom wall
7 bridges

a 2 mm capillary
b overflow trap 15 mm O. D.
c upper index mark
d lower index mark, Index bulb 26 mm O. D.
e capillary bores 0,6 mm, 0,7 mm, 0,8 mm
f SW tubing 6 mm
g suspended level bulb 17 mm O. D.
h heavy-wall tubing 8 mm
i SW tubing 22 mm

**Figure 7 — Gravity flow capillary viscometer (Modified Ubbelohde type)**

**3.1.1.3** What is measured and how

Normally the test examined measures the following different type of viscosity for Newtonian

fluids: 1) Apparent viscosity, 2) Dynamic viscosity and 3) Kinematic viscosity.

In these cases the kinematic viscosity (the resistance of a fluid to flow under gravity) is determined by measuring the time for a fixed volume of fluid to flow through the capillary calibrated glass viscometers and multiplying it by the viscometer calibration factor. The viscosity or coefficient of viscosity is obtained multiplying the kinematic viscosity for the density of the fluid. Dynamic viscosity is the same of viscosity or coefficient of viscosity. Apparent viscosity is the ratio between apparent shear stress to apparent shear rate. Where apparent shear stress may be determined by measuring the die entrance pressure for a specific die, then applying appropriate geometric factors; apparent shear rate is the shear rate of the material passing through the capillary die.

Only in some methods are measured:

— Relative, inherent, intrinsic viscosity

— Viscosity index;

All these parameters are related to mass concentration of material:

— relative viscosity the ratio of the viscosity of the solution to the viscosity of the solvent;

— inherent viscosity the ratio of the natural logarithm of the relative viscosity to the mass concentration of the material;

— intrinsic viscosity the limiting value of the reduced viscosity of the inherent viscosity at a infinite dilution of the material examined;

The range of Kinematic viscosity is 0,5-100.000 cSt (centistokes), where cSt = 1 $mm^2/s$

**3.1.1.4** Material to be examined

The test are mainly employed for the following materials:

— Transparent and opaque liquids;

— Road oils and bitumens;

— Petroleum products;

— Dilute polymers solutions

- Solutions of polymers;
- Thermoplastic compounds.

**3.1.1.5** Feasibility of methods to the materials of investigation

None of the methods examined or of the apparatus used can be transferred to the materials of investigation; it can be due to two main factors:

- Materials for horizontal project are
  - treated bio-waste
  - sludges
  - soils
- None of them have a Newtonian behaviour and the dimension is not feasible with those of gravity or glass capillary rheometers;
- The methods examined are related to laboratory tests not well accepted in the strategy where the widest diffusion of methods is preferred.

On conclusion all the methods of capillary rheometers section cannot be adopted for Horizontal.

### 3.1.2 Penetrometer

A lot of standard methods are provided for testing the consistency of a wide range of materials from fluid fresh mortars to solid soils by measuring the resistance to penetration of cylindrical or conical tips. The methods can also be used to establish a relationship between penetrometer resistance and water content of the sample.

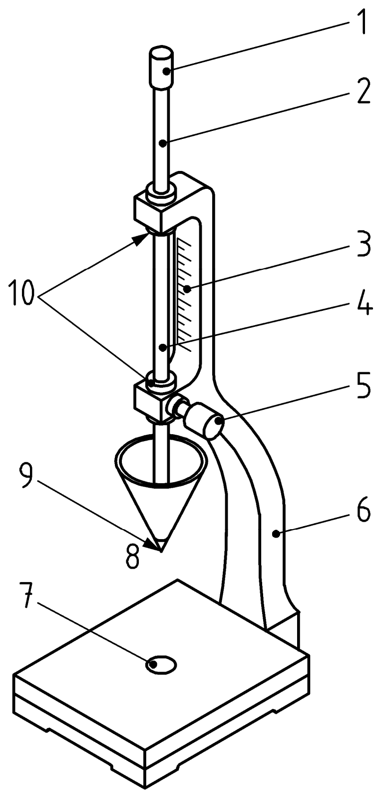
**3.1.2.1** Laboratory or field test feasibility

Laboratory penetrometers are quite simple instruments that could also be used for field tests as they do not request electric power supply and can operate at room temperature. The field soil penetrometer are specific apparatus designed to supply data on engineering properties of soil and include cone and friction-cone penetrometer of both mechanical and electric types.

**3.1.2.2** Apparatus

In the examined methods the following seven apparatus are described:

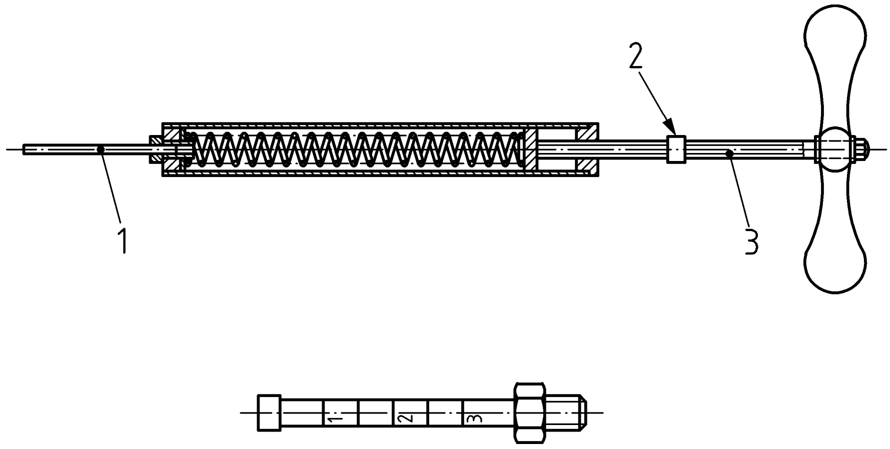
- Laboratory penetration tester with pointed steel rods
- Vicat apparatus with cylindrical or conical plunger (see Figure 8)
- Laboratory soil penetrometer consisting of specific spring dynamometer and a set of needles of various end area (see Figure 9)
- Field soil penetrometer
- Magnesium penetrometer cone for yield stress measurements (see Figure 10)
- Falling needle viscometer
- Vertical tube falling ball viscometer




**Key**

1 weight
2 plunger support rod
3 modified scale
4 graduation mark
5 lock screw
6 hinged support bracket
7 rubber insert
8 conical plunger
9 stainless steel tip
10 TFE_Fluocarbon bushings

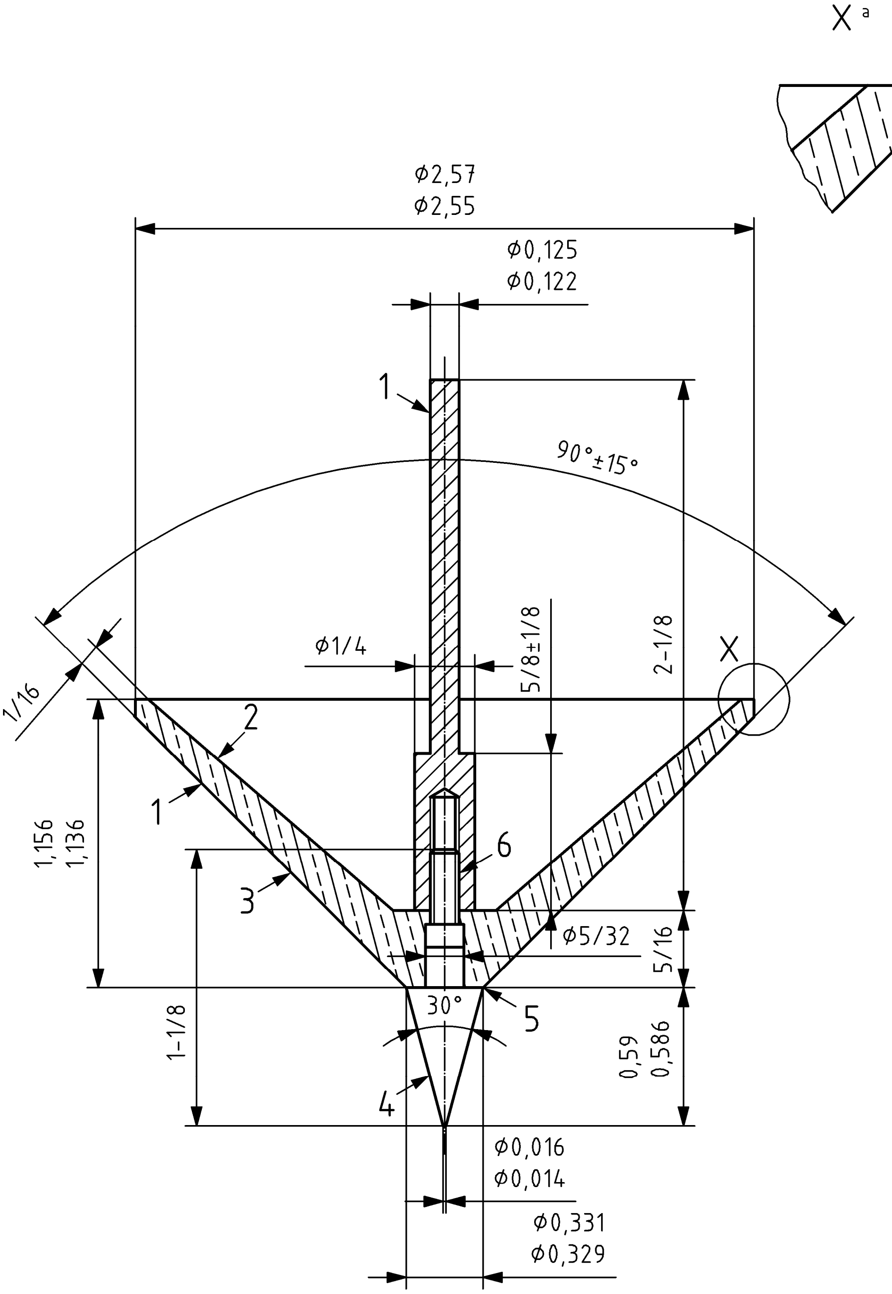
**Figure 8 — Modified vicat apparatus (Conical pluger method)**




**Key**

1 needle
2 sliding ring
3 stem

**Figure 9 — Soil penetrometer**




**Key**

1 magnesium
2 machine to desired weight
3 smooth and polished surface
4 magnesium tip
5 no shoulder
6 6-32 N.C. thread
7 break all sharp corners

**Figure 10 — Magnesium penetrometer cone [40]**

**3.1.2.3** What is measured and how

Laboratory apparatus for cement and gypsum consistency tests consist of a frame bearing a movable rod of fixed diameter, loaded with variable weights, with the lower end cylindrical or conical. The standard cone or needle is released from a certain height and allowed to drop freely into the sample. The depth of penetration in a fixed time interval is measured at constant temperature. A graduated scale attached to the frame permits to measure the penetration of the plunger. The results are expressed as mm of rod penetration at the test conditions. The consistency can also be expressed as the number of ml of water to be added to 100 g of the sample to obtain a determined rod penetration.

The laboratory soil penetrometer consists of a special spring dynamometer with pressure-indicating scale graduated in kg and an attached needle of known terminal area, selectable among a set of different needles according to the consistency of the sample. The operator shall measure the resistance to penetration of a mixture of soil, sieved at 4,75 mm and compacted with water, by use of the soil penetrometer with attached needle of known area.

The results calculated by the ratio between the penetrating force and the needle terminal area are expressed in kPa. The field soil penetrometer measures the end bearing and side friction component penetration resistance, which are developed during the steady slow penetration of pointed rod into soil. The apparatus uses both cone and friction-cone tips and may be mechanical or electric type. The mechanical penetrometer uses a set of inner rods to operate a telescoping penetrometer tips and to transmit the components of penetration resistance to the surface for measurements. The electric penetrometer uses electric-force transducers built into a non-telescoping penetrometer tip for measuring the components of penetration resistance. The results are reported as cone and friction-cone resistance expressed in 100 kPa with depth in metres. The method for yield stress measurement of heterogeneous propellant makes use of a standard magnesium cone penetrometer. The penetration is determined at 25 °C by releasing the cone-test rod assembly from the penetrometer and allowing the assembly to drop for 5 s. The cone will be essentially at the rest in less than this time, so the exact timing is not critical.

The yield stress is the measure of the maximum shear stress that can be applied without causing permanent deformation and is expressed in Pa. This value is calculated by measuring the cone penetration depth and by using a proper equation that takes into consideration the balance of the involved forces. Both falling needle and falling ball viscometer methods consist of determining the liquid viscosity of fluids by measuring the velocity of cylindrical needles or steel balls as they fall through the test liquid under the influence of gravity. In the falling needle method it is possible to measure the apparent viscosity and the yield stress of non-Newtonian liquids by using a series of needles of the same geometry but different densities.

**3.1.2.4** Material to be examined

The methods are utilised to test the following materials:

- Thermal insulating cement
- Gypsum plaster and concrete
- Fine-grained soil
- Soil and soft rocks
- Fresh mortar and cement
- Heterogeneous propellants both of the gel and emulsion type
- Polymers and resins emulsified or dispersed in the liquid
- Thermosetting moulding compounds and prepregs
- Bituminous materials

— Construction sealant

Generally the materials are in the solid form or as concentrated water suspensions.

#### 3.1.2.5 Feasibility of methods to the materials of investigation

The laboratory penetrometer designed to test the consistency of fresh mortar, gypsum and concrete could be used to test the flowability of concentrated sludge or sludges at higher dry content as these materials may assume a paste -like consistency. The yield stress determination by cone penetrometer, described in *ASTM D 2884* method, could also be applied to test the flowability of concentrated sludge. In fact the yield stress is considered a reliable parameter to test the flowability of non-Newtonian materials. These instruments could also be used for field tests, as they are very simple to operate and do not request electric power supply or accessory apparatus. The soil laboratory penetrometer that is used to test the consistency of compact mixtures of soil and water cannot be used for flowability tests, but it could be utilised to measure the solidity limit. The field soil penetrometer is a special apparatus designed to test very compact material. The falling needle and falling ball viscometers are laboratory devices that can measure the absolute viscosity of various liquids. Their use is not recommended with non-homogeneous fluids due to the uneven falling velocity of the ball or of the needle caused by the presence of coarse particles. Therefore these instruments are not suitable for analyses of the materials of investigation.

### 3.1.3 Rotational viscometers

Rotational viscometers are commonly used to measure the absolute viscosity of liquids of different physical and chemical characteristics in a wide range of viscosity values. A lot of standard methods are proposed by using different apparatus and procedures according to material and properties to be analysed.

**3.1.3.1** Laboratory or field test feasibility

As a small variation of sample temperature will result in a significant change in viscosity, all the tests must be carried out in laboratory using a thermostatic bath or at carefully controlled room temperature.

**3.1.3.2** Apparatus

Different types of rotational viscometers are reported in the methods:

— Brookfield viscometers (see Figure 11)

— Coaxial cylinder viscometers (see Figure 12)

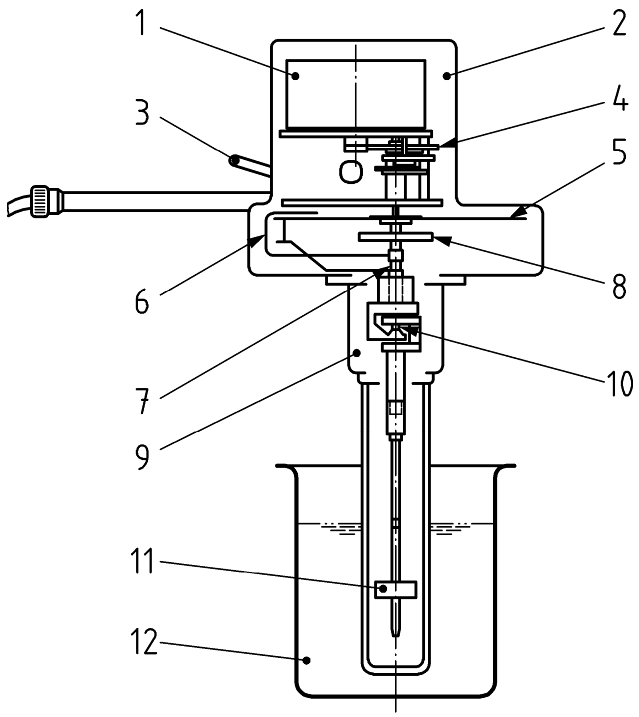
— Stormer viscometers (see Figure 13)

— Vane device with torque measuring system

— Controlled shear stress and controlled shear rate mini-rotary viscometer

— Torsion strain oscillating rheometer

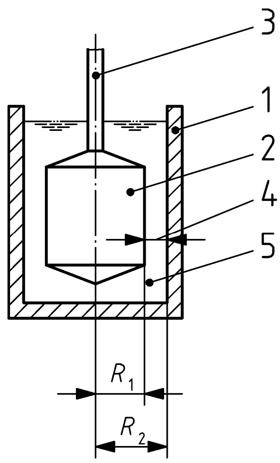
Brookfield and coaxial cylinder viscometers are used to measure the rheological properties of Newtonian, non-Newtonian and time dependent materials, while Stormer viscometer is generally used for consistency tests of paints and adhesives. The Vane device is used to test the shear strength of compact material as clay soils. Controlled shear stress and controlled shear rate mini rotor viscometers are applicable to measure the rheological properties of Newtonian and non-Newtonian oils at low and high temperatures. To test the viscoelastic properties of materials as rubber compounds a torsion strain oscillating rheometer is proposed.




**Key**

1 synchronous motor
2 housing
3 clutch
4 gear train
5 dial
6 pointer
7 root shaft
8 calibrated special spring
9 cup
10 jewelled dealing
11 spindle
12 sample container

**Figure 11 — Brookfield viscometer**




**Key**

1 beaker
2 cylinder
3 axle (spindle)
4 thickness of sample being measured
5 sample under test

**Figure 12 — Coaxial cylinder viscometer**

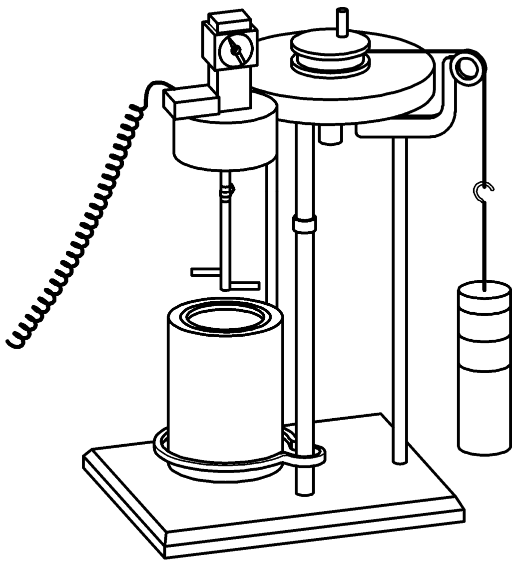


**Figure 13 — Stormer viscometer**

**3.1.3.3** What is measured and how

In rotational viscometers the fluid sample contained in a cylindrical cup is sheared as a result of the rotation of an internal cylinder or paddle -type rotor. Brookfield and coaxial cylinder viscometers utilise an apparatus that consists of an internal cylinder (bob) rotating in a static outer cylinder (cup) filled with the liquid to be examined immersed in a thermostatic bath at controlled temperature. The rotor is driven at fixed or programmed speeds by a DC motor. The resistance of the sample to flow is defined by measuring the torque applied to the rotor at the set speed. Signals proportional to the speed and torque are transmitted to a control unit for processing and display. By using a set of spindles and different rotational speeds a variety of viscosity range can be examined.

For Newtonian liquids the results are expressed as dynamic viscosity in Pa.s. The dynamic viscosity is defined as the ratio between the applied shear stress (Pa) proportional to the torque and the shear rate ($s^{-1}$) proportional to the rotating speed. For non-Newtonian liquids the following additional information can be obtained:

— Shear thinning index

  This index is calculated dividing the apparent viscosity at low rotational speed by the viscosity at a speed ten times higher. The higher the ratio the greater the shear thinning index.

— Degree of thixotropy

  The degree of thixotropy is measured by increasing and decreasing the viscometer speed stepwise and recording the viscosity values after ten revolutions at each speed.

  The degree of thixotropy is calculated from the ratio of the slowest speed viscosity taken with increasing speed to that with decreasing speed.

  The degree of thixotropy can also be calculated by the ratio of the slowest speed viscosity taken after rest period to that before the rest period. The higher the ratio the greater is the thixotropy.

Stormer viscometer consists of a double flag paddle -type rotor immersed in a cylindrical cup containing the sample. The rotor is driven (via a cord and pulley arrangement) by a falling weight. The standard defines the consistency as the weight required to producing a rotation frequency of 200 rpm. The consistency of paints applied to brush or rollers is generally expressed in Krebs Units (KU). This scale is the log function of the load necessary to produce 200 rpm. A calibration of the viscometer with different standard oils having assigned value of load to produce 200 rpm permits the conversion of load in grams to absolute viscosity in P. The normal range of the Stormer is covered by standard oils having a viscosity from 4 to 14 P (from 400 to 1400 mPa.s).

The miniature vane shear test may be used to obtain estimates of the shear strength of fine-grained soils. The vane assembly shall consist of four rectangular blades vane inserted in a cylindrical tube containing the sample that is rotated at a constant rate of 60 °/min to 90°/min by a motorised vane device. The torque required to cause a cylindrical surface to be sheared by the vane is measured by a torque transducer. The torque is then converted to a unit shearing resistance (Pa) of the cylindrical surface area by means of a vane blade calibration constant.

With mini-rotary constant shear stress viscometers it is possible to test the viscosity and the yield stress of oils at low temperature. Rotation of the rotor is achieved by applied load acting through a string wound around the rotor shaft. Time of rotation is measured electronically by a device attached to the timing wheel. The apparent viscosity is determined by attaching a fixed mass to the string and measuring the revolution time. The yield stress value of an undisturbed sample is determined by increasing gradually the load as far as to initiate the rotation of the internal cylinder. The rheological properties of viscoelastic material are measured by torsion strain oscillating rheometers. The elasticity of the sample is proportional to the delayed response between preset deformation and measured stress. By producing an oscillating movement of the driven text fixture elastic sample reacts with a delayed stress response characterised by its stress amplitude and phase shift in reference to the sinusoidal deformation. From both signal strain and stress characterised by their amplitude and phase shift it is possible to calculate the loss modulus, the storage modulus and the dynamic viscosity.

**3.1.3.4** Material to be examined

The methods are utilised to test the following materials:

— Free-flowing adhesives

— Automotive fluids and lubricants

— Paints and varnishes

— Bitumen and bituminous binders

— Epoxy resins

— Catalysed chemical grouts

— Clayey soil

— Raw rubber and unvulcanised rubber compounds

— Polymer and resin dispersions and emulsions

**3.1.3.5** Feasibility of methods to the materials of investigation

Brookfield and coaxial cylinder viscometers are used for laboratory testing of non-Newtonian and time dependent fluid. In order to study the rheological properties of non-Newtonian substances a diagram shear stress/shear rate is drawn to find the rheological model that best fits the experimental data. Brookfield viscometer is very simple to set and to operate and is particularly used in routine control laboratories. In Brookfield viscometers the fluid is contained in a cup whose radius is much larger than the rod. As there is a non linear distribution of the shear rates over the concentric gap, particularly for non-Newtonian fluids, to reduce the error in shear rate determination it is advisable to reduce the gap as small as possible. Coaxial cylinders viscometers designed to study non-Newtonian fluids use a measuring system with a narrow gap (ratio between the cup and bob radius <1.1). The narrow gap can create problems in measuring the viscosity of disperse systems due to the presence of coarse solid particles, but by the choice of the proper measuring system configuration and test procedures optimum conditions can be achieved. Rotational viscometers, both at controlled shear rate and controlled shear stress, cannot be used for field tests, but they can be adopted as reference laboratory methods to carry out parallel tests for the standardisation of simple field flowability methods. Stormer viscometer is used to measure the consistency of paints and adhesives. This equipment operates at a fixed shear rate and can be utilised, by calibration with standard oils, for measuring the viscosity of Newtonian substances in the limited range between about 200 to 5000 mPa.s. The Stormer viscometer method is not usable with non-Newtonian or thixotropic fluids where viscosity is a function of the shear rate and previous history of the sample. The miniature vane shear test method is applied to measure the shear strength of fine-grained soils but cannot be used to test the flowability of sludges.

### 3.1.4 "Flow" apparatus

Flow analysers described in the selected methods are quite simple devices used in empirically way to test the fluidity or consistency of various materials for quality control purpose. Generally the time for fluid material to flow under gravity from a standardised vessel through an orifice or through a tube is measured. For pourable fluids the consistency may be determined by measuring the distance covered by the sample freely flowing along a horizontal channel. For paste-like substances the consistency is expressed as the diameter occupied by a cone shaped sample after it has been spread on a vibrating table or by the measure of the compactability time. Different non standardized techniques such as inclined plane test, modified slump test and extrusion tube viscometer have been proposed by different authors for the determination of yield stress of viscoplastic materials based on gravity-induced flow or deformation of the sample. The yield stress has practical and fundamental significance since it is related to the physical properties of internal structures and it governs flow start and flow stoppage. The disadvantage with many of these simple techniques is their inability to generate more than one value to characterise the fluid.

**3.1.4.1** Laboratory or field test feasibility

Many of the selected flow methods may be utilised for field tests as they make use of simple equipment and analytical procedures.

**3.1.4.2** Apparatus

In the examined methods the following main types of apparatus are described:

— Flow table;

— Flow channels;

— Flow cones (see Figure 14);

— Hollow perforated tube (see Figure 15);

— Extrusion tube viscometer (see Figure 16);

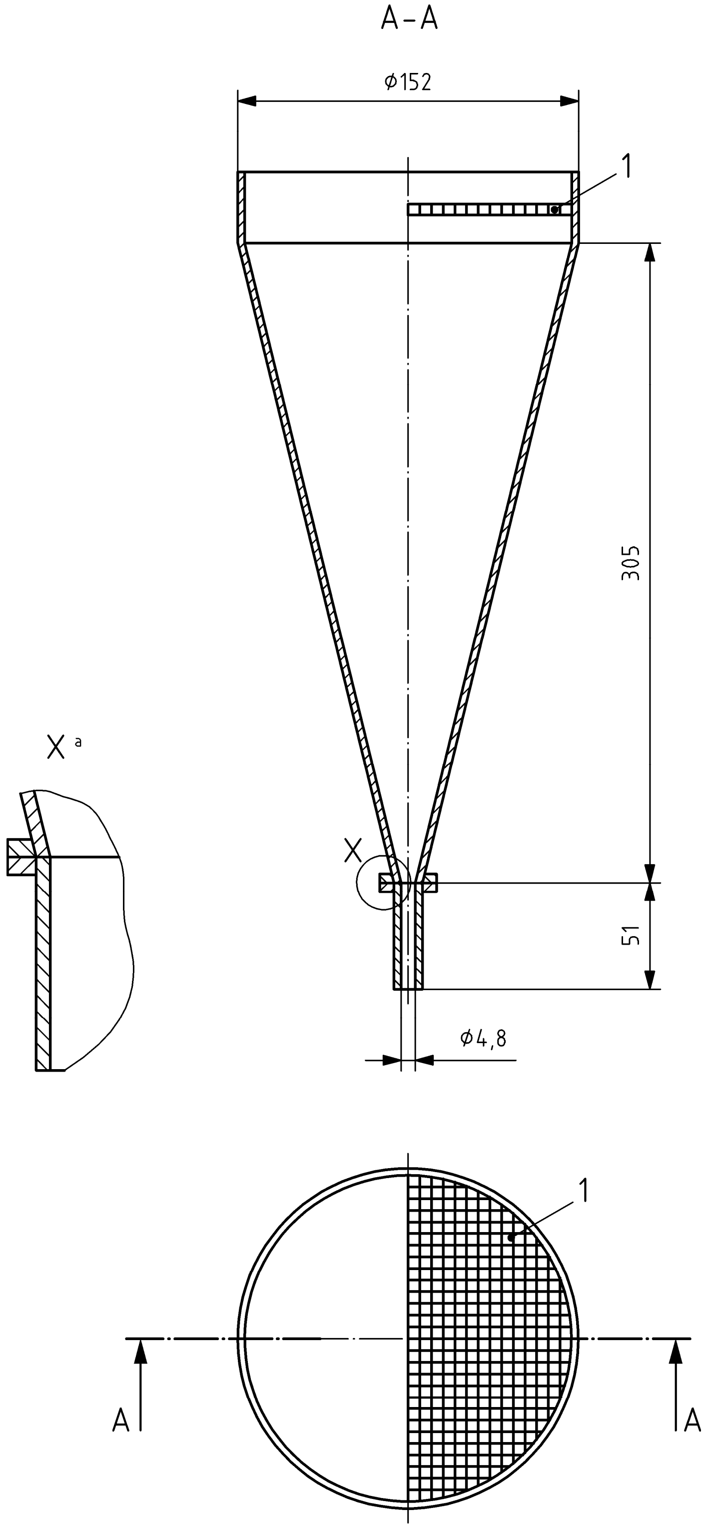
— Inclined plane;

— Modified slump test;

— Paint can.

Another type of apparatus that is not reported in the standard methods, but may be considered a promising tool to test the flowability of concentrated sludges is the extrusion tube viscometer (Kasumeter) described by Schulze von B., et al., 1991 [62] and Spinosa and Lotito, 2003 [63], (see Figure 16).

Dimensions in millimetres

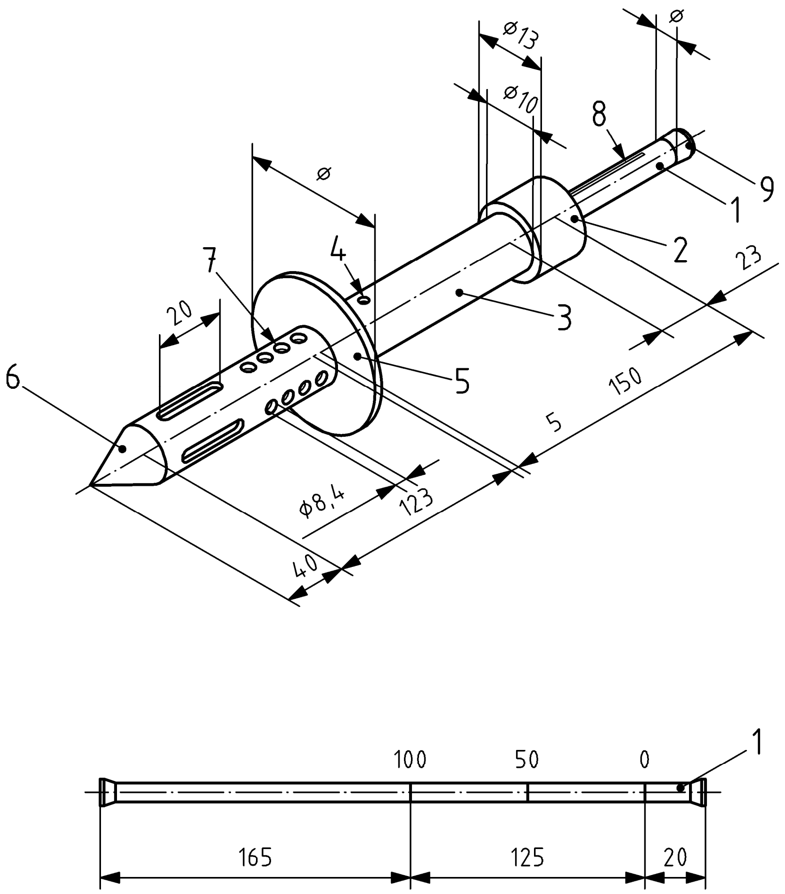



**Key**

1 semi-circular shaped sieve

**Figure 14 — Flow cone**

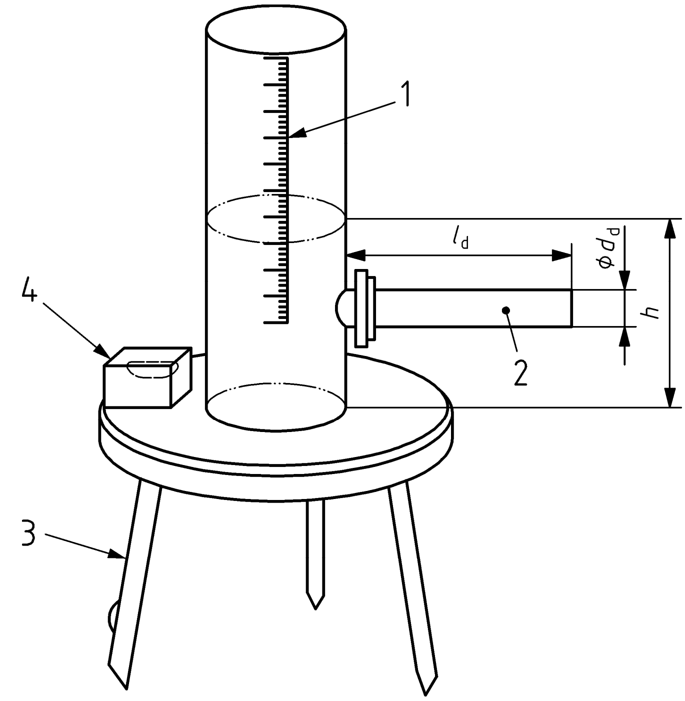
Dimensions in millimetres




**Key**

| | |
|---|---|
| 1 | measuring rod |
| 2 | scale datum |
| 3 | hollow tube |
| 4 | holding pin |
| 5 | collar floater |
| 6 | tube point |
| 7 | round holes |
| 8 | scale |
| 9 | cap |

**Figure 15 — Hollow perforated tube for flow tests**




**Key**

1 scale
2 capillary tube
3 tripod
4 bubble level

**Figure 16 — Tube extrusion viscometer (Kasumeter)**

**3.1.4.3** What is measured and how

Flow tables are used to test the consistency of mortar and fresh concrete by submitting a conical sample to operation of a vibrating table. The consistency is expressed as % or mm of sample spread on the table after vibration. In the Vebè test the time necessary for the compactability of a conical sample put on a vibrating table is measured. Horizontal flow channel apparatus is used to test the consistency of pre-blended expansion mortars for grouting and other pourable fluid substances. A determined volume of the sample is introduced in a funnel placed upright over a horizontal channel. The sample is let to flow from the funnel into the channel and the consistency is determined by measuring the distance covered by the sample along the channel. The fluidity of cementitious injection products and other fluid substances is often measured by the flow cone method. A specified volume of the sample is introduced in a standardised cone put in a stand in vertical steady position. The sample is let to flow from the cone into a receiving container through a calibrated discharge tube. The fluidity of the product is expressed as efflux time measured in seconds. To avoid unevenly flow or a complete stop of the flow due to the presence of course solid particles or lumps of unmixed material, the flow cone is provided with a semicircular shaped sieve for a pre-treatment of the sample. The accuracy of the apparatus is periodically tested by using water as reference fluid. Flow test method in accordance with ASTM C 1362 measures the flow of concrete or mortar into a perforated tube. This method is applicable for concrete with coarse aggregates less than 37,5 mm in diameter. The instrument consists of a hollow perforated tube that is inserted in the concrete sample, after which the material is allowed to flow into the hollow tube. The height of sample in the hollow tube measured by a calibrating rod is considered a measure of the flow properties of the concrete. In the following non-standardized methods the yield stress is measured by different techniques, since it is considered as reliable parameters to define the flowability of visco-plastic sludges. The extrusion tube viscometer (Kasumeter) consists of a cylindrical container to which calibrated pipes of different diameters can be fixed at the bottom. By measuring the height of the sludge suspension remaining in the container at the end of continuous flow, the yield stress can be calculated. The inclined plane test method calculates the yield stress of pasty materials measuring the final depth of the sludge remaining on the inclined plane at the end of the gravity induced flow of a certain volume of a fluid. In the modified slump test method the yield stress of pasty materials is calculated measuring the final height loss of a volume of a fluid contained in a PVC cylinder, suddenly left flowing on a horizontal plane, by rapidly lifting the cylinder. Fluids with high yield stress values can be analysed by putting a cylindrical hat on the top of the samples, in which several masses can be added.

In the standard test method for determining whether a material is a liquid or a solid described in [56. ASTM D4359] the material under test is held at 38 °C in a tightly closed can. The lid is removed and the can inverted. The flow of the material from the can is observed to determine whether it is a solid or a liquid. A material e.g. that flows a total of 50 mm or less within 3 min is considered a solid. Otherwise it is considered a liquid.

**3.1.4.4** Material to be examined

The methods are used to test the following materials:

— Mortar and grout mixtures

— Free-flowing adhesives

— Soil

— Cementitious injection products

— Fresh concrete

— Castable refractory

— Hydraulic fluids

— Sludges

— Paints and varnishes

— Polymeric and mineral suspensions

**3.1.4.5** Feasibility of methods to the materials of investigation

Flow table methods cannot be used for the evaluation of flowability of sludges as they are designed to test the consistency of high concentrated materials with none or poor tendency to flow. Horizontal flow channels are used as laboratory and field tests to analyse the flow properties of pourable fluids. The results obtained with this empirical equipment are utilised only for quality control purpose and are not comparable with viscosity values measured by reference laboratory methods. It follows that flow channel methods cannot be applied for the determination of flowability of all materials. Flow cones are simple devices commonly used for laboratory and field analyses of relative viscosity of fluid substances. Often for Newtonian fluids the results can be converted into absolute viscosity values by calibration with standards. For non-Newtonian and time dependent fluids the use of flow cone is not recommended. In fact Newtonian fluids flow out from the cone at constant rate because their flow rate is independent of shear rate. On the contrary non-Newtonian fluid flow out at different initial and final velocity due to the decrease of shear rate caused by the decreasing hydrostatic force. Hence for such substances the efflux time is not linearly dependent on viscosity. Materials that are highly thixotropic generate flow time that are much longer than that predicted by steady-state concentric cylinder data because the total shear in flow cones is small compared with that of rotational viscometers.

The hollow perforated tube is a specific device for laboratory and field testing of concrete consistency. For field analyses of higher concentrated sludge, for which the value of yield stress can be considered a good indicator of their flowability, the application of a simple specific apparatus such as:

— extrusion tube viscometer (Kasumeter);

— inclined plane test ;

— modified slump test

should be evaluated.

In conclusion:

— flow cone could be used as field test for the determination of flowability of diluted sludges that show an approximately Newtonian behaviour;

— extrusion tube viscometer could be applied to the analysis of yield stress of non-Newtonian concentrated sludges;

— modified slump and inclined plane tests can be used for concentrated sludges showing a pasty behaviour to determine yield stress.

— In any case the development of reliable analytical procedures and the validation of the obtained results should be confirmed by parallel reference laboratory tests performed with rotational viscometers.

## 3.2 Solidity

— Shearing apparatus

— Vane testing apparatus

— Penetrometers

### 3.2.1 Shearing apparatus

The shearing apparatuses in this section include among other things the apparatuses for direct shear tests, triaxial compression tests and for tests for determination of unconfined compression strength.

#### 3.2.1.1 Laboratory or field test feasibility

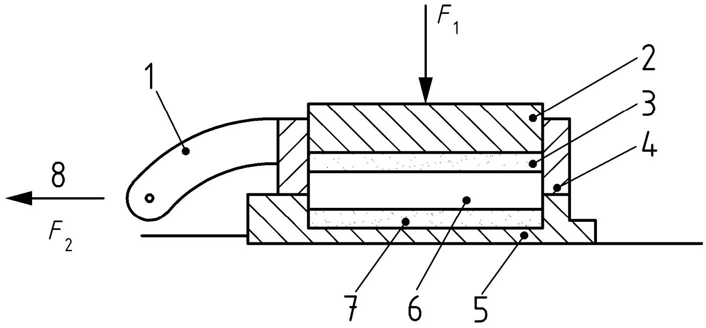
Most of these tests are performed in the laboratory, because the shear devices often require in parts large space, thermostatic conditions, special force loading devices, which need electrical power, etc.

#### 3.2.1.2 Apparatus

The tests examined use the following types of shearing apparatus:

— Shear devices (shear box) (Figure 17)

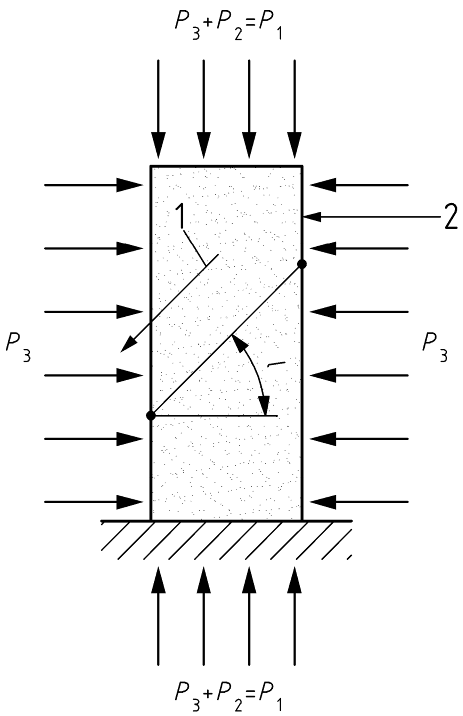
— Triaxial equipment (Figure 18)




**Key**

1 upper frame
2 piston
3 porous stone
4 upper frame
5 lower frame
6 specimen
7 porous stone
8 shearing force

**Figure 17 — Test samples in single and double shear (shear box) [31]**




**Key**

1 potential sliding failure
2 soil sample encased in a flexible membrane

**Figure 18 — Triaxial compression test showing test pressure a. assumed plane of failure *AB* [32]**

**3.2.1.3** What is measured and how

For the determination of shear strength by direct shear test described in *ASTM D3080* first moisten and drain the test sample under normal stress. Then unlock the frames that hold the test sample, and displace one frame from the shear device (shear box) horizontally and measure the shearing force and horizontal displacement.

For the determination of the load controlled cyclic triaxial strength described in *ASTM D5311* a number of factors have to be considered. It is a very expensive procedure to determine the shearing strength (*ASTM D2850*). That is why this test method will be discussed no further.

The test method for determining the shear resistance of Geosynthetic clay liner (GCL) described in *ASTM 6243* measures the total resistance to shear within a GCL or between a GCL and adjacent material. The shear force is recorded as a function of the horizontal displacement of the moving section of the shear box.

**3.2.1.4** Material to be examined

The tests are mainly employed for the following materials

— Soil material (e.g. saturated soils in either undisturbed or reconstituted states)

— Lime treated material

— Geosynthetic clay liner (GCL)

In general the materials are in solid forms.

**3.2.1.5** Feasibility of the methods to the materials of investigation

In these existing standards only soil materials are investigated. For application on other materials like sludge or bio-waste these materials must have a certain threshold of the consistency to produce reasonable results. For solid material, however, the shearing apparatus can be a reliable laboratory reference method to determinate the shear strength and thus the consistency. Especially the direct shear test method is suited for relatively rapid determination of consolidated drained strength properties, because the drainage paths through the test sample are short, thereby allowing excess pore pressure to be dissipated more rapidly than with other drained stress tests.

As the methods have to be suitable for the whole range of sludge materials and not only for soil materials the shearing apparatuses aren’t appropriate.

### 3.2.2 Vane testing apparatus

Vane testing apparatus are used to measure the vane shear and determine the shearing strength of compact materials like direct shear test or triaxial compression test. For the investigated materials it is important that these materials are cohesive, because otherwise no shearing strength occurs.

**3.2.2.1** Laboratory or field test feasibility

In general vane shear tests are applicable for both laboratory and field test. There are simple apparatus for measuring in the field and special apparatus for vane shear tests as laboratory reference. The laboratory apparatus can be driven by electric power or manually.

**3.2.2.2** Apparatus

The tests examined use the following types of vane testing apparatus:

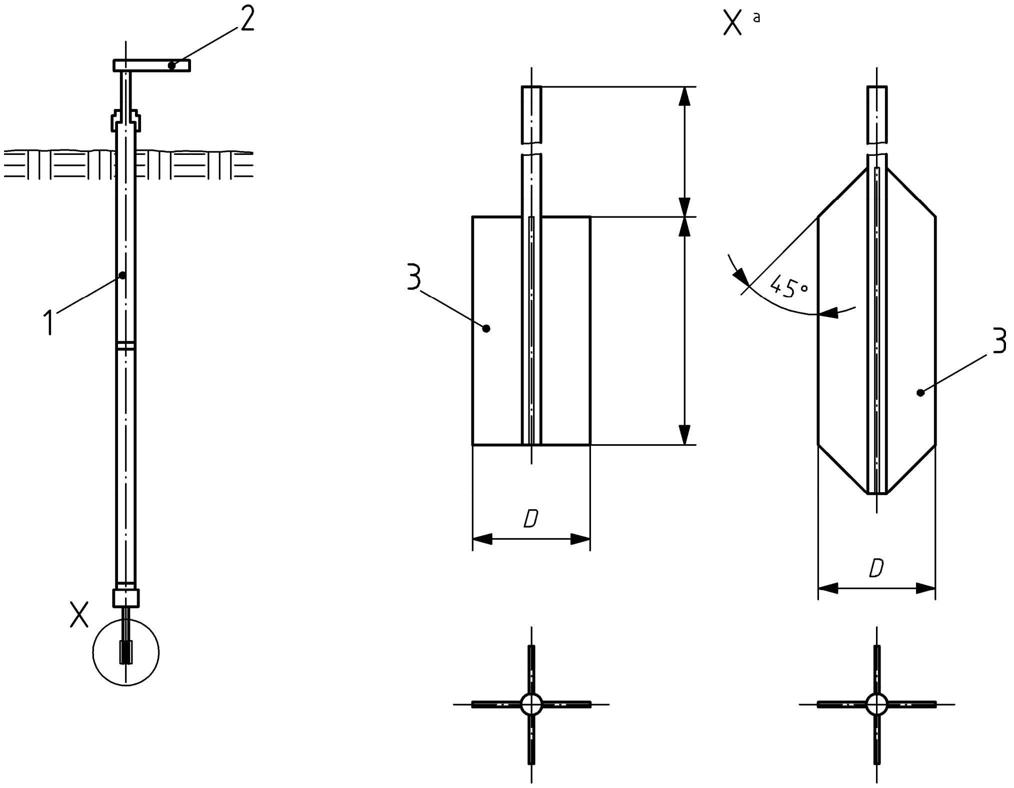
— Shear vane apparatus (four bladed vane; in situ; Figure 19)

— Vane device with torque measuring system (laboratory miniature vane; Figure 20)

Besides these apparatuses another types of apparatuses, which are not reported in the standards, but may be considered as a promising tool to test the solidity of the analysed materials - like concentrated sludges e.g.-, are the following apparatuses:

— Laboratory vane shear apparatus (Figure 21)

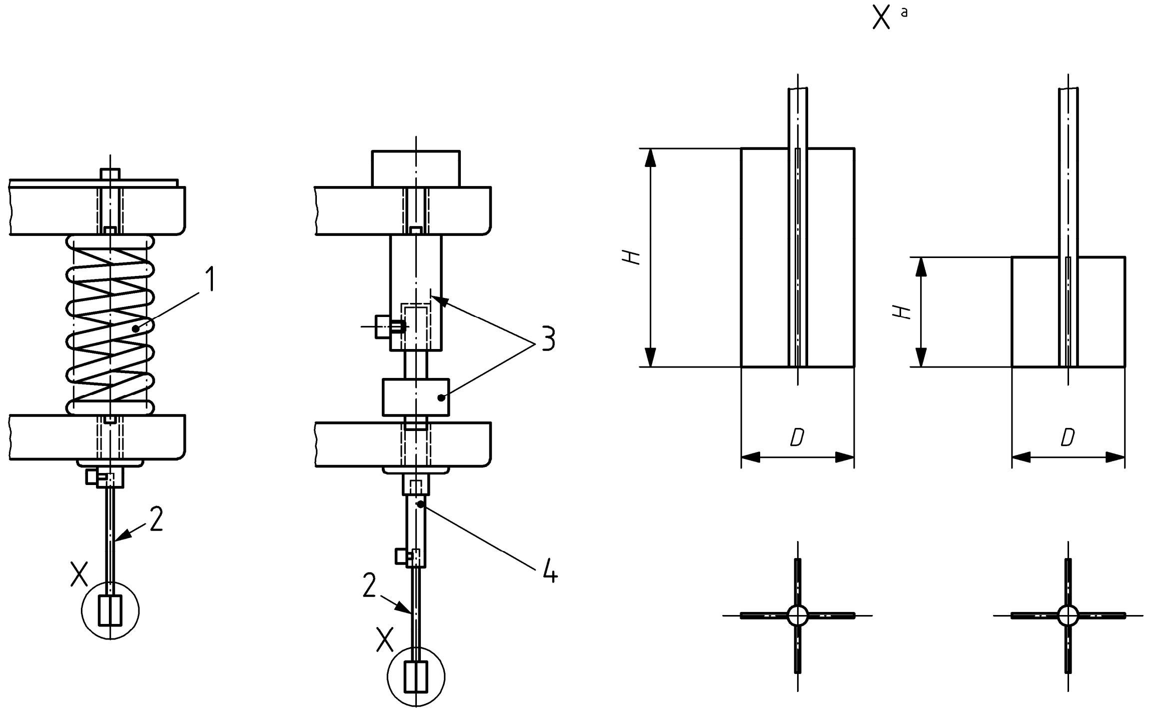
— Pocket vane shear apparatus (Figure 22)




**Key**

1 casing pipe
2 torque wrench
3 vane

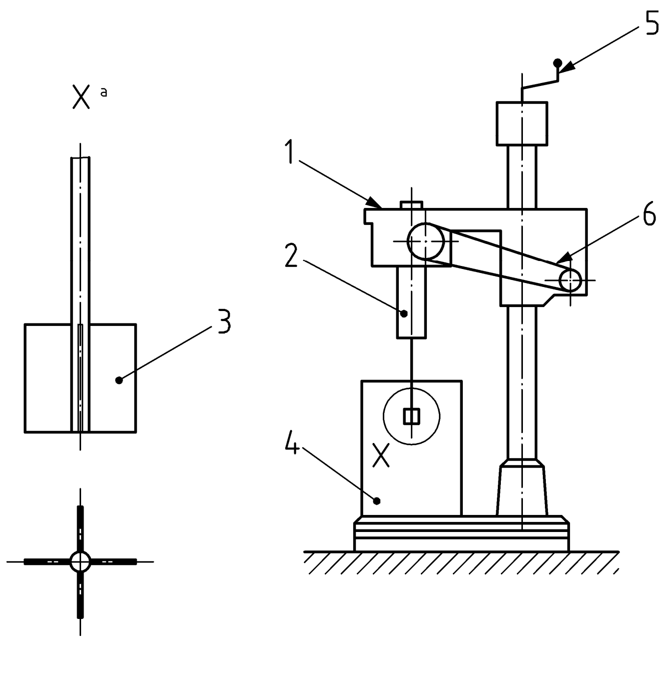
**Figure 19 — Vane shear apparatus and geometry of field vanes [32, 33]**




**Key**

1 calibrated torque spring
2 vane blade
3 torque spring spacer
4 full bridge strain gauge torque transducer

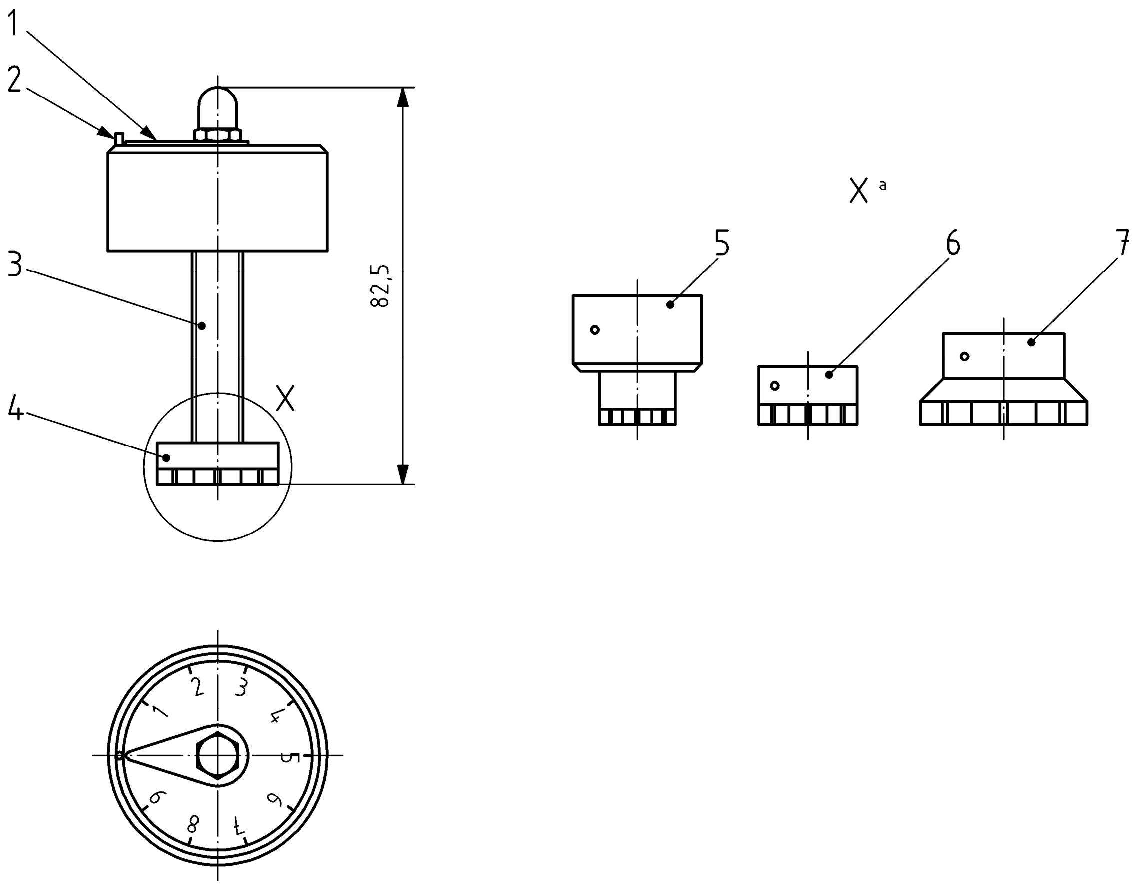
**Figure 20 — Vane torque spring, electrical transducer details geometry, miniature vane blade geometry [34]**




**Key**

1 angle of rotation display with drag indicator (360° scale)
2 spring with defined resistance
3 vane
4 specimen
5 height adjustment
6 electric drive for vane rotation

**Figure 21 — Laboratory vane shear apparatus [23]**




**Key**

1 pointer
2 carrier
3 spring
4 vane
5 vane No 1
6 vane No 2
7 vane No 3

**Figure 22 — Pocket vane shear apparatus [15]**

**3.2.2.3** What is measured and how

Vane shear testing described in *ASTM D2573* and *DIN 4094-4* is one of the most common in-situ methods for the estimation of the undrained shear strength of the soil. The vane is introduced into the borehole to the depth where the measurement of the undrained shear strength is required. Then it is rotated and the torsional force required to cause shearing is calculated.

The Laboratory miniature vane shear test described in *ASTM D4648* may be used to obtain estimates of the shear strength of fine-grained soils. The vane assembly shall consist of four rectangular blades. The vane inserted in a cylindrical tube containing the sample is rotated at a constant rate of 60 °/min to 90 °/min by a motorised vane device.

A torque transducer measures the torque required to cause a cylindrical surface to be sheared by the vane. The torque is then converted to a unit shearing resistance (Pa) of the cylindrical surface area by means of a vane blade calibration constant.

For determination shearing strength by laboratory vane shearing apparatus described in the *ATV-Arbeitsbericht (1989)* [23] first prepare the sample (e.g. *after DIN 18127 (1997)* [30]): The material – in this case sludge e.g. – is filled into the small Proctor vessel in three equal portions and is compressed with ten knocks by the small Proctor hammer (2,5 kg weight). Then the horizontal torque form shearing the sample through the penetrated vane is determined. The vane consists of four rectangular blades. The vane apparatus, which is connected with the display over a torque spring, is manually or electrically rotated and the vane cuts a cylindrical sample. The torque is determined by the angle of rotation and the spring constant. When the sample shears, the angle of rotation and the applied max. torque is kept by the scale. The torque is then converted to a shearing strength with the aid of a constant.

<u>The pocket vane shear apparatus</u> described in the publication *Mechanical properties of waterworks' sludges* [15] has only a height of 82,5 mm and does not need electrical power. Because of the small size this apparatus is capable for simple field tests. It consists of a shear apparatus with several vanes of different sizes, which can be applied for a wide range of materials. The pointer and carrier of the display indicate the value, which is converted to a shearing strength with the aid of a constant: The manufacturer of the apparatus has given scaling factors to transform the measured values to shearing strength. These factors are developed for soil measurements.

#### **3.2.2.4** Material to be examined

The tests are mainly employed for the following materials

- Soft saturated, cohesive soils
- Saturated fine-grained clayey soils
- Saturated fine-grained soils (clay, silt and organic soils)
- Sewage sludges for landfilling
- Waterworks' sludges

#### **3.2.2.5** Feasibility of the methods to the materials of investigation

In general the vane shear test is a test whose use is limited to the testing of cohesive materials and cannot be used on coarse-grained materials. For soil materials e.g. it is not suitable for clays containing any appreciable amount of silt or sand. Most of the materials, which should be investigated, have this property.

The described standards and investigations in the past [23, 15] showed the feasibility of the vane shearing test methods for cohesive materials like soils and sludges. For bio-waste and related wastes of interest further investigations are necessary.

### 3.2.3 Penetrometer

A lot of standard and non-standardised methods are provided for testing the consistency of a wide range of materials from fluid fresh mortars to solid soils by measuring the resistance to penetration of cylindrical, conical or spicular tips. The methods can also be used to establish a relationship between penetrometer resistance and water content of samples of identical materials.

#### **3.2.3.1** Laboratory or field test feasibility

Laboratory penetrometers are quite simple instruments, which could also be used for field tests as they do not request electric power supply and can operate at room temperature.

The field soil penetrometers are specific apparatus designed to supply data on engineering properties of soil and include cone and friction-cone penetrometers of both mechanical and electric types.

#### **3.2.3.2** Apparatus

The tests examined use the following types of penetrometer:

- Soil cone penetrometer (Figure 23)

— Vicat apparatus with different forms of needles (Figure 24)

— Laboratory penetration tester with three pointed steel rods/spears (Figure 25)

— Penetration piston, loading machine (CBR)

— Penetrometer, standard cone and other forms of cone tips (Figure 26)

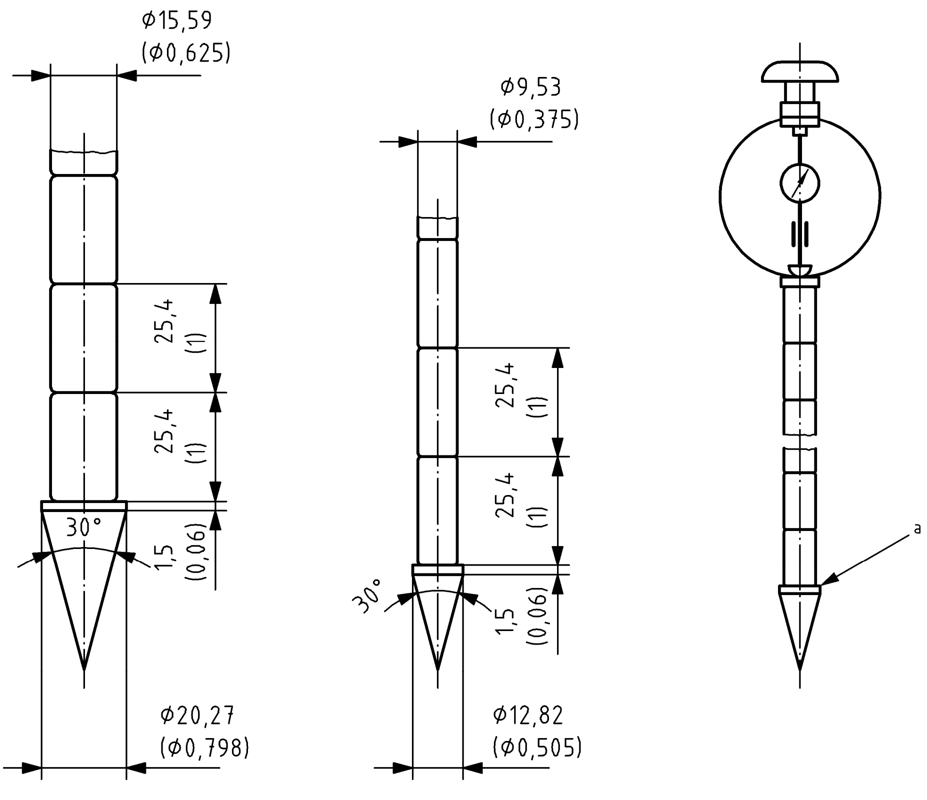
— Magnesium cone penetrometer for yield stress measurements (Figure 27)

— Field soil penetrometer (Field cone and friction cone penetrometer)

Besides these apparatuses other types of apparatuses, which are not reported in the standards, but may be considered as a promising tool to test the solidity of the analysed materials - like concentrated sludges -, are the following apparatuses:

— Pocket penetrometer after Neuschäfer (Figure 28)

— Pocket cylinder penetrometer (Figure 29)

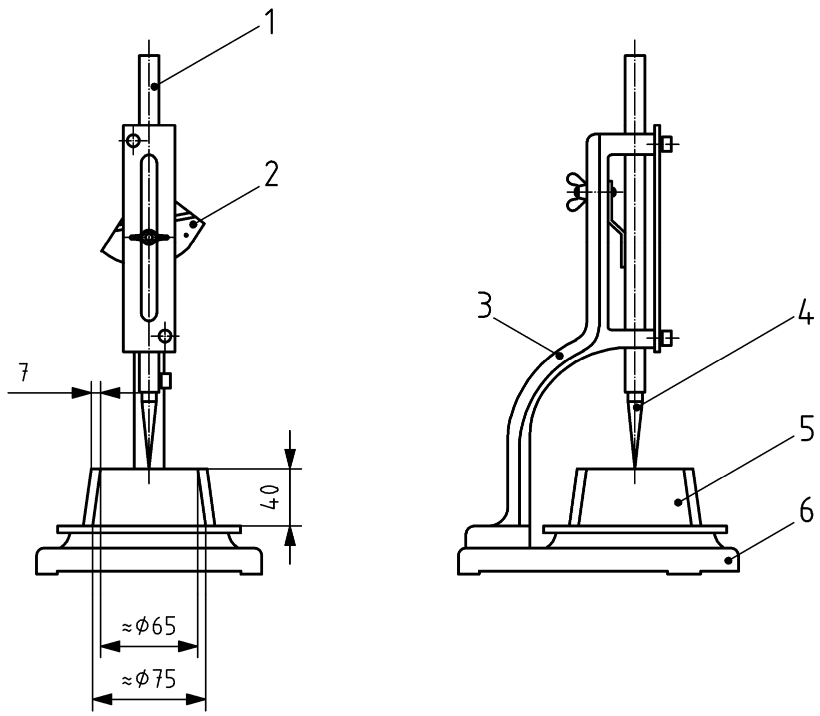



**Key**

a Use cone sizes A) or B)

**Figure 23 — Soil cone penetrometer [35]**

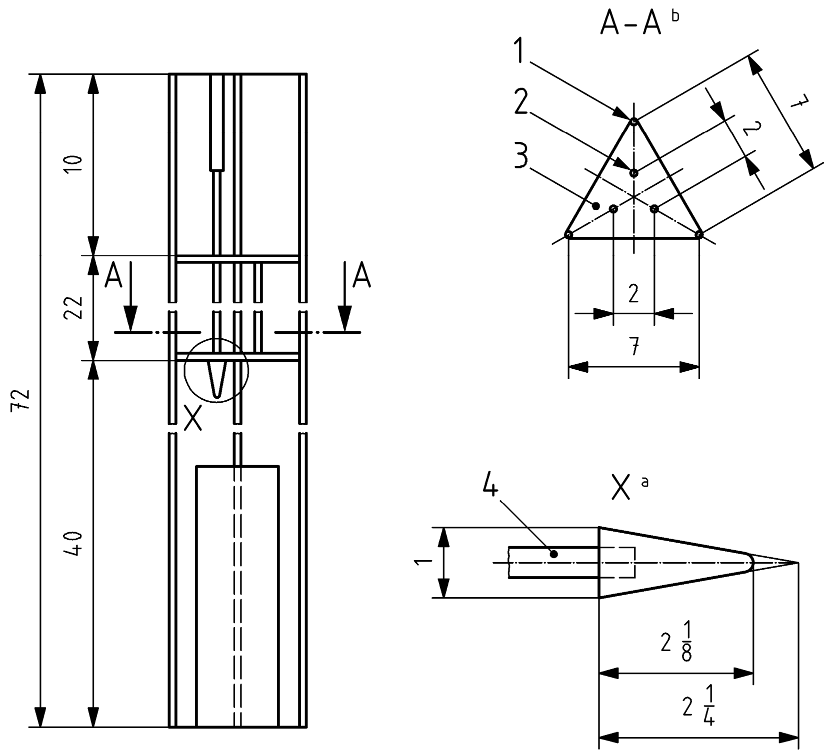
Dimensions in millimetres




**Key**

1 movable rod
2 initiating device
3 frame
4 plunger cone
5 rigid conical ring
6 glass plate

**Figure 24 — Vicat apparatuses [36]**




**Key**

1 supports-3-1/2 Pipe

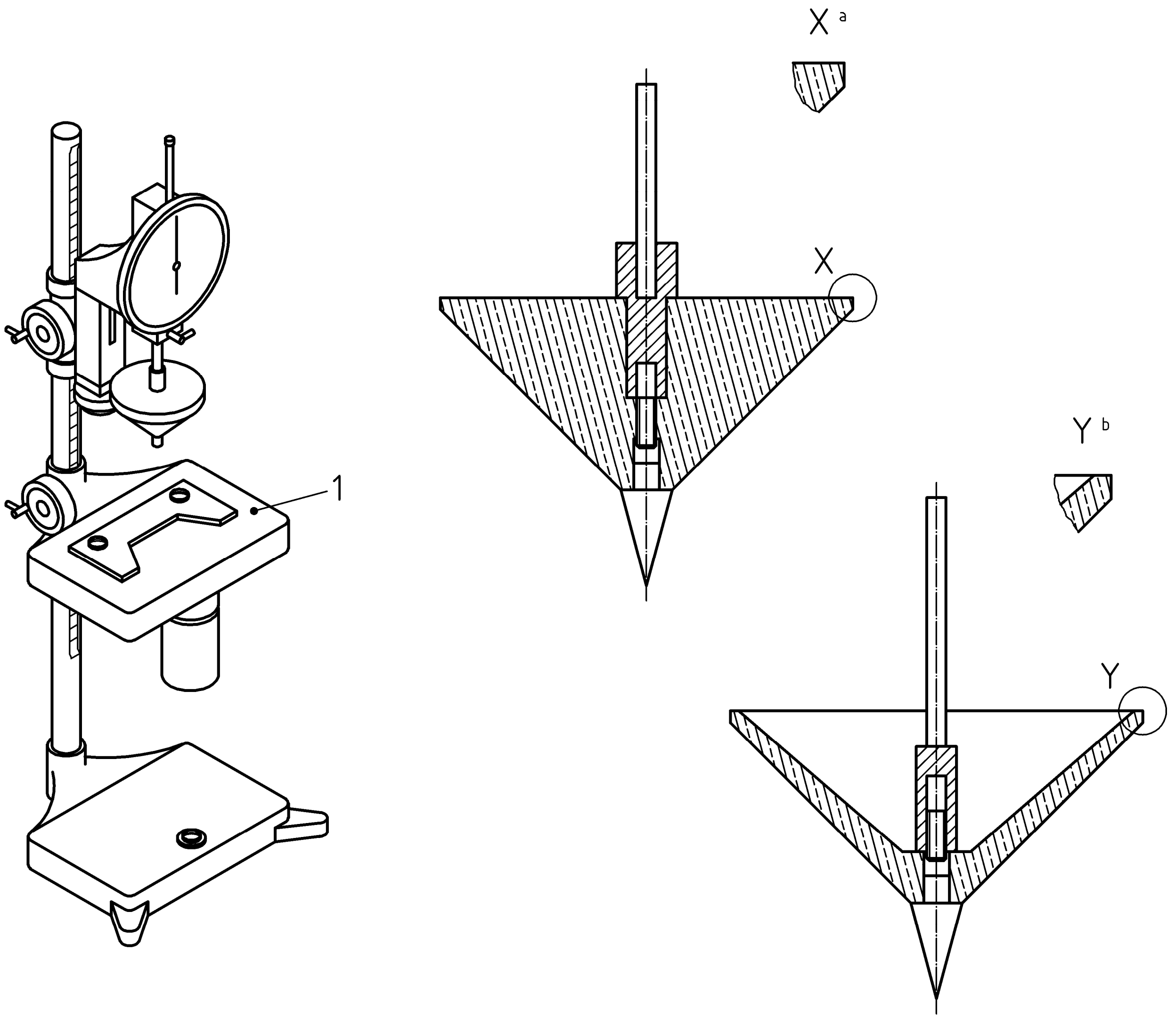
2 guides – 3-3/8 Pipe

3 section through supports and guides

4 3/8 Steel Rad-threaded into point

**Figure 25 — Apparatus for measuring consistency by penetration [37]**

Dimensions in millimetres




**Key**

1 centring device

**Figure 26 — Penetrometer with standard and optional penetration cone [38, 39]**

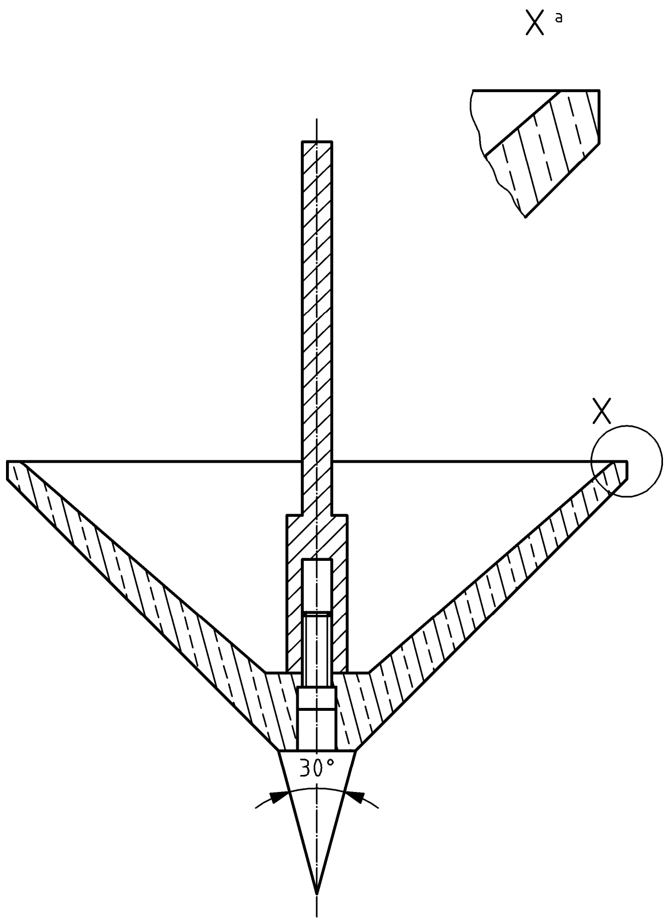
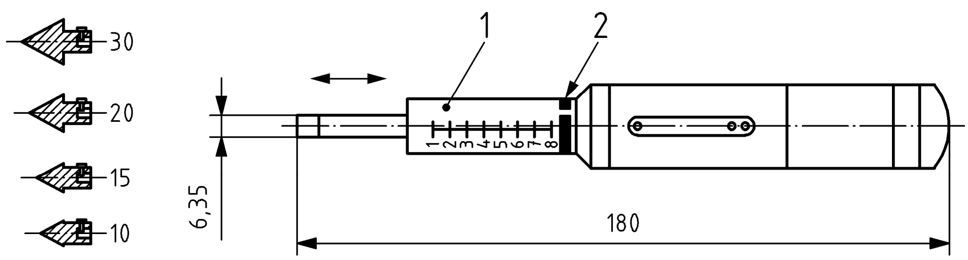



**Figure 27 — Magnesium penetrometer cone [40]**




**Key**

1 scale from 0 to 700 psi
2 indicator sleeve

**Figure 28 — Pocket penetrometer after Neuschäfer [23]**

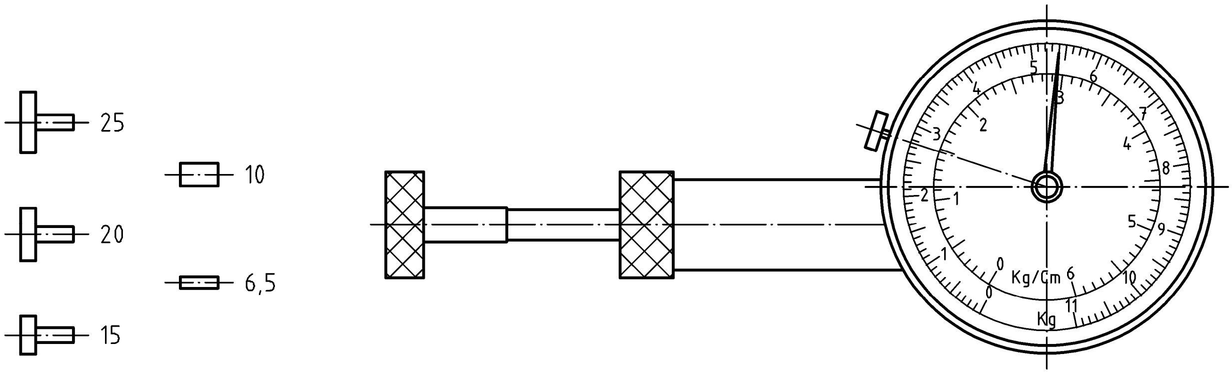



**Figure 29 — Pocket cylinder penetrometer [41]**

**3.2.3.3** What is measured and how

The Soil Cone Penetrometer described in *ASAE S313.2 / ASAE S313.3*, which is operated by hand, has two cone base sizes. After selection the adequate cone is pushed into the soil at a uniform rate of approximately 30 mm/s. The surface reading is measured at the instant the base of the cone is flush with the soil surface. Marking on the shaft of the cone penetrometer indicates the depths. Five to seven readings should be taken to establish the cone index and to verify the presence of unique layers in the soil profile.

For determination of consistency by Vicat apparatuses described in *ASTM C187*, *ASTM C807* and *DIN 1168-2* first the sample is prepared. Contact the needle with the surface of the sample and then release the needle. For the standard ASTM C187 the paste shall be a normal consistency if the rod settles to a point (10 ± 1) mm below the orig. surface in 30 s after being released. For the standard ASTM C807 the Vicat apparatus is modified and the preparation is much more complex. Settlement for 30 s, then determine the penetration of the needle at this time and every 30 min until the needle fails to penetrate to the bottom of the mould (time of setting). In the standard DIN 1168-2 the duration (in minutes) the cone sticks at a defined depth is measured. The beginning of stiffening is reached if the needle remains in the sample at (18±2) mm above the bottom. The duration is measured from the filling in the mould till sticking in the sample. The tips can also be in form of a plunger.

The penetration tester described in *ASTM C405* consists of three pointed steel rods resp. spears. After preparation of the sample release the spears by means of the electrical switch. Measuring the depth of penetration of each of the spears 30 s later, and take the average depth of penetration of the three spears as the measure of the consistency for that test.

For the determination of the California bearing ratio (CBR) described in *ASTM 1883* use laboratory compacted samples. The test apparatus consists of a penetration piston and loading machine. Measuring of curves of load-penetration-relation and determination of the water content is performed.

For measuring the consistency with the (standard cone) penetrometer partly described in *ASTM D217, ASTM D937, BS 2000-49, BS 3712, EN 13880-2, DIN 51580, DIN EN 1426, ISO/DIS 13765-1 and ISO 2137* and in ref. [55] determine the depth, in tenth of a millimetre, that the needle or cone (in part several cone tips of different sizes) penetrates the sample under specific conditions of weight/load (~150mg-150g depending on the analysed material), time (fixed duration of ~5s), and temperature (~25°C); mostly classification of penetration range, e.g. (500x0,1) mm.

The method for yield stress measurement of heterogeneous propellant makes use of a standard magnesium cone penetrometer described in *ASTM D2884*. This method is similar to the previous described methods. The penetration is determined at 25 °C by releasing the cone-test rod assembly from the penetrometer and allowing the assembly to drop for 5 s. The cone will essentially be at the rest in less than this time, so the exact timing is not critical.

The yield stress is the measure of the maximum shear stress that can be applied without causing permanent deformation and is expressed in Pa. This value is calculated by measuring the cone penetration depth and by using a proper equation that takes into consideration the balance of involved forces.

In the standard test method for deep, quasi-static, cone and friction-cone penetration tests of soil described in *ASTM D3441* the end bearing and side friction component penetration resistance is measured, which are developed during the steady slow penetration of a pointed rod into soil. The apparatus uses both cone and friction-cone tips and may be mechanical or electric type. The mechanical penetrometer uses a set of inner rods to operate a telescoping penetrometer tips and to transmit the components of penetration resistance to the surface for measurements. The electric penetrometer uses electric-force transducers built into a non-telescoping penetrometer tip for measuring the components of penetration resistance. The results are reported as cone and friction-cone resistance expressed in 100 kPa with depth in metres.

In the same way operates the standard test method for performing electronic friction cone and piezocone penetration testing of soils described in *ASTM D5778*: A penetrometer tip with a conical point having a 60° apex angle and a cone base area of 10 cm² or 15 cm² is advanced through the soil at a constant rate of 20 mm/s. The force on the conical point (cone) required to penetrate the soil is measured by electrical methods, at a minimum of every 50 mm of penetration. Stress is calculated by dividing the measured force (total cone force) by the cone base area to obtain cone resistance. A friction sleeve is present on the penetrometer immediately behind the cone tip, and the force exerted on the friction sleeve is measured by electrical methods at a minimum of every 50 mm of penetration. Stress is calculated by dividing the measured force by the surface area of the friction sleeve to determinate friction sleeve resistance.

The pocket penetrometer after Neuschäfer (CT-421) described in the *ATV-Arbeitsbericht (1989)* [23] has a diameter of ca. 2 cm, a length of ca. 18 cm and a weight of ca. 200 g. Manually a force will be put via a spring on the sample. The penetration resistance can be read directly from the scale. For an exact determination several cones (adapters) according to the consistency of the investigated materials exist. The adapters have a relation diameter to height of 1:1. For determination of consistency three penetrations in the same sample are necessary. Then the penetrometer bearing is determined by a nomogram; the number of the cone size and the indicated value from scale are the input values. The penetration bearing is converted in this case by empirical regression line into a vane shearing strength.

The pocket cylinder penetrometer (Geotester) is originally capable for measuring the unconfined compression strength (cp. *ASTM D2166* and *ASTM D5102*), angle of internal friction $\phi$ and the cohesion C.

The sample is loaded in compression to failure: The force required for shearing the material is measured, it could be read directly from the scale. Stress is calculated by dividing the measured force by the surface area of the cylinder (plunger) to determinate penetration resistance. Like the penetrometer after Neuschäfer there do exist several cylinders of different sizes according to the consistency of the materials.

#### **3.2.3.4** Material to be examined

The tests are mainly employed for the following materials

- Thermal insulating cements
- Hydraulic cement (mortar)
- Building plasters
- Refractory mortars
- Soil material (soil and soft rocks)
- Heterogeneous propellants (gel and emulsion type)
- Lubricating greases and petroleum

- Petrolatum
- Petroleum waxes
- Bitumen and bituminous binders
- Semi-solid and solid bituminous material
- Hot applied joint sealants
- Sewage sludges
- Waterwork sludges

**3.2.3.5** Feasibility of the methods to the materials of investigation

The penetrometers and Vicat apparatuses designed to test the consistency can be used to test the solidity of substances like concentrated sludge, sludge at higher dry content or other materials as they may assume a paste-like or solid consistency. The Penetration tester and the CBR method are not as suitable as the methods described before. The apparatus are specialised for the investigated materials like cement and soil respectively.

The cone and friction-cone penetration tests described in *ASTM D3441* and *ASTM D5778* provide a good test method for determining the bearing capacity of soils. Most of the friction-cone apparatuses request electrical power and are due to their sizes not easy to handle. Therefore these test methods won't be further investigated.

## 3.3 Thixotropic behaviour of solid materials

For the determination of the thixotropic behaviour of solid materials a standard method does not exist. From there it should be investigated a combination of methods for determination of the solidity like penetration, etc. and an energy-input in terms of "flow" apparatus to simulate the shear stress.

### 3.3.1 Laboratory or field test feasibility

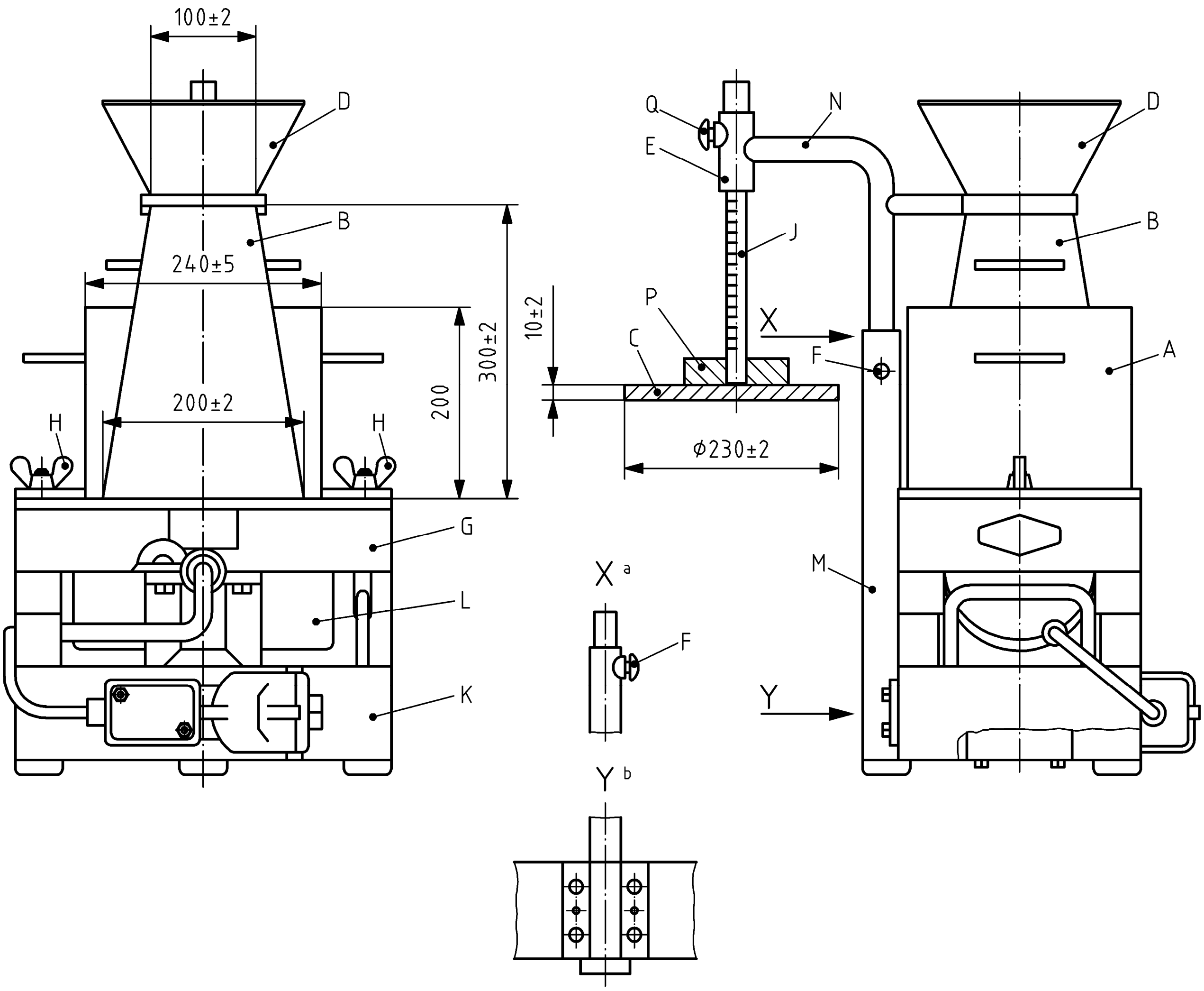
Due to the fact that many methods require large space and electrical power, the test methods have to be generally simulated in the laboratory. The energy-input respectively the vibration of the described apparatus mimics the transport conditions. For simple field test methods could be created a small-sized robust design of the laboratory method, which is driven electrically, if electric power is available, or manually by a hammer e.g.

### 3.3.2 Apparatus

The tests examined use the following types of apparatus:

- Vebe meter (Consistometer) (Figure 30)
- Flow table (Figure 31, Figure 32)
- Vibrating table (Figure 33)
- Vibrating hammer (Figure 34)

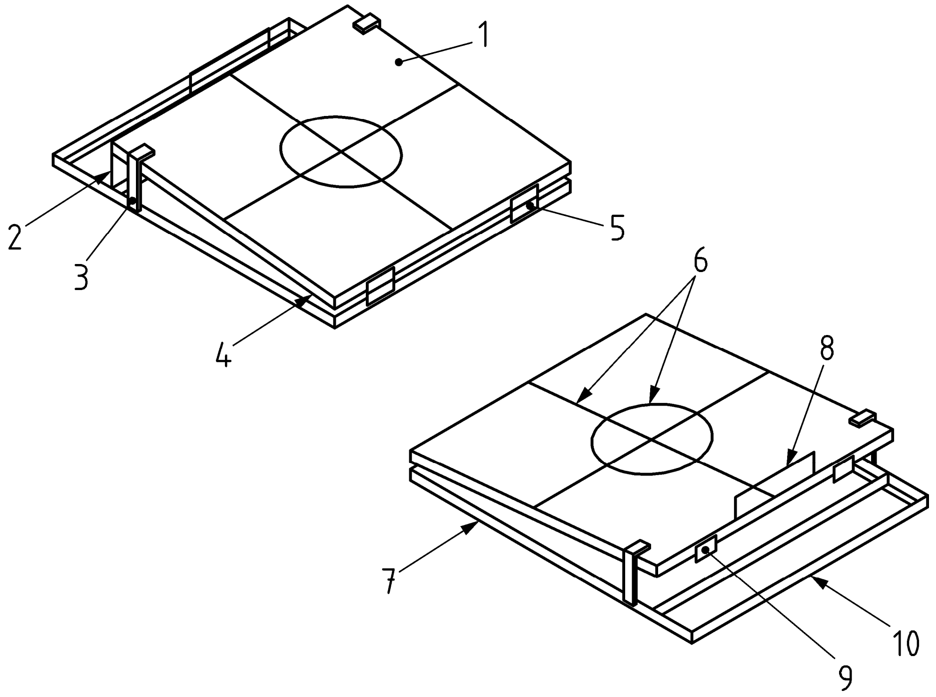
Dimensions in millimetres




**Key**

A container
B mould
C disc
D funnel
E sleeve
F screw
G vibrating table
H wing nuts
J scale
K hollow base
L vibrator unit
M holder
N swivel arm
P weight
Q screw

**Figure 30 — Consistometer (Vebe meter) [42]**

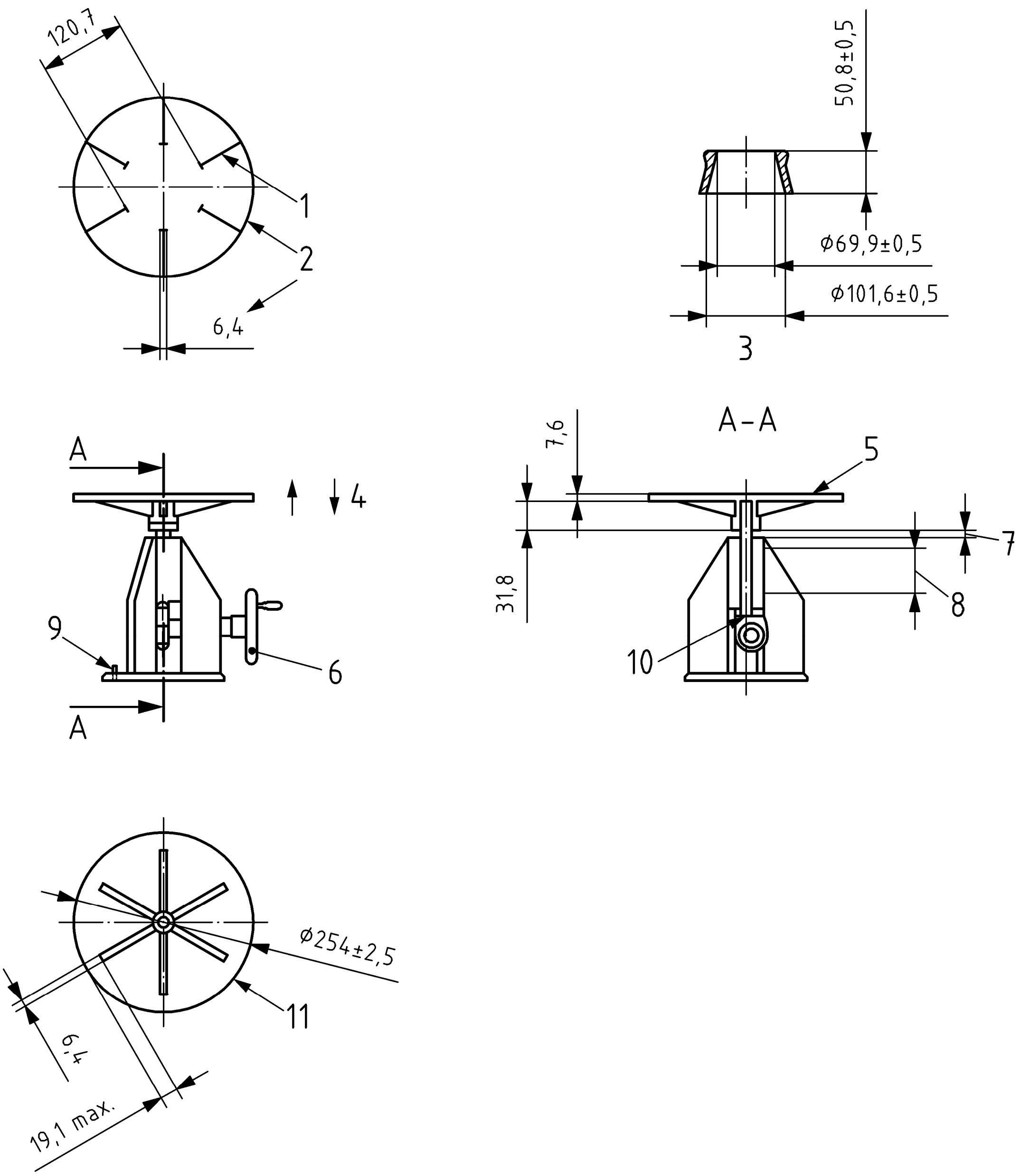



**Key**

1 metal plate
2 travel limited to 40 ± 1
3 upper stop
4 table top
5 external hinges
6 markings
7 base frame
8 lifting handle
9 lower stop
10 toe board

**Figure 31 — Flow table [43]**

Dimensions in millimetres

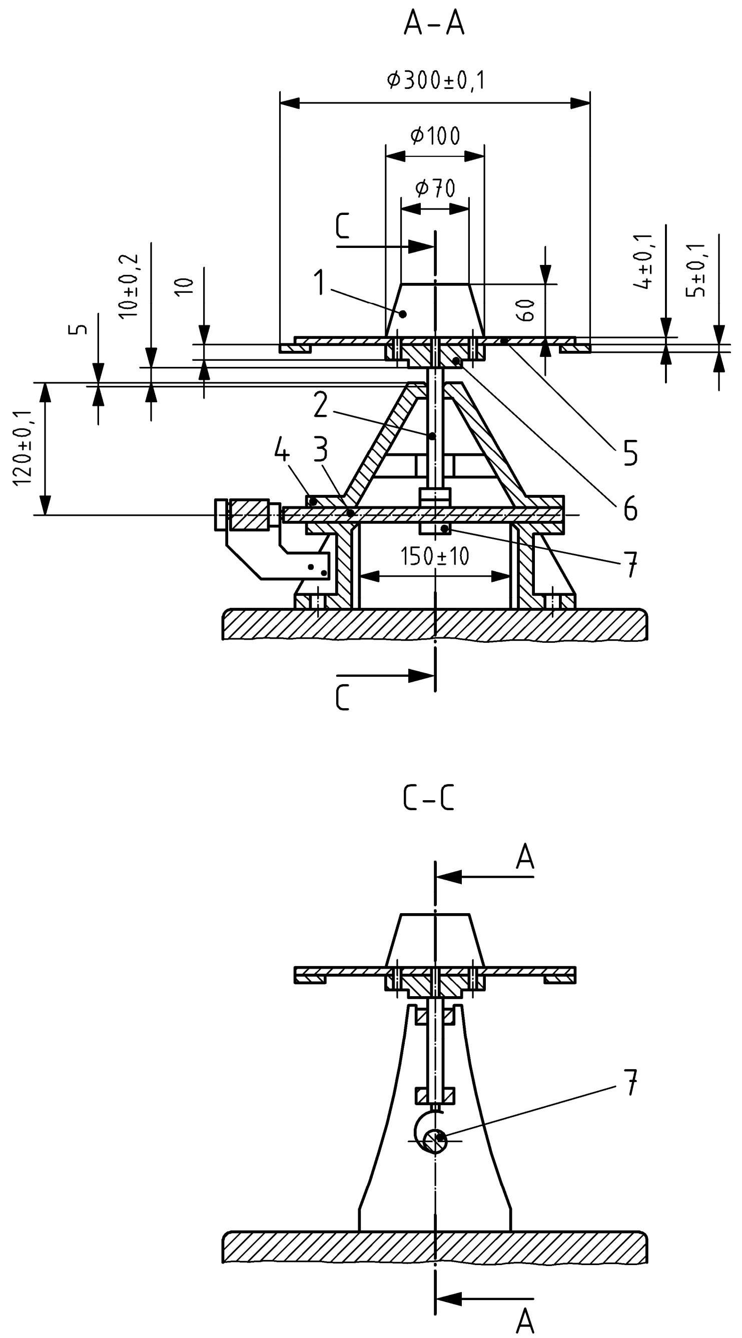



**Key**

1 lines to be scribed on face of table
2 table (brass or bronze)
3 conical mould
4 movement of flow table: up and down
5 table to be fixed to shaft and total mass to be 4,1 ± 0,5 kg
6 handle, or alternatively, motor drive through flexible shaft
7 drop
8 machined to give a drop of 12,7 ± 0,1
9 fixing holes
10 hardened contact faces
11 view on underside of table

**Figure 32 — Flow table and conical mould [44]**

Dimensions in millimetres

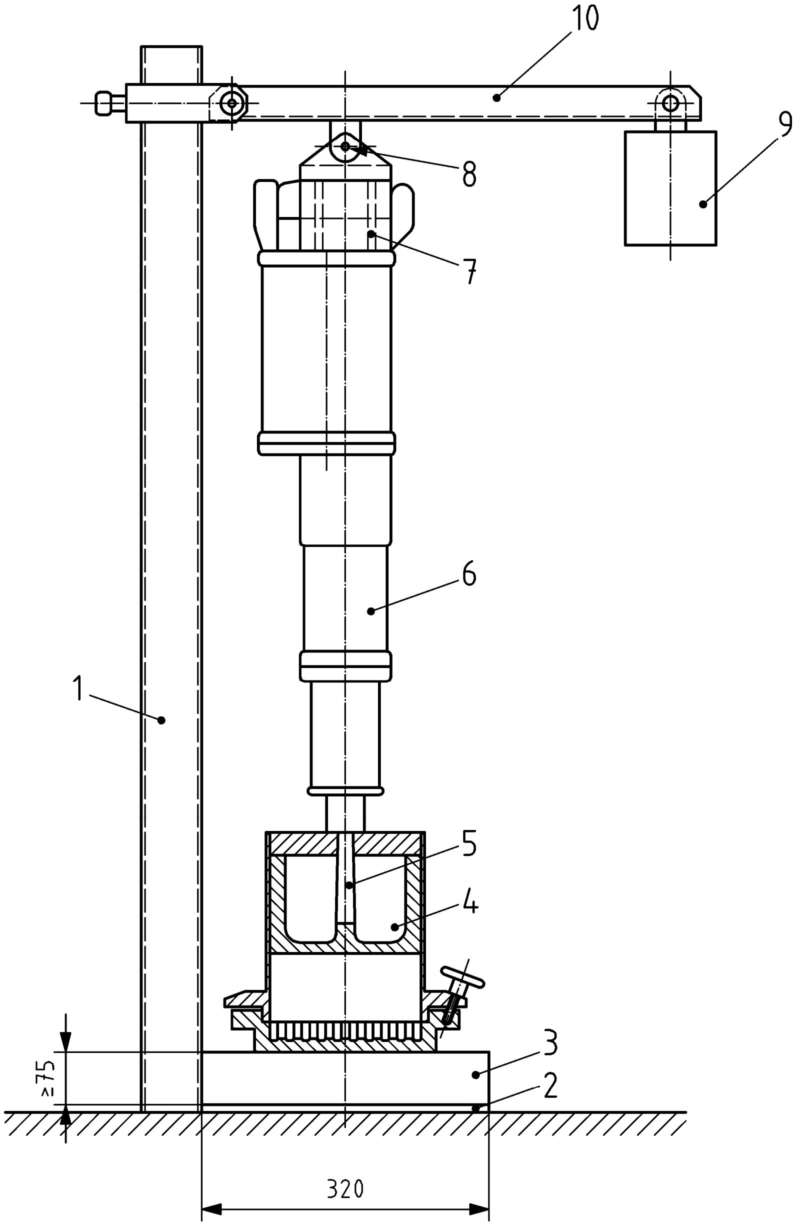



**Key**

1 truncated conical mould
2 disc
3 rigid table plate
4 lifting carn
5 stand
6 horizontal shaft
7 lifting spindle

**Figure 33 — Vibrating table [45]**

Dimensions in millimetres




**Key**

1 support frame
2 rubber mat
3 steel base, minimum area 0,1 m³
4 compaction mould and anvil
5 shank
6 vibrating hammer
7 hammer grip assembly
8 securing pin to allow hammer to hang vertically
9 surcharge mass
10 lever arm

**Figure 34 — Compaction rig assembly with vibrating hammer [46]**

### 3.3.3 What is measured and how

For testing fresh concrete with the Vebe meter described in *EN 12350-3* the time taken for the surface of a disc to be fully in contact with the grout is measured (Vebe time). This time should be in the range of 5 s to 30 s. For determination of the degree of compactibility described in *EN 12350-4* the concrete is compacted by vibration. The distance from the surface of the compacted concrete to the upper edge of the container is used to determine the degree of compactibility. For determination of the consistency of concrete with the aid of a flow table described in *EN 12350-5* the spread of concrete (diameter) on a flat plate, which is subjected to jolting, is measured. In a similar way the standard *EN 13395-1* operates with mortars (cp. also *EN 1015-3*).

For the determination of the relationship between the dry density and water content of unbound and hydraulically bound mixtures samples are compacted into a CBR-type mould using an electrical powered vibrating hammer described in *EN 13286-4* over a range of water contents. The range includes the optimum water content at which the maximum dry density for the specified degree of compaction is obtained. Another apparatus for compaction is a Vibrating table described in *EN 13286-5:* The mixture is compacted in a mould by means of a load on the top of the mixture. Then the laboratory dry density and corresponding water content are determined.

For the method described in *EN 1168* the diameter of the water-building plaster-mixture is measured after it has been spread by operation (several hubs) of the table.

In the method described in *ISO 13765-2* the consistency of a refractory mortar is assessed by the increase of the diameter of a sample, when it is subjected to mechanical agitation using a reciprocating flow table.

#### 3.3.4 Material to be examined

The tests are mainly employed for the following materials

— Fresh concrete

— Hydraulic cement mortars

— Unbound and bound mixtures

— Building plasters

— Refractory mortars

#### 3.3.5 Feasibility of the methods to the materials of investigation

For the feasibility of methods concerning the determination of the thixotropic behaviour the combination of penetration tests and energy input is of importance. This input can be applied electrically by vibrating apparatus or manually by hubs or knocks. For the investigations all energy-input-apparatus are suitable. To simulate transport conditions the vibrating apparatuses are well appropriate, whereby especially the external apparatuses like vibrating table are rather suitable than the internal apparatuses like vibrating hammer. Also the handling with the test samples is important (preparation, size of moulds, etc.). These items have to be determined in further investigations.

### 3.4 Piling behaviour

— Slump test apparatus

— Compacting apparatus (e.g. Oedometer)

— CPB- Cubic piling box (after E. Pasqualini [47])

— "Turned Box"

#### 3.4.1 Laboratory or field test feasibility

These test methods are mostly performed in the laboratory, because they require a lot of preparation. Special field test methods for determination of the piling behaviour don't exist so far. But for some apparatuses it is possible to create for simple field tests a small-sized robust design of the laboratory apparatus according to the analysed material.

### 3.4.2 Apparatus

The tests examined use the following types of apparatus:

— Moulds, if needed: laboratory tools (results: Figure 35)

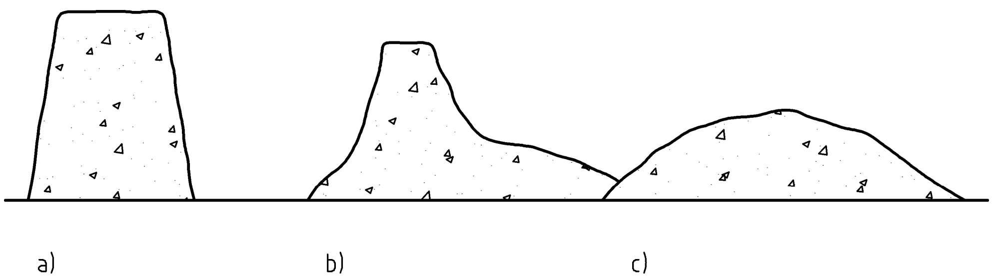
— Compacting apparatus (e.g. Oedometer) (Figures 36 - 38)

— Plastic or metallic tube, flat plate

Besides these apparatuses another types of apparatuses, which are not reported in the standards, but may be considered as a promising tool to test the piling behaviour of the analysed materials, are the following apparatuses:

— Cubic Piling Box (CPB) (Figure 39, Figure 40)

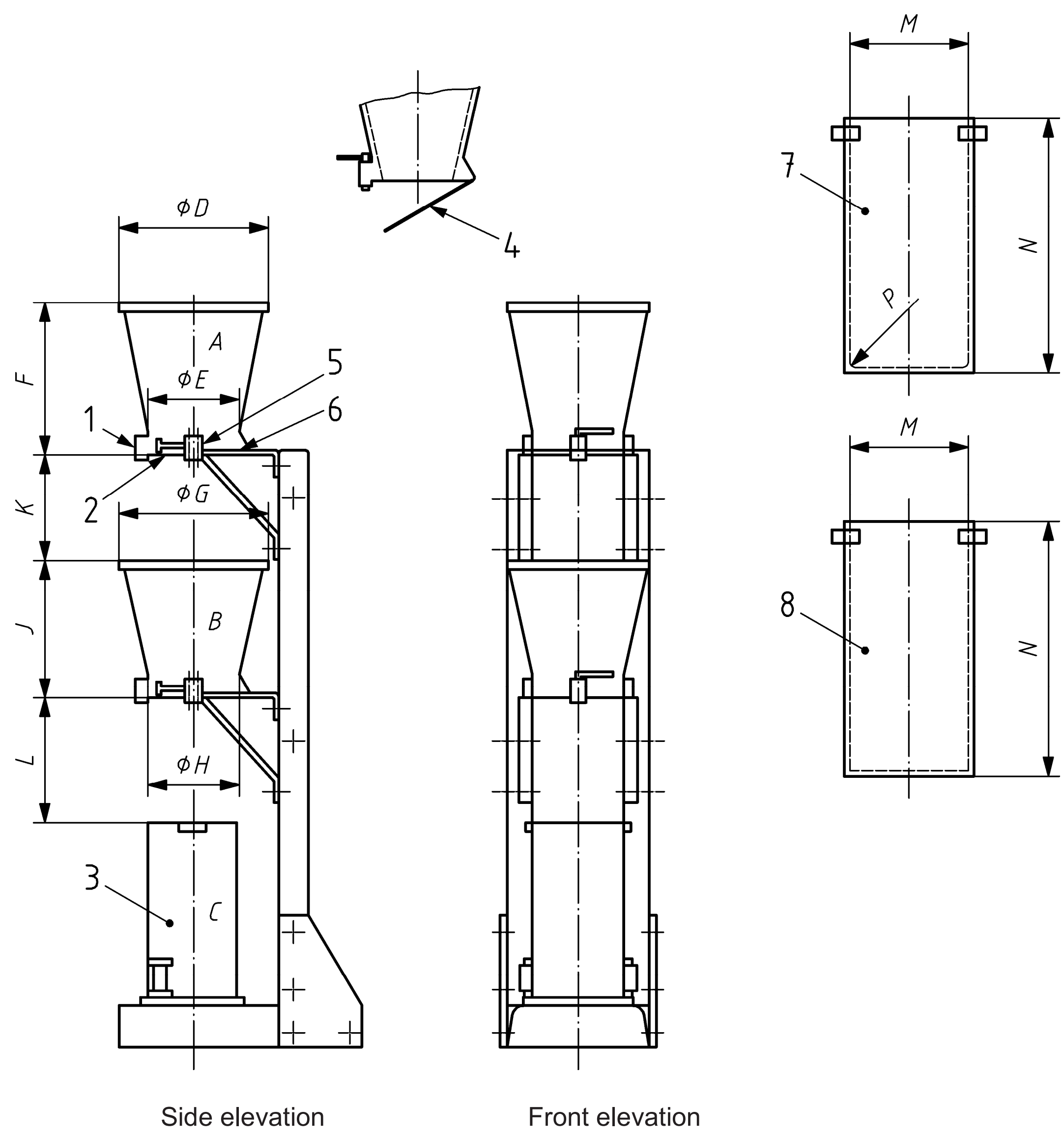
— "Turned Box"




**Key**

a) true slump
b shear slump
c) collapse slump

**Figure 35 — Forms of slump [48]**

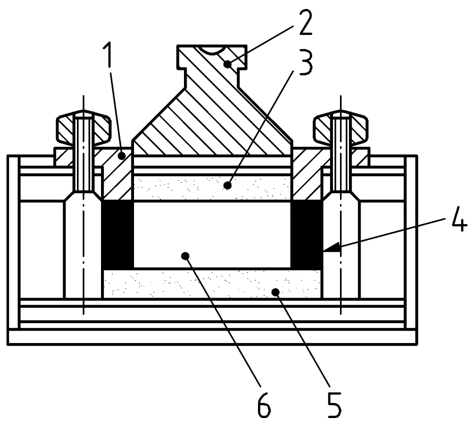



**Key**

1 catch
2 trap door
3 cylinder clamp each side at diametrically opposite corners
4 view of trap door partly open
5 hopper clamp
6 hinge
7 cylinder C – preferred apparatus
8 cylinder C – alternative apparatus

**Figure 36 — Compacting factor apparatus [49]**

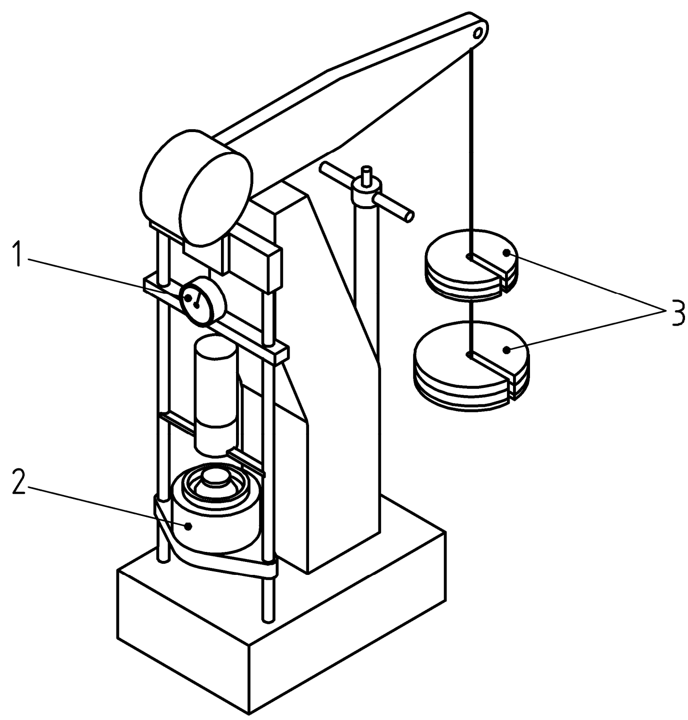
A special form of compacting apparatus is the Oedometer




**Key**

1 cap
2 load
3 porous disk
4 rigid mould
5 porous disk
6 sample

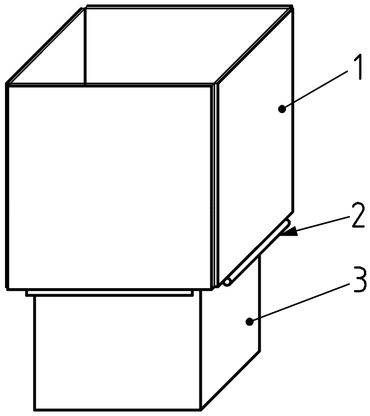
**Figure 37 — Oedometer [47]**




**Key**

1 micrometer
2 oedometer
3 load

**Figure 38 — Device for loading [47]**




**Key**

1 sidewall
2 hinge
3 base

**Figure 39 — Cubic piling box at start of test (closed sidewalls) [47]**

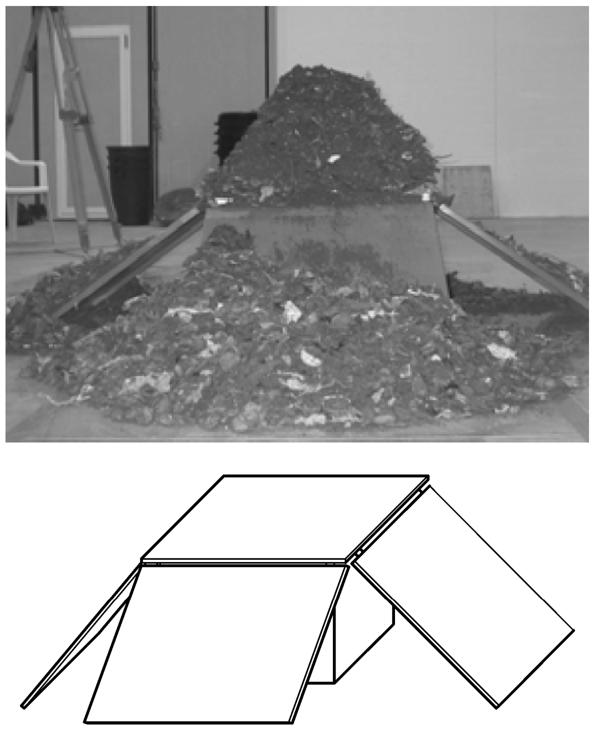


**Figure 40 — Cubic piling Box at the end of test (opened sidewalls) [47]**

### 3.4.3 What is measured and how

For the determination of the slump of hydraulic cement and concrete the sample is placed and compacted in a mould. The mould is raised, and the concrete allowed subsiding. The vertical distance between the original and displaced position of the centre of the top surface of the concrete is measured and reported as the slump of the concrete.

For the determination of the Compacting factor within 150 s of placing the sample in the upper hopper from the compacting apparatus the cylinder and its contents are weighted. After that, the concrete in the upper hopper is mixed with concrete of the same sample, place in a cylinder in six layers and compact each layer by using either the compacting bar or the vibrator (hammer); weight the cylinder again. Stroking with the compacting bar or use Vibrating hammer/ table. Calculate the ratio of the mass of partially compacted concrete dividing by the mass of fully compacted concrete.

Another variant to determine the slump is following method: The sample (200 $cm^3$) is filled in a tube placed on a table with several circles, then the tube is lifted vertically; after 30 s read the number of the circle reached by the spreading matrix. This test method needs a special flow table with some circles; due to the spreading the slump is determined “horizontally”, not “vertically” like the other slump test methods.

The Oedometer test method covers procedures for determining the magnitude and rate of consolidation of soils when it is restrained laterally and drained axially while subjected to incrementally applied controlled stress loading. According to the standard procedure, the test is performed with constant load increment duration of 24 h. The specific loading schedule will depend on the purpose of the test. Each stress increment is maintained until excess pore water pressure is completely dissipated. During the consolidation process, measurements are made of change in the sample height and these data are used to determine the relationship between the effective stress and void ratio or strain, and the rate at which consolidation can occur by evaluating the coefficient of consolidation. The data from the consolidation test are used to estimate the magnitude and the rate of settlement of structures and embankments.

The test method with the aid of the Cubic Piling Box (CPB) can be used for determining the piling angle (angle of rest) of pieces. The particular material is placed into the box and, if necessary, it can be compacted. Then the sidewalls are suddenly overturned and the stable final configuration is surveyed. The natural slope angle of the remaining material can be measured on each side of the sample remaining on the box base.

### 3.4.4 Material to be examined

The tests are mainly employed for the following materials

- Hydraulic cement (concrete)
- Concrete
- Glass-fibre reinforced cement
- (Fine-grained) soils
- (Solid) waste

### 3.4.5 Feasibility of the methods to the materials of investigation

The standardised methods like slump tests require a certain threshold of consistency of the materials of interest. For determination of the piling behaviour new methods were developed, for example the "Turned Box" and the "Cubic Piling Box", which were created in France and Italy [47] respectively.

As far as piling behaviour is concerned, the Oedometer test can be used to characterize the compressibility of the material and to have an indication of how much a stratum of piled material can settle due to the loading of the material placed above. For piling behaviour, the selection of the loading schedule (maximum value, load increments and the duration of each increment) will be defined on the basis of the total height of piling and of the piling sequence. In particular, the maximum load will be necessarily lower than that used according to the standard for soils. The size of the mould that contains the sample can vary on the basis of the maximum grain size of the material to be tested.

The CPB test [47] can be easily performed on particular pieces like dewatered sludges, waste and soils, etc. as well as on cohesive materials e.g. compacted materials. As far as piling behaviour is concerned, the CPB can be used to immediately and easily assess the natural slope angle of a material.

# 4 Critical point and recommendations

In this section mainly the methods, which are considered for further investigation, are discussed.

Standardisation procedures for material examination will consist of

- Sampling, transport, preservation, storage
- Pre-treatment
- Measurement and evaluation of results.

## 4.1 Flowability

### 4.1.1 Comparison (discussion: pro/contra)

Flow cone apparatus (existing method/parameter)

Water and wastewater sludges, treated bio-waste and soil can have a rheological behaviour similar to Newtonian fluids, non reference number of rheological properties and time flow are suggested or recommended, this simple measurement apparatus allows to have information useful in sludge, soil, mixture of cement and soil handling and managing;

Pro: flow cone is a simple, not expensive and easy to use apparatus, it does not require electrical power supply and specialized personnel for its use.

Contra: The *flow cone apparatus* is only suitable for Newtonian fluids like soil. The use for other materials of commercial apparatus or the best geometrical dimension must be investigated in further experimental work. Sludge and treated bio-waste can contain coarse particles that can occlude the discharge orifice, so a sieving pre-treatment is necessary. The flow cone apparatus has been already modified to absolve this item for using it for soil but it must be verified for sludge and treated bio-waste.

Vicat and Magnesium Cone penetrometers (existing method/parameter)

The test measures the consistency in samples at high solid concentration, the measurement is used to characterize sewage and water works sludges.

Pro: Magnesium cone penetrometer is a simple apparatus easy to use suitable for testing in both laboratory and field, the measure is very fast (5 s) and the yield stress obtained can be compared with results of reference laboratory method.

Contra: *”penetration cone*”, Vicat apparatus (*ASTMC 472-84; ASTMC 807-99*) measures only the sample deformation. An absolute value of yield stress is obtained with magnesium penetrometer (*ASTMD 2884-93*); for field application a stable plane is necessary. The apparatus is not standardised, by measuring the height of the sludge suspension remaining in the container at the end of continuous flow, the yield stress can be calculated.

Pro: the apparatus is simple and offers a direct measure of yield stress; it constitutes a field apparatus easy to use; previous investigation of its employment for mineral sludges [64] showed a good correlation with laboratory results of yield stress up to 40 Pa. Further experimental work is needed to clarify the suitability.

Contra: A pre-treatment of sample is required almost using calibrated pipes at lower diameters; the pipe diameter and time required to execute the test must be optimized.

Inclined plane

The method is not standardized and can be used to calculate the yield stress of visco-plastic fluids by determining the depth of the sludge remaining at the rest on the inclined channel.

Pro: it is possible to measure the yield stress of coarse suspension without any pre-treatment of the sample.

Contra: the apparatus has been tested on synthetic suspension (clay-water) in a narrow yield stress range (from 35 Pa to 90 Pa).

Modified slump apparatus

This is a non-standardised method for measuring the yield stress of a pasty sludge at different solid concentration by determining the height loss of a cylindrical sample left flowing on a horizontal plane.

Pro: the slump test has the advantage to involve a small volume of material and it is not influenced by the presence of coarse particles.

Contra: the sludge flow using clay-suspensions could be not so well controlled as inclined plane flow or extrusion tube viscometer from which it is in principle possible to calculate the exact value of yield stress.

Rotational viscometer

Rotational viscometers are commonly used to measure the absolute viscosity of liquids of different physical and chemical characteristics in a wide range of viscosity values.

Pro: *"rotational viscometer"* (existing method/parameter)

The apparatus can be used for every flow curve of the fluid, or time depending fluids to determine the absolute viscosity and rheological properties.

Contra: (existing method/parameter)

The narrow gap between rotational and fixed cylinders, concentration gradient of samples and the high investment cost limit the use of this apparatus only for a laboratory use. In addition the shear rate is highly dependant of velocity profile inside the gap and consequently, the data given by the rheometer are false (the sheared thickness of the material is smaller than expected).

All apparatus used

Contra: The fluid which gradually changes its physical state from liquid to gel cannot be measured with methods proposed; a simple apparatus energy spending must be employed together with the selected methods.

### 4.1.2 Recommendations

#### **4.1.2.1** Laboratory reference

Laboratory test (Method 1): Rotational viscometer apparatus must be used

Controlled shear rate rotational viscometers are laboratory devices for determining absolute viscosity of Newtonian and non-Newtonian substances in a wide range of viscosity values. They can be used to test the viscosity of sludges or to correlate rheological properties with field test results. In order to avoid the problems connected with unsheared regions a comparison between rotational and torque viscometers will be performed.

Recommendations: pre-treatment of samples for cell narrow gaps, temperature control.

#### **4.1.2.2** Field test

Field Test (Method 1): *Flow cone apparatus*

Viscosity is related to the time for a determined volume of sample to empty the cone by flowing under gravity through a calibrated discharge tube.

Recommendations: pre-treatment of samples, optimization of geometric dimensions and devices.

Field Test (Method 2): *Magnesium penetration cone*

Magnesium penetration cone method, as reported in *ASTM D2884*, allows the yield stress to be quantitatively determined.

Recommendation: a stable stand, free of vibration must be supplied.

Field test (Method 3): *Extrusion tube viscometer (Kasumeter)*

The use of an extrusion tube viscometer is a promising method for field determination of the yield stress of high concentrated slurries. The method is not standardised. The apparatus consists of a calibrated pipe 200 mm long of different diameters placed at the bottom of a cylindrical vessel filled with the sample. Measuring the time of flow until it is continuous and the height of the suspension remaining in the cylinder at the end of the continuous flow, the yield stress can be calculated. This method allows determining the quantitative value of the yield stress.

Recommendations: optimization of geometric dimensions of apparatus and devices, pre-treatment especially for materials with gel-sol or sol-gel transformation.

Field test (Method 4): *Inclined plane*

A cheap and simple method for field determination of the yield stress of concentrated suspensions. The method is not standardised. Applicable to coarse suspensions without pre-treatment of the sample.

Recommendations: optimisation of the dimensions of the channel and of the operative conditions (slope, quantity of sample, time of flowing, etc.)

Field test (Method 5): *Modified slump test*

The modified slump test is a practical non-standardised method based on a theoretical approach to estimate the fluid yield stress from its slump.

Recommendations: the application of this test to the materials of investigation shall be accurately evaluated especially when analysing thixotropic fluids.

## 4.2 Solidity

### 4.2.1 Comparison (discussion: pro/contra)

Laboratory vane shear apparatus

In general the "Vane shear strength" is one of the most commonly used parameter in the field of soil mechanics. There are a lot of standards available. In Germany e.g. also exists such a parameter in the field of waste disposal / landfilling: For landfilling of wastes the *TA Siedlungsabfall (1993)* [14] gives a minimum value for the solidity of 25 kN/m². The vane shear strength of sewage sludge depends in some extent on the dry solid content, but there is no good correlation for different sludges. For the laboratory method there are two similar apparatus described in *ASTM D4648* and *ATV-Arbeitsbericht* [34, 23].

Contra: For reliable measurements of the vane shear strength the investigated materials have to be cohesive. Therefore the vane testing apparatus can't be applied for non-cohesive materials like coarse-grained soils.

Pro: Because of reliable results of investigations with sewage and waterworks sludge, which were performed with the laboratory vane shear apparatus (*ATV-Arbeitsbericht* and *Wichmann et al.* [23, 15]), this method should be preferred as the laboratory reference method. The calculation of the shear strength - after sample preparation and measurement - consists of the following equations after *Zweck 1969* [50]):

$$\tau_v = \frac{M}{K} \qquad [\mathrm{kN/m^2}] \tag{4}$$

where

$M$: max. torque in [kN m],

$$K: \quad \frac{\pi}{2} \cdot d^2 \cdot h \cdot \left(1 + \frac{d}{3h}\right) \qquad [\mathrm{m^3}] \tag{5}$$

where

$d$: diameter of the sheared cylinder = width of the vane,

$h$: height of the vane.

The shear strength $\tau_v$ is equal to the cohesion $c_u$ of the undrained soil by (fast) shearing. For h = d:´

$$\tau_v = \frac{3 \cdot M}{2 \cdot \pi \cdot d^3} \tag{6}$$

For a constant shear rate the vane should be driven electrically e.g. with a velocity of 10 degree per minute, because the measured shear strength is influenced by the shear rate. The characteristics of this apparatus are [16]:

— length of time: ca. 60 min (for 3 measurements)

— good handling (motorised test procedure)

— reproducibility of test values (average ± 10 %)

— measuring range: 1- 180 kN/m$^2$.

Another investigated vane testing apparatus - pocket vane shear apparatus - with several vanes of different sizes was not as suitable as other methods for determining the shear strength of materials with lower dry solids content. This aspect was pointed out by investigations performed with waterwork sludges (*Wichmann et al.* [15]).

Penetrometer

The described apparatuses are suitable to obtain reliable results of the measurements in both laboratory and field.

Contra: The procedures with penetrometers are time-dependent. The measurement has to be carrying out within a fixed duration of e.g. 5 s. This aspect affords a precise test execution.

Pro: The advantage is the great variation of the penetrometer tip according to the investigated material. There is a range of different tips in form of a plunger, a cone or a needle available. That is why several methods are considered for further investigations. Explicit determinations, what kind of tip for a special material is the best, have to be investigated in further experiments. The procedure of calculation is quite simple: According to *Hansbo (1957)* [54] the sludge cohesion (undrained shear strength $c_u$) can be derived from the cone penetration depth using the empirical equation:

$$c_{\mathrm{u}} = \frac{K \cdot m \cdot g}{i^2} \cdot \left(1 + \frac{h}{i}\right) \tag{7}$$

where :

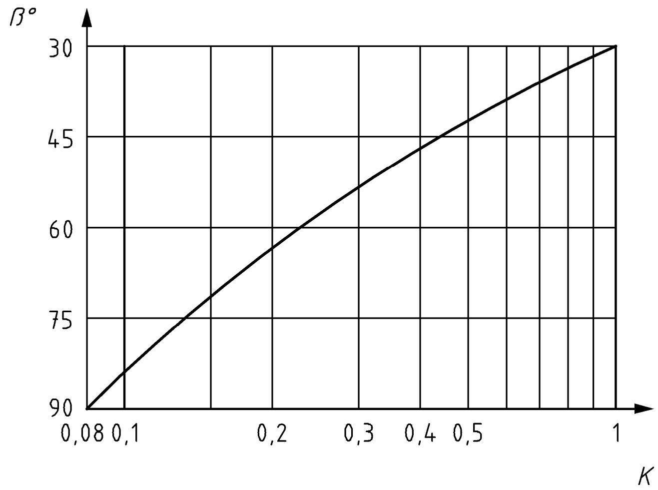
m: mass of the cone,

g: acceleration of gravity,

i: depth of penetration (indentation),

h: distance between cone apex and soil surface when the cone is dropped (normally h = 0),

K: function of the apex angle of the cone (Figure 41).




**Key**

$\beta^\circ$ apex angle
$K$ parameter

**Figure 41 — Relation between the parameter K and the apex angle $\beta$ of the cone**

Further advantages are [16]:

— length of time: ca. 5 min (for 3 measurements)

— very good handling

— reproducibility of test values (average ± 10 %)

— costs: ca. 40 % of the Laboratory Vane Shear Apparatus

Vicat apparatus

The Vicat apparatus is a special kind of a penetration apparatus and has a tip in terms of a needle. In particular the beginning and end of solidification is measured in order to determine the shear strength. This aspect has to be developed in further investigations; it is one part of research needs in the field of determining solidity.

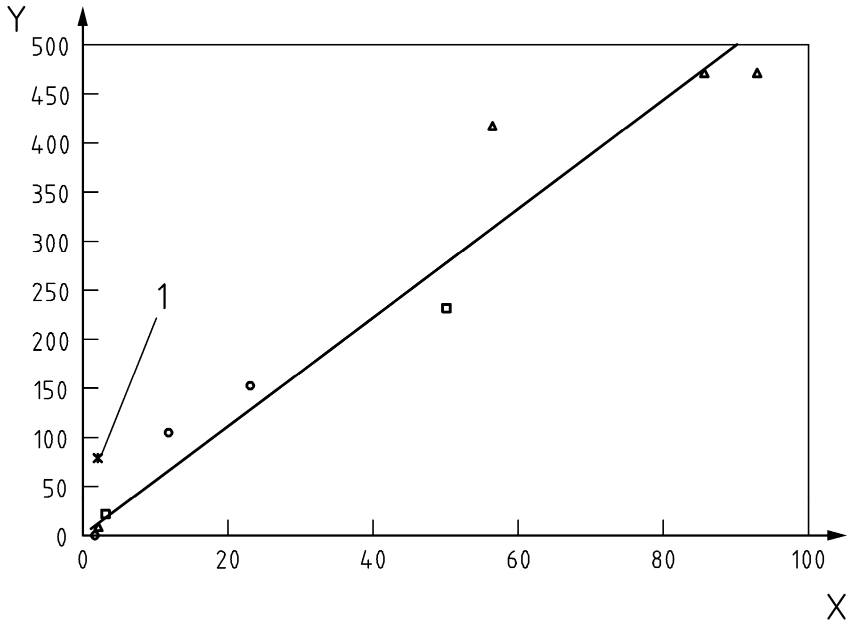
Contra: Because of the shape of the penetration tip this apparatus could be only suitable for solid materials like dewatered sludges, particular bio-waste and soil. In further investigations this assumption has to be confirmed.

Pro: The Vicat apparatus is a special penetration apparatus, which operates time-independent. For the measuring procedure there is a fixed depth, which the needle has to be reached. For determining the consistency of materials this aspect could be much more comfortable during the measurements.

Pocket penetrometer

Pro: A promising tool for simple field tests – determination of the penetrometer bearing capacity $p_P$ – is the pocket penetrometer after Neuschäfer. After sample preparation and performance the value of penetration resistance can be read directly on the scale of the penetrometer. The penetrometer bearing capacity is determined with the aid of a nomogram, whereby the average of the values, which are indicated by the scale within three measurements, and the factor of the cone tip (see Figure 28) are the input values. Furthermore the obtained value can be converted to the parameter "vane shear strength $\tau_V$" by a regression line e.g. for waterworks sludges (Figure 42). In general the equation for the regression line [23] is determined as

$$\tau_V = (-0{,}56) + 0{,}156 \cdot p_P \quad (8)$$




**Key**

X Laboratory vane shear strength [kN/m²]
Y Penetrometer bearing capacity [kN/m²]
1 Not taken into account

o Al-Floccolation sludge
▲ Fe-Floccolation sludge
□ Iron sludge
✖ Lime sludge

**Figure 42 — Comparison penetrometer bearing capacity vs. laboratory vane shear strength [16]**

Further advantages of this method are [16]:

— consisting of several cone tips for a wide range of materials

— length of time: ca. 5 min (for 3 measurements)

— good handling

— reproducibility of test values (average ± 8 %)

— measuring range: 0 - ca. 90 kN/m$^2$ (Vane Shear Strength)

— low costs: ca. 8 % of the Laboratory Vane Shear Apparatus.

Pocket cylinder penetrometer

Contra: Only a few investigations were carried out in the past. For certain conclusions further investigations will be necessary.

Pro: However, this penetrometer can be also a capable apparatus for field tests, in this case for measuring the unconfined compression strength $q_u$. The *TA Siedlungsabfall (1993)* [14] gives for landfilling of wastes a minimum value for the unconfined compression strength of 50 kN/m² with an axial deformation of max. 20%. It could be a helpful tool for the choice of the best suitable instrument, that there do exist such default values. The unconfined compression strength is related to the undrained shear strength $c_u$ in terms of Eq. 3 (compare 1.5.2):

$$q_u = 2 \cdot c_u$$

Further characteristics are [16]:

— length of time: ca. 5 min (for 3 measurements)

— very good handling

— reproducibility of test values (average ± 8 %)

— low costs: ca. 6 % of the Laboratory Vane Shear Apparatus

### 4.2.2 Recommendations

For the determination of the shear strength it is recommended that the triaxial test should be the absolute laboratory reference method (e.g. after *DIN 18137-2* [51]).

**4.2.2.1** Laboratory reference method

Method 1: Laboratory vane shear apparatus (*ATV-Arbeitsbericht 1989*)

Method 2: Penetrometer (cone or needle)

Method 3: Vicat apparatus

**4.2.2.2** Field test

Method 1: Pocket penetrometer after Neuschäfer

Method 2: Pocket cylinder penetrometer

## 4.3 Thixotropic behaviour of solid materials

### 4.3.1 Comparison (discussion: pro/contra)

For standardisation of the thixotropic behaviour of solid materials a combination of standardised methods will be determined. Because of the experiences of the experts committee the combination "Penetrometer + energy-input" will be preferred. The advantage of penetrometers was already explicated in 4.2.1. The energy input can be applied electrically by vibrating apparatus or manually by hubs or knocks. In the following only the apparatuses for the energy-input are described:

Contra: Vibrating hammer

The vibrating hammer is an internal apparatus. During the measurement of the thixotropic behaviour this instrument could influence the procedure and therefore the results would be not certain.

Pro: Vibrating table

To simulate transport conditions the vibrating apparatuses, especially external apparatuses, are rather suitable than internal apparatuses or hand-made methods. For laboratory reference the apparatus can be more exact and thus more complex than a simple field method.

For simple field test methods could be created a small-sized robust design of the laboratory method, which is driven electrically, if electric power is available, or manually by a hammer e.g.

#### 4.3.2 Recommendations

**4.3.2.1** Laboratory reference method

Method 1: Penetrometers + energy-input (e.g. vibrating table)

**4.3.2.2** Field test

Method 1: Penetrometers + energy-input (e.g. vibrating table: small-sized apparatus, which should be easy to operate)

Method 2: Penetrometers + energy input (e.g. by a hammer, if electric power is not available; adequate mould)

### 4.4 Piling behaviour

#### 4.4.1 Comparison (discussion: pro/contra)

Oedometer

The strain condition of a material due to piling are different from those in the Oedometer, where lateral strain are prevented. Nevertheless, if the material is piled in lifts that are large enough in comparison with their height (three times at least), oedometic strains can be considered as representative. The settlement of the lower portion of the material can be evaluated.

The minimum sample diameter and height should be 20 cm in order to test a representative volume of material. To avoid or reduce the friction at the interface between the material and the ring as negligible a film of grease should be placed on the internal wall of the mould before placing the sample. A compaction mould for Proctor compaction (ASTM D698-91 [52]) can be used as well.

Pro: Important advantages of this method are that the procedure is well known – it is a standard in France and USA and an overall method in practise in many other countries - and it is easy to handle. From there this method should be preferred as an additional laboratory reference method.

Cubic Piling Box (CPB)

The Cubic Piling Box should be preferable to the French method (cp. Annex 1, No. 256 in [60] and [61]) because the friction between the sidewalls and the material to be tested does not affect the final result in terms of slope angle. The influence of the CPB dimension on the slope angle should be negligible provided that the side of the box is much greater of the maximum grain size of the sample. This method was originally proposed for solid waste materials having a maximum grain size of about 5 cm (Pasqualini et al., 2003 [47]). In that case, the sides of the cubic box were equal to 60 cm. For other materials a minimum of 30 cm x 30 cm should be considered in order to test a representative volume of material. The dimension of the cubic box should be at least 5 times greater than the maximum grain size of the sample to be tested. For detailed results of other materials further investigations are necessary.

Pro: Important advantages of this method are the very simple handling, the feasibility to both in the laboratory and in situ, no specialised personnel and the great variability due to the different sizes of the Cubic Piling Box related to the grain size of the material.

#### 4.4.2 Recommendation

**4.4.2.1** Laboratory reference method

Method 1: Cubic Piling box (CPB)

Method 2: Oedometer

**4.4.2.2** Field test

Method 1: Cubic Piling Box (CPB)

### 4.5 Summary of recommended methods

#### 4.5.1 Flowability

The methods selected for further investigation are reported in Table 8 and can be divided in two groups: laboratory and field methods.

**Table 8 — Summary of the recommended apparatuses for flowability**

| Method (Apparatus) | Employment |
|---|---|
| Shear controlled coaxial cylinders viscometer | Laboratory test |
| Flow cone | Field test |
| Penetration cone for yield stress measurement | Field test |
| Extrusion tube viscometer (Kasumeter) | Field test |
| Inclined plane | Field test |
| Modified slump test | Field test |

#### 4.5.2 Solidity, thixotropic behaviour and piling behaviour

The following Table 9 gives an overview of the recommended apparatuses and methods for determination of the solidity, thixotropic behaviour and piling behaviour.

**Table 9 — Summary of the recommended apparatuses for solidity, thixotropic behaviour and piling behaviour**

| Method (Apparatus) | Solidity | Thixotropic behaviour of solid materials | Piling behaviour |
|---|---|---|---|
| Penetrometer | Shear strength (Lab / field) | Shear strength (Lab*/field$^{+/**}$) | |
| Vicat apparatus | Shear strength (Lab) | | |
| Laboratory vane shear apparatus | Vane shear strength (Lab) | | |
| Pocket penetrometer (Neuschäfer) | Bearing capacity[a] (Field) | | |
| Pocket cylinder penetrometer | Unconfined compression strength[b] (Field) | | |
| Vibrating table | | Shear strength (Lab*/ field$^{+}$) | |
| Hammer (manual) | | Shear strength (Field$^{**}$) | |
| Cubic piling box (CPB) | | | Piling angle (Lab/field) |
| Oedometer | | | Compactibility (Lab) |

[a] Conversion into shear strength is possible

[b] Conversion into undrained shear strength is possible.

Combinations for thixotropic behaviour: *+*; $^{+}$+$^{+}$; $^{**}$+$^{**}$

## 4.6 Research needs

### 4.6.1 Basics of methods

Methods proposed will measure the flowability (yield stress, viscosity) of materials using apparatus already available, also if an optimization of methodology and apparatus must be performed to be adapted to the material of investigation. The field test results will be compared with laboratory ones used as reference.

The mechanical property "solidity" can be determined directly by the shear strength or by the penetration bearing capacity respectively the unconfined compression strength, which could be both converted into shear strength. The measure of the shear strength during an energy-input in terms of vibration should describe the “thixotropic behaviour”. For the “piling behaviour” it is recommended to measure the compactibility and/or piling angle.

The proposed methods for determining the parameters mentioned above are existing standards, methods used in practise or methods, which are still in a development stage. However, for improvement of the methodologies most of the procedures and used apparatuses have to be optimised by further examinations. For all parameters do exist methods for both laboratory and field, whereby the field test results will be compared with laboratory ones used as reference.

### 4.6.2 Applicability of methods to the materials of investigation

To apply the proposed methods to the materials of investigation, some aspects regarding the pre-treatment of sample before to perform the measure and the optimization of apparatus (dimension, tube diameter, weight of cone etc.) and procedure must be studied. For example using rotational viscometers or flow cone it will be studied how to pre-treat the sample by wet sieving to eliminate the coarse particles that can cause severe interference in the measure or the obstruction of the discharge tube. In all apparatus pipe diameter, tube length and diameters must be optimized. For thixotropic material (sol/gel-liquid) a vibration table pre-treatment or similar apparatus must be studied to pre-treat the sample and optimize the measure.

The proposed methods should be applicable to the whole range of materials (sludge, bio-waste and soil). Investigations in the past were often carried out only for several materials like sewage sludge or waterworks sludges for the parameter solidity or solid, particular bio-wastes for the parameter piling behaviour. But during these performed tests the influences of the material properties (grain size and shape, dry solids, etc.) were exhibited. By means of this knowledge it is possible to assume behaviour in general by using a special method.

For adaptation of methods for all materials mentioned above or for a more exact measure it is possible to vary the dimensions of the apparatuses: For example the cone size and shape or weight of the penetrometer tip according to the consistency of the investigated sample. Another example is to vary the size of the sidewalls of the Cubic Piling Box (CPB) according to the grain size of the investigated material. These variations are promising tools for optimisation of the measuring procedures. Further examinations of the proposed methods, which have to be carried out in the future, should confirm the theoretical considerations.

Another item is the pre-treatment of the samples before carrying out the investigations. Especially a rough determination of the consistency before application of a special method could be advantageous for the measurement. Different types of sampling and pre-treatment are required for each method. Important for the pre-treatment are the parameters:

- particle size of the material,
- dimensions of the mould,
- compaction of the materials,
- moreover the density of the sample.

For example the pre-treatment for measuring the vane shear strength is explained as follows [23]:

1) The material – if needed, dewatered – is reduced to small pieces with particle sizes of about 10 mm
2) The mould – in this case the small Proctor vessel – has an internal diameter of 100 mm and a height of 120 mm.
3) The material is filled into the Proctor vessel in three equal portions (each about 100 mm before compaction) and is compressed with ten knocks by the small Proctor hammer (falling weight of 2,5 kg, height of falling 300 mm, diameter of the plunger 50 mm, diameter of compensating plate 99,5 mm, compare *DIN 18127* [30]).
4) After finishing emplacement and before test performance the density have to be determined for a uniform starting position.

The procedure mentioned above could be transmitted to the pre-treatment of measuring the shear strength or related parameters - penetration bearing capacity and unconfined compression strength – in common and also for examination of the thixotropic behaviour, whereby the shear strength is measured during shear stress. For determination of the piling behaviour (piling angle) a pre-treatment in terms of reducing to small pieces with a uniform particle size - regarding the size of the sidewalls of the Cubic Piling Box (CPB) – is proposed. It should be a similar pre-treatment like the method for the shear strength, but instead of the Proctor vessel and hammer the CPB and, if needed, according compaction apparatuses are used.

#### 4.6.3 Questions to be answered

Research must define - through interlaboratory tests - precision, repeatability and reliability of methods proposed, where:

- *Repeatability* is defined as the ability of a method to reproduce a measurement while being tested under an unchanging set of conditions. This does not imply that the obtained value is correct, but rather that it is the same every time;
- *Reproducibility* is the same as repeatability, but at a different set of conditions. It is therefore a more realistic indication of a method to reproduce a measurement, whenever a predefined set of conditions is recreated.

However, as far as these methods are concerned the use of *fresh sludge* could be required, so the following problems arise:

- the sludge characteristics change with time of storage, also considered that any preservation practice (e.g. freezing) makes things worse;
- the sludge characteristics are strongly affected by transport and handling;
- fresh sludge requires particular precautions and authorization for transport by ordinary delivering systems.

#### 4.6.4 Route, how to answer them

CEN/TR 15252 concerns the validation of physical parameters [53]).

#### 4.6.5 Steps to be taken

- Laboratory tests (cp. 4.6.4)
- Evaluation (cp. 4.6.4)
- Final proposal methods to be standardised (Research Report, Version 1)
- Consulting with CEN/TC's/WG's, etc.
- Revision of proposal for methods to be standardised (Research Report, Version 2)
- Interlaboratory tests including evaluation
- Final draft for methods to be standardised

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