Second Draft

Physical Properties

- Flowability -

Desk study

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CONTENTS

LIST OF TABLES

LIST OF FIGURES

SUMMARY

The "*Horizontal*" project has the objective to develop horizontal and harmonised European standards in the fields of sludge, soil and treated bio-waste 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 and land-filling.
The importance of the physical consistency is also true for the characterization of treated 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 operation 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, divided into four parameter groups: solidity, flowability, thixotropy and piling from which it can be seen that more than 250 standards and non standardized methods are potentially applicable to consistency evaluation.
On the basis of the selected list for further consideration the methods for the determination of flowability of sludge, treated bio-waste and soil, have been divided into four groups, according to the instruments used for measuring: capillary viscometers, penetrometer, rotational viscometers and flow apparatus. For each group it was evaluated the laboratory or field test feasibility. Apparatuses of the measuring procedure and existing application to different materials were described. On these basis the applicability of the described methods to the materials for the Horizontal project was evaluated and documented in the list of analized standards.

The recommended methods for further investigation can be divided in two groups. The first group includes methods and instruments suitable to characterise Horizontal materials in the field. The second group includes methods and instruments suitable to characterise Horizontal materials in laboratory scale. The laboratory apparatus is the coaxial cylinder viscometer while field apparatus are flow cone, magnesium penetration cone and extrusion tube viscometer. They must be tested and optimized to adapt design and part dimensions to Horizontal material in a future experimental activity.

For the research needs first the basics of methods are explicated and o the applicability of methods to Horizontal 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 defined since they are important for following procedures within the Horizontal project.

The results of the circulation within the consulting CEN/TC's/WG1's/TG's – Committee's are documented by means of listed actions, received comments, and official meetings (four WP7 meetings, two CEN/TC308/WG1 meetings, one CEN/TC308/WG1/TG3, one CEN/TC292/WG2). In the Horizontal Report N° 21 a total of 4 proposals for draft standards are given, whereby one laboratory method and three field tests exists.

1. INTRODUCTION

1.1 The Horizontal project

The revision of the sewage Sludge Directive 86/278/EEC, the upcoming 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, land-filling 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 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 was issued (16/12/02).
The "*Horizontal*" project has the objective to develop horizontal and harmonised European standards in the fields of sludge, treated 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, treated 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.

1.2 The Work Package 7

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, land-filling.
In fact, the selection of the most suitable equipment and procedure for land application, storage and transportation of sludge, 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 must 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 land-filled, 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 land-filled 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 must 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 operator must 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 land-filling; 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 treated bio-waste and soil. Also handling and utilization of many other materials, such as cement and asphalt, are strictly depending on their physical consistency.
For WP7 two desk studies were performed - n° 21 for the parameter "Flowability" and n° 22 for parameter "Solidity", "Thixotropic behaviour" and "Piling behaviour". The work was coordinated in WP7 and done in cooperation of the involved teams. Therefore, the reports n° 21 and n° 22 contain some chapters (from 1.1 to 1.5, and 2) and the lists of standards and non standardized methods (Annex 1) in common.

1.3 Desk studies 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 define 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 from "the solid to the liquid state and vice versa" must be evaluated, together with the *piling* behaviour referred both to "compaction and physical stability".
Also the CEN/TC292/WG2, in the method prEN 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).
Unless 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 Horizontal material examination will consist of :

- sampling, transport, preservation, storage,
- pre-treatment,
- measurement and evaluation of results.

1.4 Evaluation of needs for control of operation and material characteristics

1.4.1 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. Material considered are:

- sewage sludge,
- water works sludges,
- treated bio-waste and
- soils.

For handling and operation of 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 considered (i.e. viscosity, etc.).

The parameters *flowability is an overall parameters* taking into account all above 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 and vice versa. Similarly, for *treated 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 has to be considered. Finally, for fine grained soils, the water content (and therefore consistency and flowability) have 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 treated 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 solids materials is especially for dewatered sludges like sewage, water works and related sludges relevant. By dewatering and storage the sludge will become solid. During operation such as transportation the sludge is getting due to the vibration in a liquid state. The piling behaviour evaluation is for dewatered sludge particular treated by-waste and soil of importance.

The determination of the piling angle is a useful instrument to characterize the storage properties and calculate the space, which is needed for e.g. storage and transportation of the above materials. Together with the thixotropic behaviour the piling behaviour referred to the compaction and stability.

However, the development of reliable measurement procedures of all parameters is not a simple matter, just because measurements are influenced by above listed properties or characteristics. This means that those factors must be considered with great attention and methods defined by avoiding any negative interference of them on the measure.
For this reason, it is first necessary to select, if any, the most adapted standards or non standardized methods applicable to sludges, treated 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.

1.4.2 Material characteristics

1.4.2.1 Sewage sludge

Sewage sludge can be produced from several process (primary sedimentation, activated sludge process, aerobic or anaerobic digestion, etc…). Their solid content cover a wide range from

minus 1 to 30%, while total volatile solids content 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 cover 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 in Table 1 [1]).

Material	Process	TS basis % cumulative retained w.w.			TVS basis % 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

Table 1: Particle size distribution of sewage sludges

Each kind of sludge was analysed for its particle size distribution by wet sieving, using three sieves with openings of 5, 2 and 0.82 mm. According to these data the samples can be divided into four conventional classes: coarse (>5 mm), medium (from 5 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].

Sludge	Process	TS range	TVS/TS	YS	Plastic viscosity
		%		Pa	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
Mixe 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

Table 2: Rheological properties of sewage sludges

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].
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 valid in Italy).

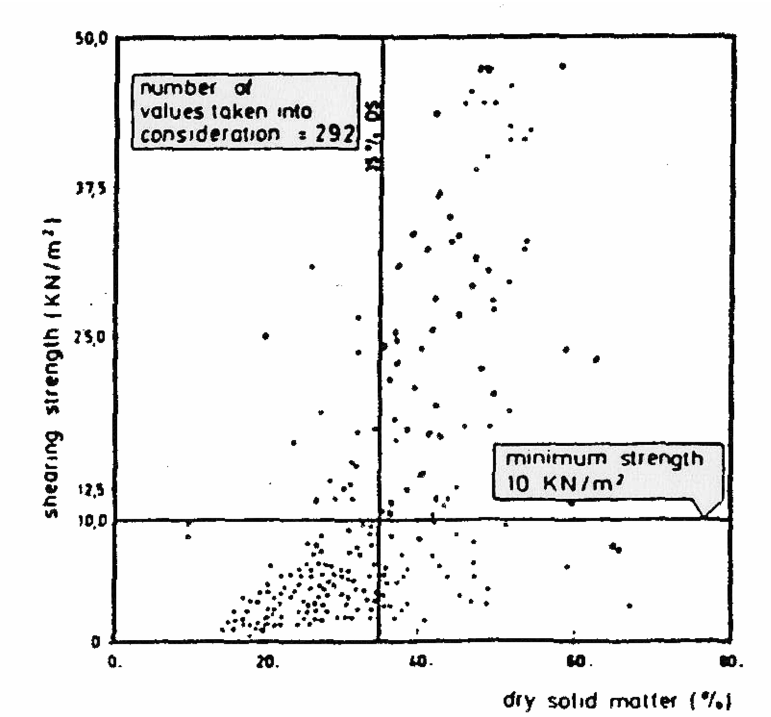


Figure 1: Correlation between shearing strength and dry solid method [9]

1.4.2.2 Water work sludge

In DVGW W221-1 [**Errore. L'origine riferimento non è stata trovata.**] 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 ordering of these phases, as well as the physical-chemical interactions between them, give 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.

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**

Table 3: Determination of the composition of sludge

An orderly utilisation and disposal of waterworks sludges needs the control of the mechanical properties in order to ensure a quality that is demanded for storage, transport and handling. The mechanical measurements are to be seen and done in connection with other measurements that have been or will be standardised. There are to 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 must 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 using ten different parameters as shown in Table 3 [11]

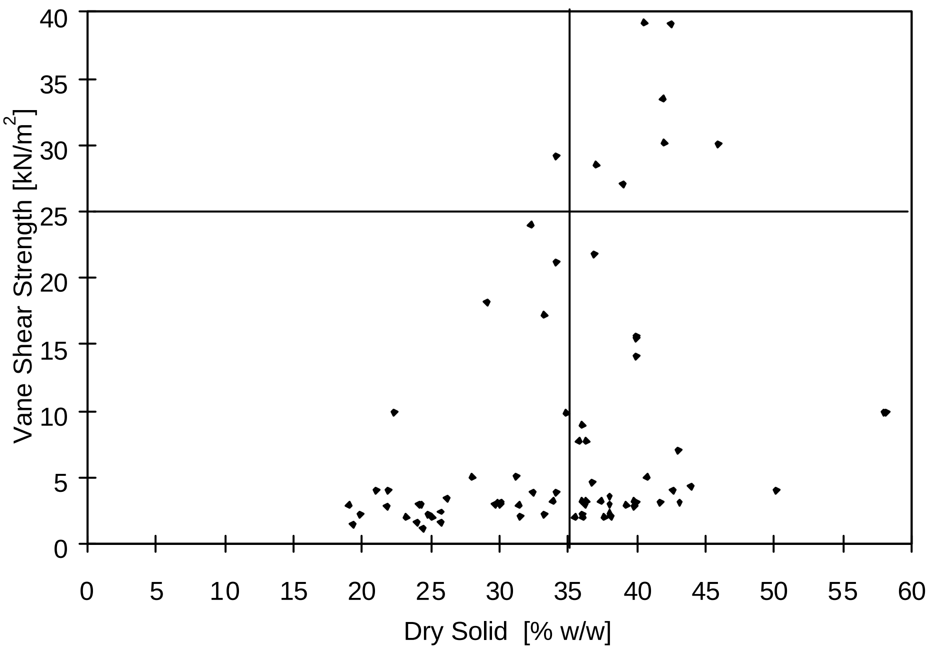


Figure 2: Comparison dry solid contents vs. laboratory vane shear strength [12]

The range of 0.2 to 80 % dry solids contents of waterworks sludges 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 sludges published. In

Figure 2 after *Mc Tigue et al. (1990)* [**Errore. L'origine riferimento non è stata trovata.**] 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)* [**Errore. L'origine riferimento non è stata trovata.**] as a minimum value for disposal of wastes in landfills. Sludges 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 for the vane shear strength that is now demanded from new regulations in the *TA Siedlungsabfall (1993)* [**Errore. L'origine riferimento non è stata trovata.**]. Approximately 90 % of the waterworks sludges tested after mechanical dewatering could not fulfil the required minimum value [1516].

1.4.2.3 Treated 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 process control. The OFMSW was analysed for its particle size distribution by wet sieving, using three sieves with openings of 5, 2 and 0.82 mm (Table 4). According to these data the samples can be divided into four conventional classes: coarse (>5 mm), medium (from 5 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 [19].

Material	Process	TS basis % cumulative retained w.w.			TVS basis % cumulative retained w.w.		
		5 mm	2 mm	0.84 mm	5 mm	2 mm	0.84 mm
Fresh mechanically sorted OFMSW	Mesophilic anaerobic digestion	8,2	11,1	18,3	6,7	21	23,5
Pre-composted mechanically sorted 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

Table 4: Particle size distribution of OFMSW

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 yie ld stress (YS) (that is the stress required to start the material flowing) and 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) [19,19].

Sludge	Process	TS range	TVS/TS	YS	Plastic viscosity	Thixotropy
		%		Pa	mPa.s	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

Table 5: Rheological properties of OFMSW aerobically or anaerobically treated

1.4.2.4 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. This 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 defined by the grain distribution curve (Table 6), 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 influence 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 defined 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 D 4318) but it is recognised to be strongly affected by the operator experience. Typical values of liquid limit for natural fine grained soils ranges from 40% up to 90% of water content and the plastic limit from 10%-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.

No tests are available concerning the soil characterization at the liquid state, although the soil behaviour at the liquid state is important, e.g. with reference to mudflows or debris flow. A mudflow is a river of rock, earth and other debris that is saturated with water. It develops when water rapidly accumulates in the ground, such as during a heavy rainfall or rapid snowmelt, changing the earth into a flowing river of mud or a slurry. A slurry can flow rapidly and strike with little or no warning. Its sediment composition typically consists of at least 50% sand, silt and clay particles. Mudflows usually occur on steep slopes where vegetation is too sparse to prevent rapid erosion, but they can also occur on gentle slopes under certain conditions. Factors other than slope include heavy precipitation in a short period, easily erodible material and chemical compounds in soil pore water.

Debris flows incorporate large qualities of sediment and are often triggered by landslides that mix with flood waters. Debris flows usually follow stream channels and construct their own levees as they move, but their exact paths are unpredictable. These types of flows are coarser and less cohesive than mudflows. A typical debris flow consists of about 2 parts sediment for every one part water.

Both flows can erode or deposit sediment along its journey downstream; this ratio can change with increasing distance from the origin of the flow.

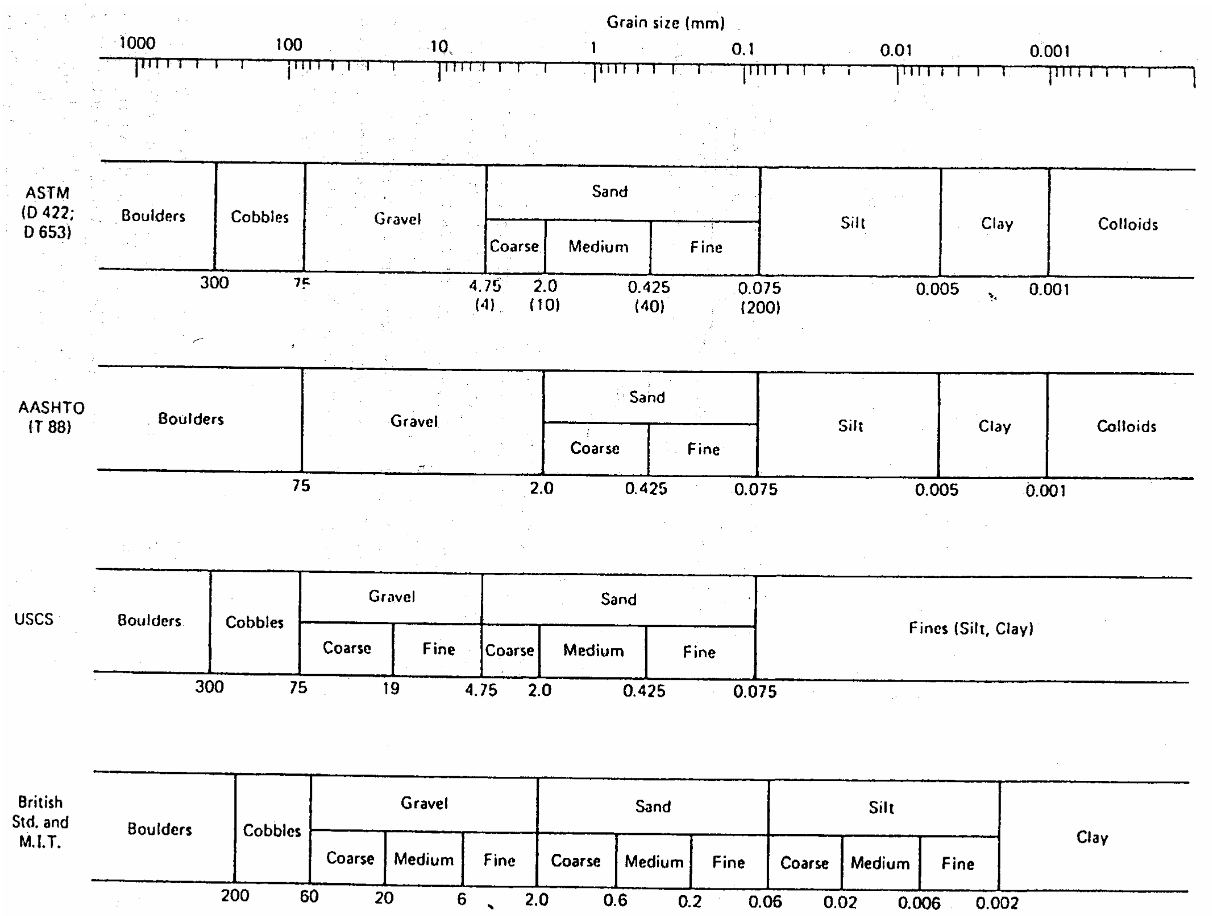


Table 6 : Grain size of soils according to different classification systems.

1.5 Search for existing standards and methods

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.

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

- ISO at www.iso.ch
- ASTM at www.astm.org
- CEN at www.cenorm.be
- 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 webstore.ansi.org

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: *consistency, viscosity, flowability, shearing, sludge, soil, physical properties, flow properties, suspensions*, *compactibility*. The different materials resulted from the research were resins, plastic, lubricant, cement, asphalts/bitumens, soil, solid waste, etc..

The complete list of standards is reported in Annex 1, divided into four keyword groups: solidity, flowability, thixotropy and piling; from which it can be seen that more than 250 standards and non standardized methods are potentially applicable to consistency evaluation.

1.6 Basic information

1.6.1 Flowability and rheology

Flowability is the state in which a material is able to "*flow*", 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 sludges 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 1, as $T=\mu D$, 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 1, many models are applicable: a general equation is $T=T_0+\mu D^n$, where T_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 [20,20].

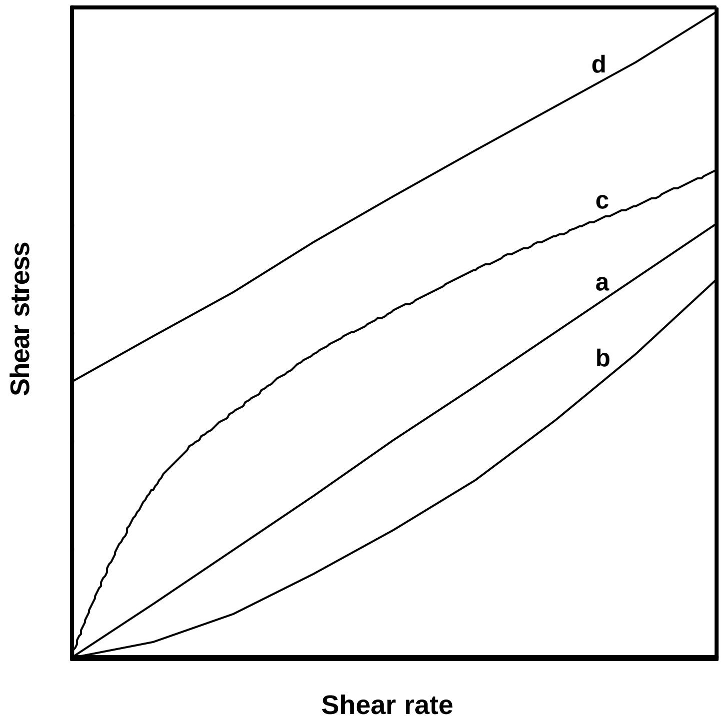


Figure 3: Rheogram types
(a=Newtonian; b=Dilatant; c=Ostwald pseudoplastic; d=Bingham plastic)

1.6.2 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).

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

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

Table 7 shows the applicability of different treatment/disposal options on sludges having different physical state [22].

1.6.3 Rheological measurements

A lot of viscometers are available. They generally fall into two general categories:

- **tube type** (often referred to as capillary viscometer);
- **rotating type** .

The use of a tube viscometer is a good approach to completely define 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 must 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 sludges. 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 must 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.

2. EXISTING STANDARDS OR DRAFT STANDARDS

The research of existing and draft standards to be utilized as laboratory and field methods for Horizontal standardization has been carried out 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 committee has submitted titles to a preliminary examination and the selected standards (considered for further discussion) have been acquired. They can be divided generally in to 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
- Viscosity
- Waste (solid)

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 has been reported in Annex 1. The list of standards considered for further discussion is presented in chapter 3.

3. EVALUATION OF DRAFTING A HORIZONTAL STANDARD

On the basis of the selected list of standard and non-standardised methods for …….. consideration the methods for the determination of flowability of sewage sludges, water works sludge, treated bio-waste and soil, have been divided into four groups, according to the instruments used for measuring:

- ✓ capillary viscometers
- ✓ penetrometer
- ✓ rotational viscometers
- ✓ flow apparatus

3.1 Capillary viscometers

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

3.1.1 Standards analysed

The list of standard methods analysed for flowability of different materials is summarized in the following Table 8.

Method	**Material**	**Parameter**	**Apparatus**	**Standard range**	**Physical meaning/purpose**	**Utility / field**	**Comments**
ASTM D 1238 - 98 Flow Rates of Thermoplastics by Extrusion Plastometer	Molten resins	Melt flow index	Extrusion plastometer	0,15-50 g/10 min	Flow rate of extrusion of the molten sample through a die by a weighted piston at controlled temperature	Quality control tests of thermoplastics. The rate of extrusion is critically influenced by the physical properties	-
ASTM D 1601 - 86 Dilute Solution Viscosity of Ethylene Polymers	Ethylene Polymers Solutions	Viscosity	Mod. Ubbelohde viscometer	Viscosity at 135°C	Directions are given for the determination of relative viscosity, inherent viscosity and intrinsic viscosity	Characterisation and prediction of process behaviour of polymers	-
ASTM D 2170 - 95 Kinematic Viscosity of Asphalts (Bitumens)	Road oils and bitumens	Kinematic viscosity	Reverse flow capillary viscometers	6-100000 cSt	Time for a fixed volume of the liquid to flow under gravity through calibrated glass capillary viscometer at a closely controlled temperature (usually at 60 and 135°C)	To establish the uniformity of shipments or sources of supply	-
ASTM D 2171 - 94 Viscosity of Asphalts by Vacuum Capillary Viscometer	Bitumen	Viscosity at 60°C	Vacuum capillary viscometer	0.036-200000 P	Time for a fixed volume of the liquid to be drawn up through a capillary tube by means of vacuum at closely controlled conditions of vacuum and temperature	The viscosity characterises flow behaviour and may be used for control requirements for cutbacks and asphalt cements	-

ASTM D 2270 - 79 Calculating Viscosity Index from Kinematic Viscosity at 40 and 100 °C	Petroleum products	Viscosity Index	Tables Calculation procedures	2 >70 cSt at 100°C	Procedures and tables to calculate the viscosity index of petroleum products from kinematic viscosity at 40 and 100°C	The viscosity index characterises the variation of kinematic viscosity with temperature	-
ASTM D 2857 - 95 Dilute Solution Viscosity of Polymers	Dilute polymer solutions	Relative viscosity Inherent viscosity Intrinsic viscosity	Glass capillary viscometer		Time for a fixed volume of the liquid to flow under gravity through calibrated glass capillary viscometer at a closely controlled temperature	Molecular characteristics of polymers (molecular weight, chain length, ..)	-
ASTM D 445 - 79 Kinematic Viscosity of Transparent and Opaque Liquids	Liqud petroleum products	Kinematic and dynamic viscosities	Glass capillary viscometer		Time for a volume of a liquid to flow under gravity through a calibrated capillary viscometer	Applicable to opaque and transparent fluids	-
ASTM D 446 - 74 Glass Capillary Kinematic Viscometers	Liquids	Kinematic viscosity	Gravity flow glass capillary viscometers		Specifications and operating instructions for various types of gravity flow glass capillary viscometers	-	-
ASTM D 5225 - 98 Measuring Solution Viscosity of Polymers with a Differential Viscometer	Solutions of polymers	Relative viscosity Inherent viscosity Intrinsic viscosity	Differential pressure stainless steel capillary viscometer		This method differs from the glass capillary in that the solvent and the solution are compared at the same time that a test is run. The pressure difference is proportional to specific viscosity of the solution	Solution viscosities are related to molecolar size of soluble polymers	-
ASTM D 5422 - 93 Measurement of Properties of Thermoplastic Material by Screw-Extrusion Capillary Rheometer	Thermoplastics and thermoplastic compounds	Apparent viscosity	Screw-extrusion capillary rheometer		The material is forced through a capillary die. The temperature, pressure entering the die, and flow rate through the die is measured. The viscosity of the sample is calculated by the extruded volume in a certain time interval	The data produced are useful in both quality-control testing and compound development	-
UNI EN ISO 3104 Petroleum products. Determination of Kinematic Viscosity and Calculation of Dynamic Viscosity	Petroleum products	Kinematic and dynamic viscosity	Glass capillary viscometer	0,5 - 100000 cSt	Time for a volume of a liquid to flow under gravity through a calibrated capillary viscometer	Applicable to opaque and transparent fluids	-
ASTM D 4624 - 93 Measuring Apparent Viscosity by Capillary Viscometer at High-Temperature and High-Shear Rates	Lubricant	Kinematic and dynamic viscosity	Met. A: Constant pressure capillary viscometer Met. B: Constant flow-rate capillary viscometer	Temp. 150°C Shear rate 10^6 s^{-1} Capillary diameters 0,1-0,2 mm	Met. A: the pressure is set and the resulting flow rate is measured Met. B: the flow rate is set and the resulting pressure is measured The viscosity is calculated by a calibration curve.	Viscosity of engine oils at the high shear rates and high temperatures that occur in engine Applicable to opaque and transparent fluids	-

ASTM D 4957 - 95 Apparent Viscosity of Asphalt Emulsion Residues and Non-Newtonian Bitumens by Vacuum Capillary Viscometer	Asphalt emulsion residues and bitumens	Kinematic and dynamic viscosity	Vacuum capillary viscometer	5 - 50000 Pa.s Capillary radius 0,25-2,0 mm	Time required for the meniscus to pass between successive pairs of timing marks	Flow behaviour of asphalt emulsion residues and bitumens	-
ASTM D 5099 - 93 Rubber-Measurement of Processing Properties Using Capillary Rheometry	Rubber	Kinematic and dynamic viscosity	Met. A: piston type capillary rheometer Met. B: screw extrusion type capillary rheometerr	Shear rates 300-1000 s^{-1} Temp. :100-125°C	Met. A: the shear rate is calculated from the speed of the piston and the shear stress from the pressure transducer at the die entrance Met. B: the shear rate is calculated from the volumetric flow and the shear stress from the pressure transducer at the die entrance	Resistance to flow of raw or compounded unvulcanised rubber. Data useful for both quality control and compound development	-
ISO/DIS 11443 Plastics - Determination of the Fluidity of Plastics Using Capillary and Slit-Die Rheometers	Melt plastics	Viscosity	Heatable extrusion rheometer with capillary die or slit die	Viscosities 10-10^7 Pa.s Shear rates from $1s^{-1}$ to 10^6 s^{-1} Temp: 150-400°C	Met. 1: the volume flow rate is measured at a specified constant test pressure Met 2: the test pressure is measured at a specified constant volume flow rate.	Fluidity of plastic melts at shear stresses, shear rates and temperatures approximating to those arising in plastic processing	-
ISO 1599 Plastics - Cellulose Acetate - Determination of Viscosity Loss on Moulding	Moulded cellulose acetate	Viscosity	Hydraulic press Mould Apparatus for the determination of viscosity ratio according to ISO 1157:1990		Determination of viscosity ratio of 5 g/l solutions of cellulose acetate	Reduction in viscosity due to depolymerization which occur when cellulose acetate is moulded	-
ISO 3104:1994 Petroleum Products - Transparent and Opaque Liquids - Determination of Kinematic Viscosity and Calculation of Dynamic Viscosity	Liquid petroleum products	Kinematic viscosity	Glass capillary viscometer	Viscosities 0,2-$3x10^5$ cSt Temp: 40-150°C	The kinematic viscosity is determined by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer at closely controlled temperature.	Applicable to opaque and transparent fluids	-

+ considered for further investigation; - considered not appropriate

Table 8: Standards examined for capillary viscosimeters

3.1.2 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.

3.1.3 Apparatus

The tests examined use five types of viscometers:

- Gravity flow capillary viscometers (see Figure 4);
- 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.

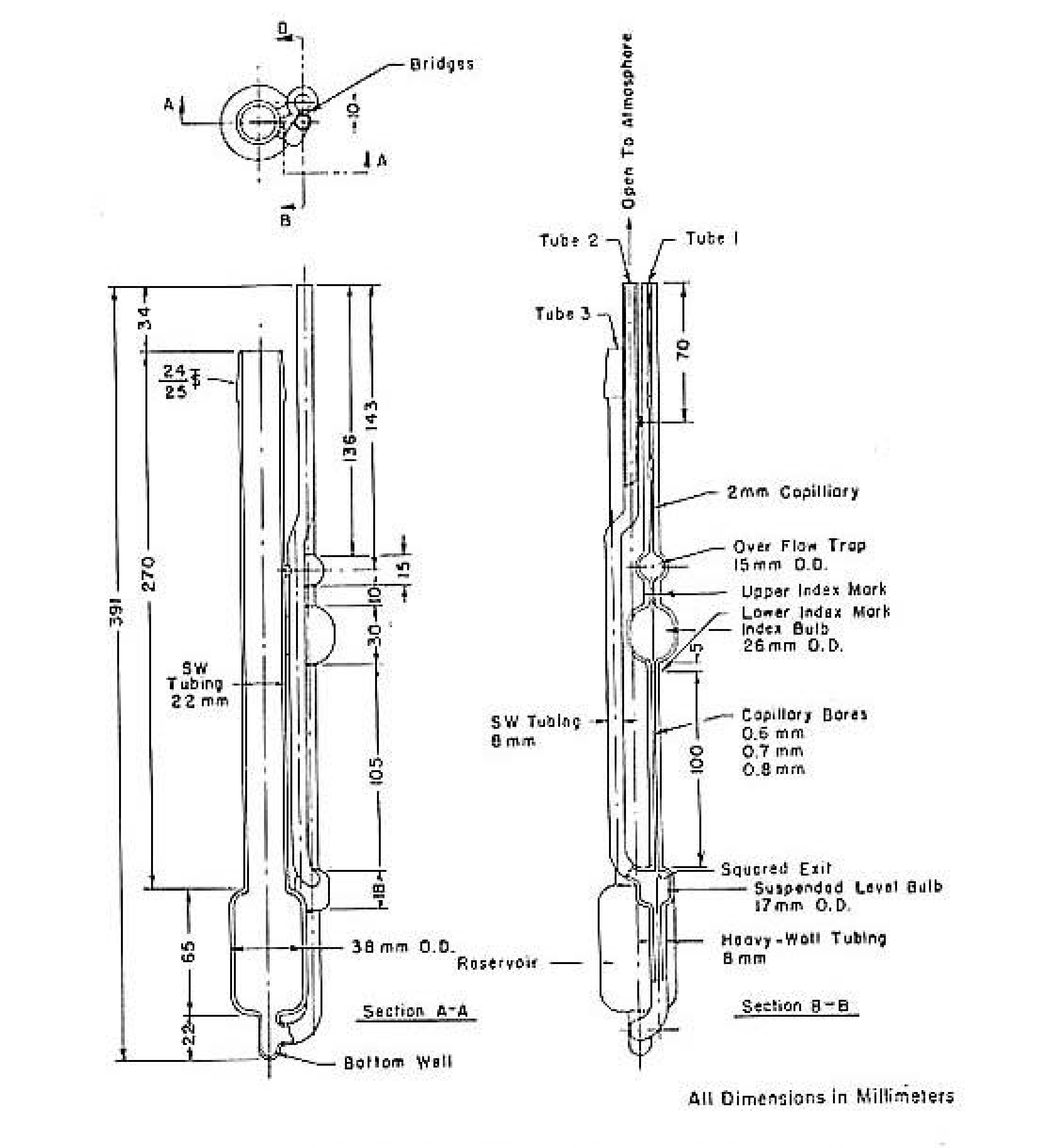


Figure 4: Gravity flow capillary viscometer (Modified Ubbelohde type)

3.1.4 What is measured and how

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

- Apparent viscosity;
- Dynamic viscosity;
- 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

3.1.5 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;

3.1.6 Feasibility of the methods to the materials for Horizontal project

None of the methods examined or of the apparatus used can be transferred to the materials for horizontal project, 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 a horizontal strategy where the widest diffusion of methods is preferred.

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

3.2 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.

3.2.1 Standards analysed

The list of the examined standard methods for the determination of flowability of different materials by penetration tests is summarised in the following table:

Method	**Material**	**Parameter**	**Apparatus**	**Standard range**	**Physical meaning/purpose**	**Utility / field**	**Comments**
ASTM C 405 - 82 Standard Practice for Estimating Consistency of Wet-Mixed Thermal Insulating Cement	Thermal insulating cement	Consistency	Method A: Dead load tester Method B: Penetration tester		Percentage of deformation of sample under a loading arm Rod penetration in mm after 30 s	The consistency of cement affects such properties as ease of trowelling, wet adhesion, drying shrinkage, dry density and thermal conductivity	-
ASTM C 472-84 Physical Testing of Gypsum Plasters and Gypsum Concrete	Gypsum plasters Gypsum concrete	Physical testing	Modified Vicat apparatus Consistometer		ml of water added to 100 g of the sample to obtain a penetration of 20 mm Resulting patty from conical vessel	-	+
ASTM D 1558 - 84 Moisture Content Penetration Resistance Relationships of Fine-Grained Soils	Fine-grained soil	Penetration resistance	Laboratory soil penetrometer		Pressure necessary to penetrate the soil with a needle of known end area	Relationship between moisture content, density and penetration resistence of a soil	-
ASTM D 3441 - 79 Deep, Quasi-Static, Cone and Friction-Cone Penetration Tests of Soils	Soil and soft rocks	Penetration resistance	Field cone and friction cone penetrometer Mechanical and electric types		Penetration resistance with depth during the steady slow penetration of a pointed rod into soil	Engineering properties of soil related to design and construction of earthworks and foundations for structures	-
UNI EN 1015 - 4 Determination of Consistence of Fresh Mortar	Fresh mortar	Consistency	Plunger penetration		Free penetration of a plunger released from a determined height	The result is related to the fluidity or water content of fresh mortar and measures the workability of the product	+
ASTM C 807 - 99 Time of Setting of Hydraulic Cement Mortar by Modified Vicat Needle	Hydraulic Cement Mortar	Consistency	Mod. Vicat apparatus		Time required to obtain the stipulate penetration of the sample	Determination of the time of setting of cement expansive	+
ASTM D 2884 - 93 Yield Stress of Heterogeneous Propellants by Cone Penetration Method	Heterogeneous propellants	Yield stress	Magnesium penetration cone and cup	42-60 mm of cone penetration at 20°C	Penetration of the cone after 5 s The yield stress is calculated by the use of a proper equation	The yield stress is a measure of the forces required to initiate and maintain flow	+

ASTM D 5478 - 98 Viscosity of Materials by Falling Needle Viscometer	Newtonian and non-Newtonian liquids	Viscosity and yield stress	Falling needle viscometer with automatic sensing device. Thermostatic bath	Viscosity range: $5x10^{-4}$ to 10^{3} Pa.s Shear rate range: 10^{-4} to 10^{3} s^{-1}	The liquid viscosity is determined by measuring the steady-state or terminal velocity of cylindrical needles as they fall through the tests liquid under gravity	Clear and opaque liquids. Rheological properties of varnishes and paints provide information to sag resistance, levelling, etc.	-
ISO/DIS 12058-2.2 Plastics - Determination of Viscosity Using a Falling-ball Viscometer Part 2: Free-Falling Ball Method	Polymers and resins in the liquid emulsified or dispersed	Dynamic viscosity	Vertical-tube falling ball viscometer	700 to 600000 mPs.s	The viscosity is determined by measuring the time taken for a ball to fall between an upper and lower marks at controlled temperature	Used for the determination of relatively high values of viscosity of transparent liquids	-
ISO/DIS 12058-1 1997 Plastics - Determination of Viscosity Using a Falling-Ball Viscometer Part 1: Inclined-tube Method	Polymers and resins in liquid emulsified or dispersed state	Dynamic viscosity	Inclined-tube falling ball viscometer	0,6-250000 mPa.s -20-+120°C	The viscosity is determined by measuring the time taken for a ball to fall between an upper and lower marks in an inclined tube filled with the liquid at controlled temperature	Used for the determination of dynamic viscosity of Newtonian fluids at controlled temperature	-
ISO 12115 1997 Fibre-Reiforced Plastics - Thermosetting Moulding Compounds and Prepregs - Determination of Flowability, Maturation and Shelf Life	Fibre reinforced thermosetting moulding compounds and prepregs	Flowability	Method 1: apparatus for applying a constant load to the tests specimen Method 2: Hydraulic moulding press		Method. 1: The sample is subjected to a constant load by a punch at room temperature. The change in height of the punch as it sinks is measured Method 2: The sample is pressed under the commonly used moulding conditions.The force necessary to spread out the sample and to fill the mould within a certain time is measured	Used to assess the influence of individual components of the moulding compound on the moulding behaviour by determining the flowability of the compound	-
BS 2000: Part 49:1993 Petroleum and its Products Part 49. Determination of Needle Penetration of Bituminous Material	Solid and semisolid bituminous material	Consistency	Penetration apparatus with conical tip needle Water bath	Usual test conditions: Temp: 25°C Load: 100g Time: 5s	The consistency is expressed as the distance in tenths of mm that a standard needle penetrates vertically into the material under specified conditions of temperature, load and duration of loading	Consistency of bituminous material for road pavements and industrial applications	+

BS 3712: Part 1: 1991 Building and Construction Sealant Part 1. Methods of test for homogeneity, relative density and penetration	Construction sealant	Consistency	Penetrometer with penetration needles and penetration cones Thermostatic bath	Usual test conditions: Temp: 25°C Load: from 50 to 150g Time: 5s	The penetration is expressed as the distance in tenths of mm that a standard needle penetrates vertically into the material under specified conditions of temperature, load and duration of loading	Properties of sealant particularly in relation to their compatibility with other materials and to their suitability for application to building joints	-
BS EN 196-3 : 1995 Methods of Testing Cements Part 3. Determination of Setting Time and Soundness	Cement	Water corresponding to standard consistence, setting time and soundness	Vicat apparatus with plunger. Le Chatelier apparatus Water bath	The standard consistence of the cement paste corresponds to a sample which produces a distance between plunger and base-plate of 6 mm The setting time corresponds to a needle penetration of only 0,5 mm	The setting time is determined by observing the penetration of a needle into cement paste of standard consistence until it reaches a specified value. The soundness is determined by the volume expansion of cement as indicated by the relative movement of two needles. The water required to obtain a standard consistence of the cement paste is determined by trial penetration tests with different water contents.	Setting time and soundness of cements	-
AFNOR XP T 51-213 Nov. 1995 Plastics- Polymers, Resins in Liquid, Emulsion or Dispersion State, Varnishes, Paints, Oils- Determination of the Dynamic Viscosity Under Low Shear Rate by Vertical Ball Falling	Emulsions or dispersions of various materials	Dynamic Viscosity	Vertical-tube falling ball viscometer Thermostatic bath	0,05-200 Pa.s	The viscosity is determined by measuring the time taken for a ball to fall between an upper and lower marks at controlled temperature	Dynamic viscosity of Newtonian fluids. Apparent viscosity of non-Newtonian fluids. Applicable for production control	-

+ considered for further investigation; - considered not appropriate

Table 9: Standards examined for penetrometer

3.2.2 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.

3.2.3 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 5)
- Laboratory soil penetrometer consisting of specific spring dynamometer and a set of needles of various end area (see Figure 6)
- Field soil penetrometer
- Magnesium cone penetrometer for yield stress measurements (see Figure 7)
- Falling needle viscometer
- Vertical tube falling ball viscometer

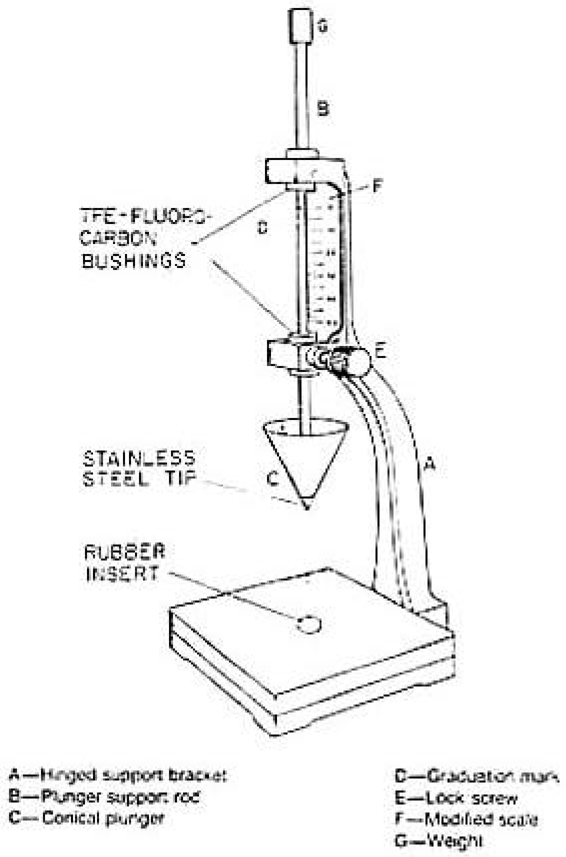


Figure 5: Vicat apparatus

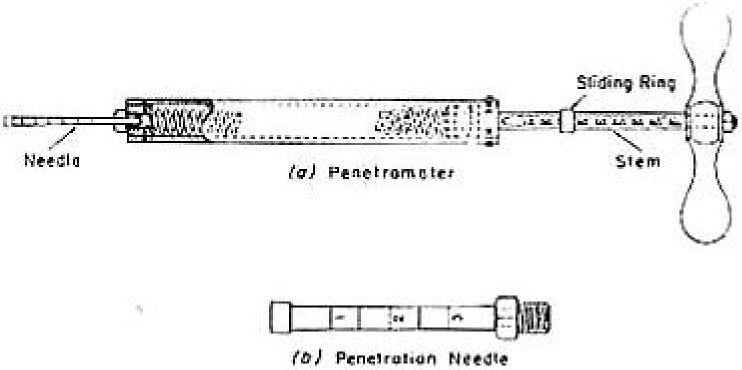
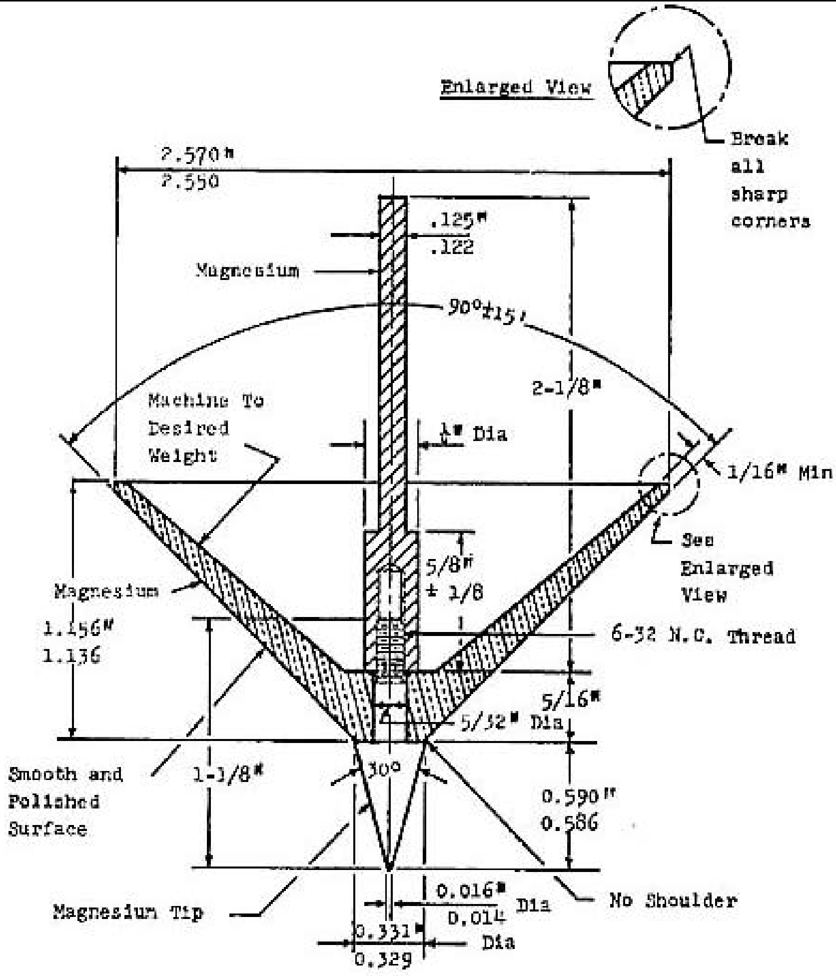


Figure 6: Laboratory soil penetrometer



NOTE 1—Tolerances on all fractional dimensions shall be 1/64 in.
NOTE 2—The total mass of the cone shall be 15.00 + 0.05 g and the total mass of its movable attachments shall be 15.00 ± 0.05 g.
NOTE 3—Table of Metric Equivalents

in.	mm	in.	mm	in.	mm	in.
0.014	0.356	1/8	3.18	0.331	8.407	1.136
0.016	0.406	5/32	3.98	0.586	14.884	1.156
1/16	1.59	1/4	6.35	0.590	14.986	2 1/8
0.122	3.10	5/16	7.94	5/8	15.88	2.550
0.125	3.18	0.329	8.357	1 1/8	28.575	2.570

Figure 7: Magnesium Cone Penetrometer

3.2.4 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.

3.2.5 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.

3.2.6 Feasibility of the methods to the materials for Horizontal project

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 and cannot be used for the scope of the Horizontal project.

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 Horizontal materials.

3.3 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.

3.3.1 Standards analysed

The examined standard methods for the determination of flowability of different substances by rotational viscometers are reported in the following table:

Method	**Material**	**Parameter**	**Apparatus**	**Standard range**	**Physical meaning/purpose**	**Utility / field**	**Comments**
ASTM D 1084 - 63 Viscosity of Adhesives	Free- flowing adhesives	Viscosity	Met. A: consistency cup Met. B: Brookfield viscometer Met. C: Stormer viscometer Met. D: Zahn viscosity cups	50-200000 cP <3000 cP	Time for gravity flow through a calibrated orifice in a consistency cup Viscosity in cP measured with a Brookfield viscometer Time for 100 revolutions in a Stormer viscometer Time for gravity flow through a calibrated orifice in Zahn cups	For newtonian fluid For newtonian fluid For newtonian fluid	-
ASTM D 2196 - 81 Rheological Properties of Non-Newtonian Materials by Rotational Viscometer	Non-newtonian liquids	Rheological properties	Met. A: Brookfield viscometer Met. B: Brookfield viscometer Met. C: Brookfield viscometer	Shear rate 0.1-50 s^{-1} Shear rate 0.1-50 s^{-1} Shear rate 0.1-50 s^{-1}	Apparent viscosity Shear thinning and thixotropy Shear thinning of sheared mat.	Time dependent materials Time dependent materials	-
ASTM D 2556 - 69 Apparent Viscosity of Adhesives Having Shear-rate Dependent Flow Properties	Adhesives	Apparent viscosity	Rotational viscometer	100-2000000 Cp	Viscosities measured at different shear rates	Non-Newtonian materials	+

ASTM D 2983 - 87 Low-Temperature Viscosity of Automotive Fluid Lubricants Measured by Brookfield Viscometer	Automotive fluid lubricants	Viscosity	Brookfield viscometer	1000-1000000 mPa.s -5 to -40°C	Low shear rate viscosity automotive oils at low temperatures	Fluid lubricants are non-Newtonian at low temperatures	-
ASTM D562 Consistency of Paints Using the Stormer Viscometer	Paints	Consistency	Stormer viscometer	Normal range 4 to 14 P at 25°C	Consistency is related to the load in grams required to produce a rotational frequency of 200 rpm for an off-set paddle rotor immersed in the paint	Product control	-
prEN 13702 - 2 Bitumen and Bituminous Binders - Determination of Dynamic Viscosity of Modified Bitumen - Part 2 : Coaxial Cylinders Method	Bitumens	Dynamic viscosity	Coaxial cylinder viscometer	Viscosity range 10^{-1} to 10^{6} Pa.s Shear rate range $1s^{-1}$ to $10^{4}s^{-1}$ Temperature range 60 to 150°C	The viscosities are measured at different shear rates in a coaxial cylinder viscometer with the ratio of radius R_1/R_2<1,1	Service condidtions of asphalt Conditions at the end of compaction asphalt Conditions during mixing of asphalt	+
prEN 13302 Bitumen and Bituminous Binders - Determination of Viscosity of Bitumen Using a Rotational Spindle Apparatus	Bitumen and bituminous binders	Viscosity	Rotating spindle apparatus	50 to 250°C	Apparent viscosity at application temperatures		-
UNI 8701 Part 3 Epoxy Resins. Determination of Viscosity by Means of Rotational Viscometer	Epoxy resins	Dynamic viscosity	Brookfield viscometer Syncro - Lectric mod. LVF	0,001 - 100 Pa.s	Viscosity of epoxide resin solutions in sutable solvents		-
ASTM D 4016 -93 Viscosity of Chemical Grouts by Brookfield Viscometer (Laboratory Method)	Catalysed chemical grouts	Viscosity	Brookfield viscometer	0,001 - 1 Pa.s at 20°C	Resistance to flow of grouts	Penetrability of grouts in soil voids and rock fissures	-
ASTM D 4648 - 00 Laboratory Miniature Vane Shear Test for Saturated Fine-Grained Clayey Soil	Saturated fine-grained clayey soil	Shear strength	Vane blade Vane device with torque measuring system	60-90°/min <100kPa	Determination of torque required causing a cylindrical surface to be sheared by vane. The torque is converted in shear resistance by a calibration constant	Testing consistency and shear strength of clay soils for engineering design and construction	-
ASTM D 4684 - 02a Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature	Engine oils	Yield stress Apparent viscosity	Concentric cylinder mini-rotary viscometer with temperature control system usable at controlled shear stress and controlled shear rate	-10 to -40°C Viscosity measurement s at > = 525 Pa and shear rate from 0.4 to 15 s^{-1}	Yield stress and viscosity of oils are measured after cooling at controlled rates over a period exceeding 45 h	Effect of cooling on oil yield stress and viscosity	-

ASTM D 6080 - 97 Defining the Viscosity Characteristics of Hydraulic Fluids	Hydraulic Fluids	Dynamic viscosity at low temperatures Kinematic viscosity at high temperatures	Brookfield viscometer	750 mPa.s from -50 to +16°C from 4 to 150 cSt at 40°C	Provide a better description of the viscosity characteristics of lubricants used as hydraulic fluids	Relationship between viscosity and hydraulic fluid performance	-
ASTM D 6204 - 01 Rubber-Measurement of Unvulcanized Rheological Properties Using Rotorless Shear Rheometers	Raw rubber and unvulcanized rubber compounds	Viscoelastic properties	Torsion strain rotorless oscillating rheometer with a sealed cavity	100 and 125°C Loss modulus G" Storage modulus G' Dynamic viscosity	Measurement of the viscous torque and elastic torque produced by oscillating angular strain of set amplitude and frequency in a completely closed and sealed test cavity	The rheological properties are related to factory processing	-
ASTM D 6243 - 98 Determining the Internal and Interface Shear Resistance of Geo synthetic Clay Liner by the Direct Shear Method	Geosynthetic clay liner (GCL)	Internal shear resistance	Shear device Normal stress loading device Shear force loading device Displacement indicator		A constant normal stress is applied to the specimen placed in a shear box and a tangential shear force is applied to the apparatus so that one section of the box moves in relation to the other section. The shear force is recorded as a function of the horizontal displacement of the box.	The method is intended to indicate the performance of the selected specimen by attempting to model certain field conditions	-
ASTM D 6821 - 02 Low Temperature Viscosity of Drive Line Lubricants in a Constant Shear Stress Viscometer	Drive line lubricants	Viscosity Yield stress	Mini-rotary constant shear stress viscometer	-40 to 10°C	Rotation of the rotor is achieved by applying load acting through a string wound around the rotor shaft. Time of rotation is measured electronically	Evaluate the low temperature performance of driveline lubricant as gear oils, automatic transmission fluids, etc.	+
ISO 2555 1989 Plastics - Resins in the Liquid State or as Emulsions or Dispersions - Determination of Apparent Viscosity by the Brookfield Test Method	Resins in a liquid state	Apparent viscosity	Brookfield-type viscometer	0,02-60000 Pa.s	The viscosity is determined by the torque exerted on the spindle rotating in the product at a fixed rotational frequency.	Applicable to non-Newtoninan fluids The viscosity depends on the velocity gradient to which the product is subjected during the measurement	-
ISO 2884-1 1999 Paints and Varnishes - Determination of Viscosity Using Rotary Viscometers- Part 1: Cone and Plate Viscometer Operated at a High Rate of Shear	Paints, varnishes and related products	Dynamic viscosity	Cone and plate viscometer	0,1-1 Pa.s 9000-12000 s[-1]	The viscosity is determined by the torque exerted on the cone rotating on a plate where the test liquid is placed. Used for routine high-shear viscosity measurements	The value obtained gives information about the resistance offered by the material to brushing, spraying and roller coating during application	-

ISO 3219 1993 Plastics - Polymers/Resins in the Liquid State or as Emulsions or Dispersions - Determination of Viscosity Using a Rotational Viscometer with Defined Shear Rate	Polymer and resin dispersions or emulsions	Dynamic viscosity	Rotational viscometer with standard geometry operating at defined shear rates and controlled temperature	10^{-2}-10^{3} Pa.s	The viscosity is determined by the torque, proportional to the shear stress, measured at defined shear rate	Applicable to Newtonian and non-Newtonian fluids	+
ISO/FDIS 2884-2 2002 Final Draft Paints and Varnishes - Determination of Viscosity Using Rotary Viscometers Part 2: Disc or Ball Viscometer Operated at a Specified Speed	Paints, varnishes and related products	Dynamic viscosity	Rotary viscometer with disc and ball spindles operating at controlled temperature	Up to 34 Pa.s	The methods gives only relative test results due to undefined velocity gradients	Applicable mainly during production and thinning	-
NF T 30-029 Oct. 1980 Paints - Method of Use a Rotational Viscometer for Measuring the Apparent Dynamic Viscosity of Varnishes, Paints and Similar Preparations	Paints, varnishes	Apparent dynamic viscosity	Rotational viscometer with standard geometry operating at defined shear rates and controlled temperature	from 25×10^{-3} to 8×10^{3} Pa.s	The viscosity is determined by measuring the torque, proportional to the shear stress, at defined shear rate	Applicable to production control	+

+ considered for further investigation; - considered not appropriate

Table 10: Standards examined for rotational viscosimeters

3.3.2 Laboratory or field test feasibility

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

3.3.3 Apparatus

Different types of rotational viscometers are reported in the methods:

- Brookfield viscometers (see Figure 8)
- Coaxial cylinder viscometers (see Figure 9)
- Stormer viscometers (see Figure 10)
- 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.

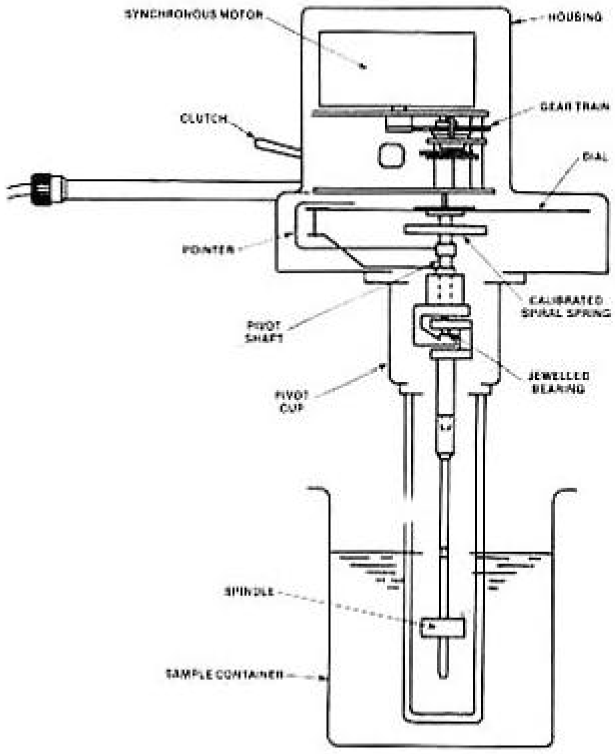


Figure 8: Brookfield viscometer

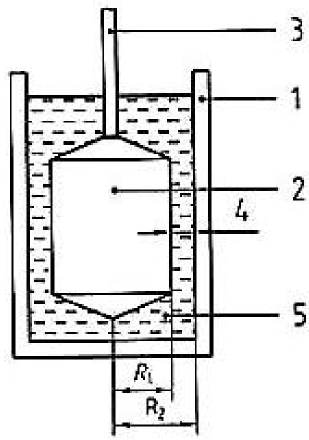


Figure 1 — Coaxial viscometer (principle)

Key

1. Beaker
2. Cylinder
3. Axle (spindle)
4. Thickness of sample being measured
5. Sample under test

Figure 9: Coaxial cylinder viscometer

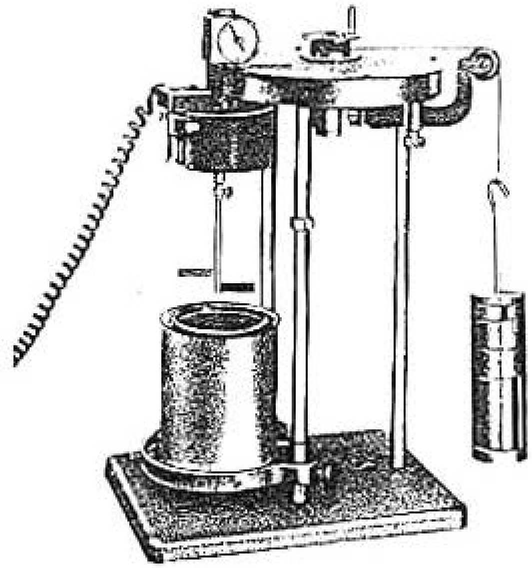


Figure 10: Stormer viscometer

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.

3.3.4 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 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 undirstubed 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.

3.3.5 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 unvulcanized rubber compounds
- Polymer and resin dispersions and emulsions

3.3.6 Feasibility of the methods to the materials for Horizontal project

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 Broookfield 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.

<u>Rotational viscometers, both at controlled shear rate and controlled shear stress, cannot be used for field tests, but they can be adopted in the scope of the Horizontal project as reference laboratory methods to carry out parallel tests for the standardisation of simple field flowability methods.</u>

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.
<u>Therefore Stormer viscometer cannot be adopted for the purpose of Horizontal project.</u>

The <u>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.</u>

3.4 "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 as 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.

3.4.1 Standards analysed

The list of standards methods examined for flow tests of different materials is reported in the following table.

Method	Material	Parameter	Apparatus	Standard range	Physical meaning/purpose	Utility / field	Comments
C 230 - 83 Flow table for Use in Tests of Hydraulic Cement	Mortars	Flow properties	Flow table and frame Mold for casting the flow specimen		Measure of diameter of the mortar after it has been spread by the operation of the table	Flow test for consistency of mortars	-
ASTM C 639 Rheological (Flow) Properties of Elastomeric Sealants	Single-component and multi-component joint sealants	Flow properties	Flow channels Refrigerator Oven	4.4 and 50 °C	Measure of the degree horizontal or vertical flow of sealants at two pre-determined temperatures	Rheological properties of single and multicomponent chemically curing sealants for use in building construction	-
ASTM C 939 - 81 Flow of Grout for Preplaced-Aggregate Concrete	Grout mixtures	Flow properties	Flow cone	Efflux time of 35 s or less	The fluidity of the concrete is measured by the time of efflux of a specified volume of grout from a standardised cone	Determination of the fluidity of grout mixtures to be used for the production of PA concrete	+
ASTM D 1084 - 63 Viscosity of Adhesives	Free-flowing adhesives	Viscosity	Met. A: consistency cup Met. B: Brookfield viscometer Met. C: Stormer viscometer Met. D: Zahn viscosity cups	50-200000 cP <3000 cP	Time for gravity flow through a calibrated orifice in a consistency cup Viscosity in cP measured with a Brookfield viscometer Time for 100 revolutions in a Stormer viscometer Time for gravity flow through a calibrated orifice in Zahn cups	For newtonian fluid For newtonian fluid For newtonian fluid	-
ASTM D 1725 - 62 Viscosity of Resin Solutions	Resin solution	Viscosity	Viscosity tubes and holder		Time for mean bubble travel	Bubble sec. is appr. equal to stokes	-
ASTM D 4318 - 84 Liquid Limit, Plastic Limit, and Plasticity Index of Soils	Soils	Water content corresponding to: Liquid limit Plastic limit Plasticity index	Liquid limit device consisting of a cup and a carriage designed to drop it onto a hard base		The liquid limit is determined by dividing the sample in the cup in two parts and then allowing to flow together by repeatedly dropping the cup. The plastic limit is determined by rolling the sample until its water content cause the thread to cramble	The method is used for engineering classification of the fine-grained fraction of the soil	-

prEN 14117 DRAFT Products and System for the Protection and Repair of Concrete Structures - Test Methods - Determination of Viscosity of Cementitious Injection Products	Cementitious injection products	Viscosity	Flow cone	21°C 60% relative humidity	The fluidity of cementitious products is measured from the time of efflux of a certain volume of the sample from a standardised cone	Determination of the technical characteristics of the product	+
prEN 13880 - 5 Hot Applied Joint Sealants - Part 5: Test Method for the Determination of Flow Resistance	Road material	Flow Resistance	Metal frame, metal plate, metal stand	60°C	The flow resistance is calculated by measuring the movement along the lower transverse edge of a sample positioned on a metal plate inclined at an angle of 75° at a test temperature of 60°C	Control of the technical properties of the product	-
UNI 20065 Petroleum Products and Lubricants Determination of Pour Point	Petroleum products and lubricants	Pour point	Test jar + cooling bath	Normal range -38 to +50°C	Lowest temperature at which movement of oil is observed	Lowest temperature at which the oil is usable	-
UNI 5656 - 65 Waterproofing of Roof - Asphalt Mastics - Determination of the Flow on a Sloping Plane	Asphalt mastics	Fluidity	Sloping plane Oven	65°C	Measure of flow of asphalt mastics placed on specimens inclined of 45° at 65°C for 8 hours	Measure of resistance of asphalt mastics to move by flow on a sloping plane at the test temperature	-
UNI 5662 - 65 Waterproofing of Roof - Asphalt Grouts - Determination of the Flow on a Sloping Plane	Asphalt grouts	Fluidity	Sloping plane Oven	65°C	Measure of flow of asphalt grouts placed on specimens inclined of 45° at 65°C for 8 hours	Measure of resistance of asphalt grouts to move by flow on a sloping plane at the test temperature	-
UNI 8997 Superplastic Mortars Determination of Consistency by a Flow Apparatus	Preblended Expansive Mortars for Grouting	Consistency	Funnel and flow channel		Consistency is expressed in cm of flow of the sample in a horizontal channel	The method is applicable to fluid products	-
UNI 9417 Fresh Concrete Classification of Consistency	Fresh Concrete	Classification of Consistency	Slump test Vebè test Compactability Flow Index		Subsidence of the cone Vebè time (s) Compactability Index Spreading in % and in mm		-
UNI EN 12350 - 3 Testing Fresh Concrete Vebè Test	Concrete	Consistency	Vebe meter (Consistometer) Mould	5 to 30 s	Measure deformation of concrete slump in static conditions. Time in seconds for a transparent disc lowered in contact with the surface of the concrete slump to be fully in contact with it under vibrating conditions. (Vebe time)	Consistency of fresh concrete to measure its workability	-

UNI EN 445 Grout for Prestressing Tendons Test Methods	Grout	Fluidity	Tube and plunger Flow cone		Method 1 (penetrometer): the fluidity is measured by the time of free penetration of a piston in the grout Method 2 (flow cone): the fluidity is measured by the time of efflux of a specified volume of grout from a standardised cone	Injection of grout in prestressed concrete constructions	+
UNI EN ISO 12115 Fibre-Reinforced Plastics Thermosetting Moulding Compounds and Prepregs Determination of Flowability, Maturation and Shelf Life	Fibre-reinforced plastics	Flowability, maturation and shelf life	Apparatus for the application of a constant load on the test tube. Hydraulic press for moulding		Method 1: Deformation of the sample under a loaded punch Method 2: Determination of hydraulic pressure for moulding at test temperature	Properties influencing the moulding process	-
UNI EN ISO 7808 - 2000 Plastics Thermosetting Moulding Materials Determination of Transfer Flow	Plastics	Fluididy	Hydraulic press Mold	165°C	The transfer fluidity is calculated by the mass of material passed through the piston during hot moulding	Production control	-
UNI ISO 4534 Vitreous and Porcelain Enamels - Determination of Fluidity Behaviour - Fusion Flow Test	Molten enamel	Fluidity	Press Flow plate and tilting frame Oven		Measure of the length and the maximum breadth of melted specimens permitted to flow at an angle of 45° for an agreed period	Fluidity behaviour of vitreous and porcelain enamels	-
ASTM C 1362 - 97 Flow of Freshly Mixed Hydraulic Cement Concrete	Concrete	Fluidity	Perforated hollow tube and Inner calibrating rod	Up to 37,5 mm in size	The concrete is allowed to flow into the hollow tube through its perforations. An inner-calibrating rod measures the height of penetrated concrete.	Laboratory and field method to test the flow and consistency of freshly mixed concrete and its change with time	-
ASTM C 143/C 143M - 00 Slump of Hydraulic - Cement Concrete	Concrete	Consistency	Mold	Up to 37,5 mm in size	The height of a cone shaped sample is measured after subsidence. The result is reported as slump test.	Laboratory and field method to test the consistency of hydraulic cement concrete	-
ASTM C 1437 - 01 Flow of Hydraulic Cement Mortar	Hydraulic cement mortar	Fluidity Consistency	Flow table Flow mold Calliper		A cone shaped sample is submitted to operation of a vibrating table The spread is measured and the consistency of the sample is expressed as percentage of the original base diameter	Determination of the water content that provides a specified flow level	-

ASTM C 1445 - 99 Measuring Consistency of Castable Refractory Using a Flow Table	Castable refractory	Fluidity Consistency	Flow table Flow mold Calliper		A cone shaped sample is submitted to operation of a vibrating table The spread is measured and the consistency of the sample is expressed as percentage of the original base diameter	Determination of the correct water amount to obtain the required consistency of refractory castable	-
ASTM D 6103 - 97 Flow Consistency of Controlled Low Strength Material (CLSM)	CLSM	Flow consistency	Flow cylinder	Up to 19 mm in size	An open-ended cylinder is filled with fresh CLSM. The cylinder is raised so the sample will flow into a patty. The average diameter is reported as the consistency of the CLSM.	Determination of the flow consistency of CSLM mixtures for use as backfill or structural fill	-
ASTM D 6351 - 99 Determination of Low Temperature Fluidity and Appearance of Hydraulic Fluids	Hydraulic fluids	Fluidity after storage at low temperature	Test jar + cooling bath		Lowest temperature at which the oil remains fluid and homogeneous after seven days	Oil ability to withstand prolonged exposure to cold temperature	-
ASTM F 1080 - 93 Determination the Consistency of Viscous Liquids Using a Consistometer	Viscous liquids	Consistency	Consistometer		The consistency is expressed as the distance of flow of the sample in a horizontal direction	Flow performance of viscous materials	-
BS EN 12350-3:2000 Testing Fresh Concrete Part 3: Vebe test	Fresh concrete	Consistency	Vebe meter (Consistometer) Mould	5-30 s	Measure of deformation of concrete slump in static conditions. Time in seconds for a transparent disc lowered in contact with the surface of the concrete slump to be fully in contact with it under vibrating conditions (Vebe time)	Consistency of fresh concrete with low/medium workability	-
ISO 2431 1993-02-15 Paints and Varnishes - Determination of Flow Time by Use of Flow Cups	Paints, varnishes and related products	Consistency	Flow cups with orifice diameters of 3, 4, 5 and 6 mm	Flow time 30 - 100 s Kinematic viscosity up to 700 cSt	Time in seconds required for the material under test to flow from the orifice of the filled cup at controlled temperature.	Determination of flow properties of Newtonian or near-Newtonian materials for quality control and development of products.	+
ISO 4103 1979 Concrete - Classification of Consistency	Concrete	Classification of Consistency	Vebe test Slump test Compactability		Vebe time Subsidence of the cone Compactability Index		-
NF P 94-052-1 November 1995 Soil: Investigation and Testing - Atterberg Limit Determination - Part 1: Liquid Limit - Cone Penetrometer Method	Soil	Liquid limit	Cone penetrometer		The liquid limit is the water content of soil to which correspond a cone penetration of 17 mm	The liquid limit characterizes the geotechnical properties of the soil	-

BS 1881: Part 102 : 1983 Testing Concrete Part 102. Method for Determination of Slump	Concrete of medium to high workability	Consistency	Mould made of metal	Suitable for concrete with slump between 5 and 175 mm and aggregates less than 40 mm	The height of a cone shaped sample is measured after subsidence. The result is reported as slump test.	Determination of cohesiveness and workability of concrete	-
BS 1881: Part 105 : 1984 Testing Concrete Part 105. Method for Determination of Flow	Concrete	Fluidity Consistency	Flow table that can be lifted manually and let to fall freely from a fixed height. Mould made of metal	Suitable for concrete with aggregates not exceeding 20 mm	The test determines the consistency of fresh concrete by measuring the spread of concrete on a flat plate which is subjected to jolting	Consistency of concrete of very high workability	-
BS EN 1015-3:1999 Methods of Test for Mortar for Masonry Part 3: Determination of Consistence of Fresh Mortar(by Flow Table)	Fresh mortar	Fluidity Consistency	Flow table with horizontal shaft and lifting cam. Mould made of metal		The test determines the consistency of fresh concrete by measuring the spread of concrete on a flat plate which is subjected to jolting	Consistency of freshly mixed mortar that gives a measure of its deformability when subjected to stress	-
BS EN 12274-3:2002 Slurry Surfacing- Test Methods Part 3: Consistency	Slurry surfacing mixtures	Consistency	Mould made of metal or hard plastic in the form of a frustum of a cone Table Measuring sheet with eight concentric circles each increasing in radius by 10 mm		The slurry mixture previously supported by the mould is allowed to flow on the table. The outflow is measured on the sheet after 10 s at four points 90° apart from the "0" circle, averaged and recorded as mm flow and % by mass of pre-wetting water	The method is used as a mix design aid to determine the amount of water required forming a stable workable mixture for roads, airfields etc.	-
BS EN 12350-5:2000 Testing Fresh Concrete Part 5:Flow Table Test	Fresh concrete	Fluidity Consistency	Flow table that can be lifted manually and let to fall freely from a fixed height. Mould made of metal	Suitable for concrete with aggregates up to 63 mm in size	The test determines the consistency of fresh concrete by measuring the spread of concrete on a flat plate which is subjected to jolting.	Consistency of freshly mixed concrete with very high workability	-
BS EN 12350-4:2000 Testing Fresh Concrete Part 4:Degree of Compactability	Fresh concrete	Compactability	Container Trowel Means of compacting the concrete	Suitable for concrete with aggregates up to 63 mm in size	The degree of compactability is determined by completely filling a container with the sample and measuring the distance from the top of the surface of the concrete compacted by vibration	Consistency of freshly mixed concrete with medium/high workability	-
NF P 18-507 Nov. 1992 Additions for Concrete-Water Retention--Method for Measurement of Fluidity by Flowing with the "Cone de	Additives (fillers or fly ash) for concrete	Flow properties % water request	Flow cone with a stem with orifice diameter of 8 mm		Determination of water request in % of solid material necessary to obtain an efflux time of 20 seconds	Determination of the fluidity of concrete mixtures	+

MARSH"							
NF P 18-358 Jul. 1985 Admixtures for Concretes, Mortars, and Grouts-Routine Grouts for Prestressing Ducts - Measurement of Fluidity and Water Reduction	Mixtures for concrete, mortar and grout	Flow properties Water reduction	Flow cone with a stem with orifice diameter of 10 mm and a length of 60 mm Sieve with openings of 3 mm		Determination of water reduction of a additivated sample with respect to a non-additivated sample having the same fluidity (the same efflux time)	Determination of the fluidity of concrete mixtures	+
Schulze von B., et al., 1991 [22]	Sintetic sludges		Tube extrusion viscometer Kasumeter		Determination of the height of the sludge suspension remaining in the container at the end of continuous flow		+
Schulze von B., et al., 1991 [19]; Spinosa and Lotito, 2003 [23],	Sintetic sludges		Tube extrusion viscometer Kasumeter		Determination of the height of the sludge suspension remaining in the container at the end of continuous flow		+
Coussot P. et al.;1995, [25]	Clay-water suspension	Yield stress	Inclined plane	35-90Pa	Determination of the final depth of the sludge remaining on the inclined plane after the end of gravity-induced flow	Fluidity of visco-plastic materials	+
Baudez J.C. et al.; 2002 [26]	Polymeric and mineral suspensions	Yield stress	Modified slump apparatus	Medium and high yield stress	Determination of the final height loss of the volume of the fluid contained in a cylindrical vessel and suddenly left flowing on a plane	Fluidity of visco-plastic materials	+

+ considered for further investigation; - considered not appropriate

Table 11: Standards examined for flow apparatus

3.4.2 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.

3.4.3 Apparatus

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

- Flow table
- Flow channels
- Flow cones (see Figure 11)
- Hollow perforated tube (Figure 12)
- Extrusion tube viscometer (Figure 13)
- Inclined plane;
- Modified slump test

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 [22]and Spinosa and Lotito, 2003 [23], (see Figure 13)

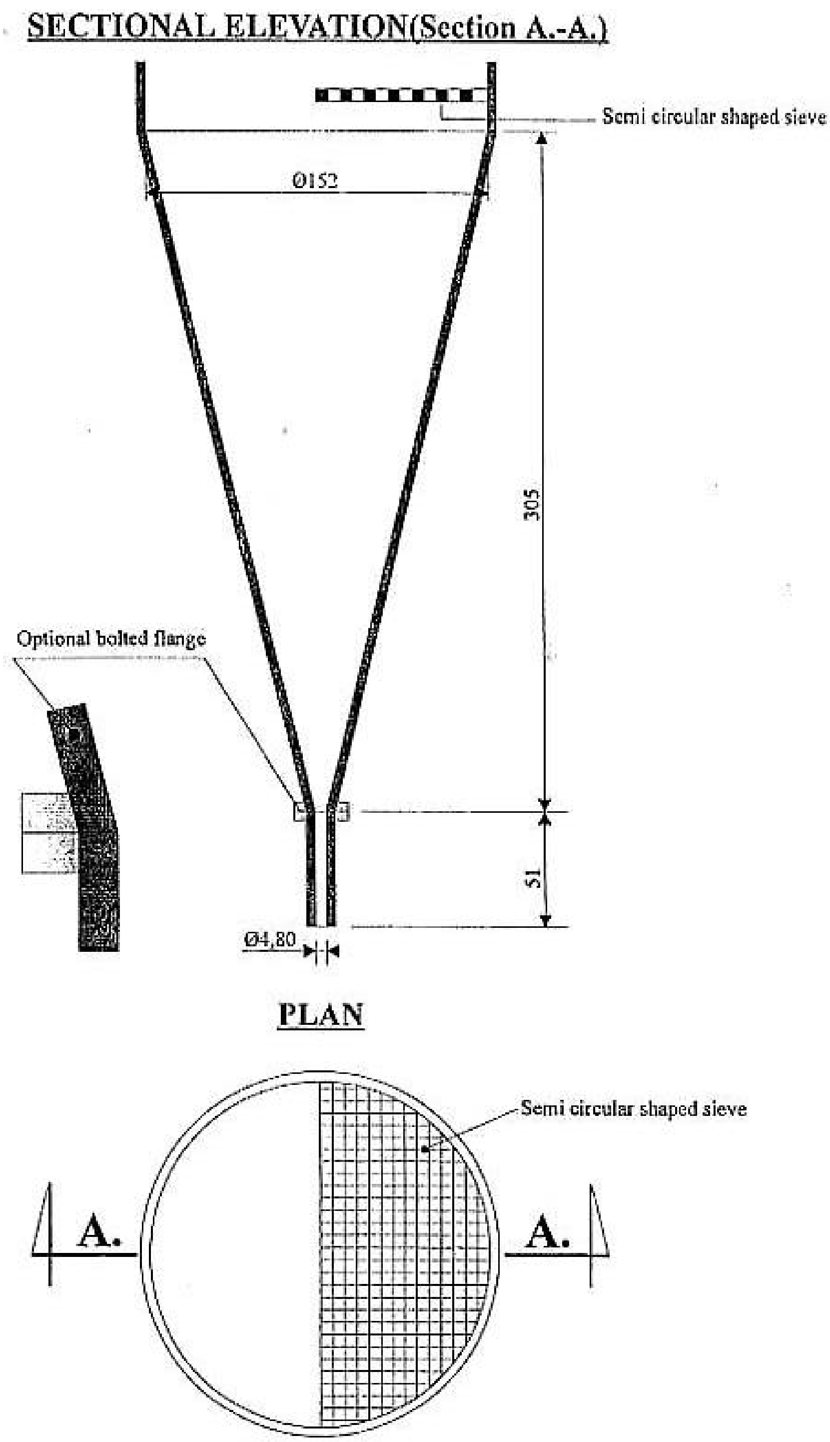


Figure 11: Flow cone (dimensions in mm)

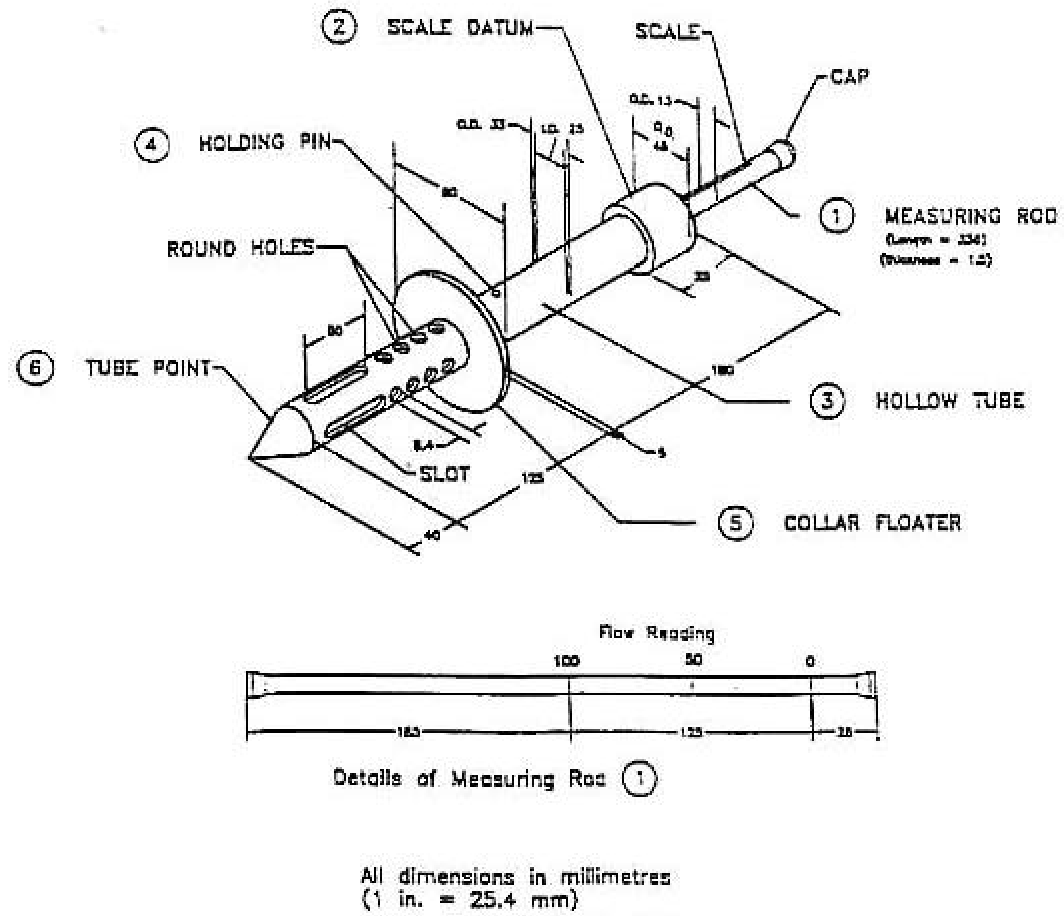


Figure 12: Hollow perforated tube for flow tests

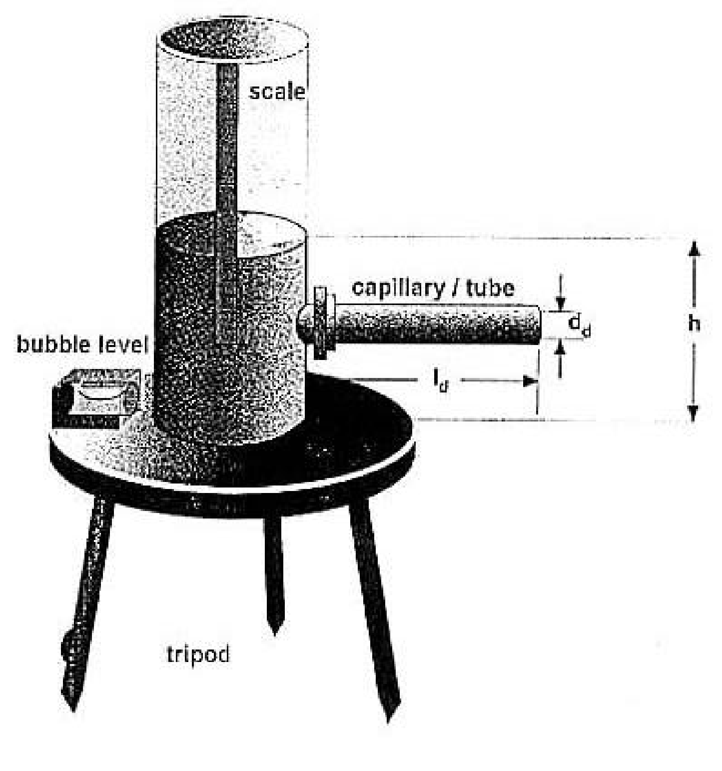


Figure 13: Tube extrusion viscometer (kasumeter)

3.4.4 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 cementitiuos 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 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 analized by putting a cylindrical hat on the top of the samples, in which several masses can be added.

3.4.5 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

3.4.6 Feasibility of the methods to the materials for Horizontal project

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 Horizontal 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 sludges, 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;
- the extrusion tube viscometer could be applied to the analysis of yield stress of non-Newtonian concentrated sludges;
- the modified slump and inclined plane tests can be used for concentrated sludges showing a pasty behaviour di 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.

4. CRITICAL POINT AND RECOMMENDATIONS

In this section mainly the methods with the expression “Considered for further investigation” in the column “Evaluation” in chapter 3 are discussed.
Standardization procedures for Horizontal material examination will consist of:

- sampling, transport, preservation and 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 sludges, 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 suitable only for Newtonian fluids like soil. The use for other Horizontal material of commercial apparatus or the best geometrical dimension must be investigated in further experimental work. Sludges and treated bio-waste can contain coarse particles that can occlude the discharge orifice, a sieving pre-treatment is necessary, flow cone apparatus has been already modified to absolve also this function using 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 sec) 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 standardized, 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 [21] showed a good correlation with laboratory results of yield stress up to 40 Pa. Further experimental work is needed to clarify the suitability for Horizontal materials.

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 to 90 Pa).

Modified slump apparatus

This is a non standardized method for measuring the yield stress of a pasty sludges 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 though 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 to determine the quantitative value of the yield stress.

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

Field test (Method 4):"inclined plane"
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,…)

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 Horizontal materials shall be accurately evaluated especially when analysing thixotropic fluids.

4.2 Summary of recommended methods

The methods selected for further investigation are reported in Table 12 and can be divided in two groups: laboratory and field methods.

Table 12 Overview of recommendations

Method	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

4.3 Research needs

4.3.1 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 Horizontal material. The field test results will be compared with laboratory ones used as reference.

4.3.2 Applicability of methods to Horizontal materials

To apply the proposed methods to Horizontal materials, 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 me optimized.
For thixotropic material (liquid-gel and gel-liquid) a vibration table pre-treatment or similar apparatus must be studied to pre-treat the sample and optimize the measure.

4.3.3 Questions to be answered (precision, repeatability, reliability, etc.)

Research must define, through interlaboratory test, 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.

4.3.4 Route, how to answer them

A protocol of validation of physical parameters will be prepared (Protocol for validation of physical parameter methods – CEN/TC308/WG1/TG3 – N29 (ref. [24] to be followed).

4.3.5 Steps to be taken

- Laboratory tests (cp. 4.3.4)
- Evaluation (cp. 4.3.4)
- 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 included evaluation
- Final draft for methods to be standardised

4.4 Contacts with CEN/TC's/WG's

4.4.1 Actions

A circulation letter (Annex 8) was sent at 31July 2003 to:

- Experts of TG3
- Secretary of WG1
- To Aldo Giove for WG2

together with the First Draft of *Physical Properties desktop study report on Flowability* and asking the comments until Wednesday, 20 August 2003 with e-mail.
All the comments received were discussed at the Hamburg meeting of CEN/TC 308 WG1 TG3 on 28.8.03 and of CEN/TC 308 WG1 on 29.8.03 to prepare the final version of the Report N° 21.

4.4.2 Comment received and incur…..

The comments received by Email are reported in the Annex 9.

4.4.3 Official meetings

WP7-Meetings
02/14/2003 in Bergamo: […]
04/04/2003 in Ancona: […]
07/03-06/2003 in Ancona: [...]
07/24-28/2003 in Bari: […]

CEN/TC308/WG1 – Meetings
03/26-28/2003 in Oslo: […]
05/22-23/2003 in Helsinki: […]

CEN/TC308/WG1/TG3 – Meetings
03/26-28/2003 in Oslo: […]
05/22-23/2003 in Helsinki: […]

5. DRAFT STANDARD (CEN TEMPLATE)

5.1 Flowability

5.1.1 Laboratory reference

5.1.1.1 Method 1

“Physical properties – Determination of flowability – Laboratory reference method by ”Laboratory rotational viscometer”

The proposal for this standard is documented in Annex 2.

5.1.2 Field test

5.1.2.1 Method 1

“Physical properties – Determination of flowability – Field test by “Flow cone apparatus”

The proposal for this standard is documented in Annex 3.

5.1.2.2 Method 2

“Physical properties – Determination of flowability – Field test by “Penetration cone apparatus”

The proposal for this standard is documented in Annex 4.

5.1.2.3 Method 3

“Physical properties – Determination of flowability – Field test by “Extrusion tube viscometer-Kasumeter apparatus”

The proposal for this standard is documented in Annex 5.

5.1.2.4 Method 4

“Physical properties – Determination of flowability – Field test by “Inclined plane”

The proposal for this standard is documented in Annex 6.

5.1.2.5 Method 5

“Physical properties – Determination of flowability – Field test by “Modified slump test”

The proposal for this standard is documented in Annex 7.

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ANNEX 1

Annex 1 is the complete list of standards and non standardized methods.
It is a common file for Report N° 21 and 22.

The file name is: ANNEX 1 Report 21 – 22.

ANNEX 2

ANNEX 3

ANNEX 4

ANNEX 5

ANNEX 6

ANNEX 7

ANNEX 8

ANNEX 9