

標題

液状化の恐れのある貨物の運送について

# ClassNK

## テクニカル インフォメーション

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各位

近年、細粒鉄鉱石やニッケル鉱石等を積載運航中に貨物が液状化したことが主な原因とみられる重大事故が報告されております。

こうした事故を避けるため、IMSBC Code が 2011 年 1 月 1 日から全船に強制適用されました。液状化する恐れのある貨物は IMSBC Code では Group A 貨物に分類され、それらの貨物の含水率が運送許容水分値(TML)以上である場合には積載が認められません。細粒鉄鉱石やニッケル鉱石等の IMSBC Code に記載されていない貨物については、荷積み港の主管庁が危険性の評価を行います。液状化の危険性があると査定された場合には Group A 貨物としての積載制限を受けます。例えば、インド政府は自国内で産出される細粒鉄鉱石を Group A 貨物と査定しています。

他方、貨物情報には貨物の水分値が TML 未満であったにもかかわらず、船積み中の激しい降雨の影響により貨物の実際の水分値が TML を超え、貨物が液状化したことが重大事故を誘発したと P&I から指摘されています。

つきましては、細粒鉄鋼石やニッケル鉱のように液状化する恐れのある貨物を船積みする際には、以下の項目について確認下さいますようお願い致します。

1. 船積みを行う前に、以下の内容について荷送人から船長へ提供される貨物情報を確認すること。(IMSBC Code 第 4 節)
  - (1) TML 及び貨物の水分値が記載されていること。
  - (2) TML を決定する水分測定が、貨物を積み込む日の 6 ヶ月以内に行われていること。
  - (3) 貨物の水分値を決定する水分測定が、貨物を積み込む日の 7 日以内に行われていること。
  - (4) 積載される貨物倉毎に貨物情報が提供されていること。(貨物の水分値が一様であることが明らかな場合を除く)
2. 貨物情報に記載された貨物の水分値が TML 未満でない限り、船長は貨物の積付を受け入れてはならない。(IMSBC Code 第 7 節 7.3.1)
3. 資料採取/試験から積載時までの間に顕著な降雨等により貨物の水分値に疑義が生じた場合には、船長は簡易試験「缶テスト」もしくは試験所での貨物水分値の再確認試験を実施すること。但し、缶テストは、TML を超えていることは示すことはできるが、TML 未満であることを保証するものではないので注意すること。試験方法については、以下を参照すること。
  - (1) 簡易試験「缶テスト」(IMSBC Code 第 8 節 8.4)
  - (2) 試験所での再確認試験 (IMSBC Code 付録 2)

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NOTES:

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4. 貨物の船積みを行う前に、貨物倉の排水設備の機能が有効であることを確認すること。
5. 出港前に貨物倉ビルジのサウンディング及び排出を行うこと。

なお、半載の貨物倉に対しては、貨物表面の荷繰りを実施するよう強く推奨します。(IMSBC Code 第5節)

なお、本件に関してご不明な点は、以下の部署にお問い合わせください。

財団法人 日本海事協会 (ClassNK)

本部 管理センター 船体部

住所: 東京都千代田区紀尾井町 4-7(郵便番号 102-8567)

Tel.: 03-5226-2017 / 2018

Fax: 03-5226-2019

E-mail: [hld@classnk.or.jp](mailto:hld@classnk.or.jp)

添付:

1. IMSBC Code 第4節
2. IMSBC Code 第5節
3. IMSBC Code 第7節
4. IMSBC Code 第8節
5. IMSBC Code 付録2

## Section 4

### Assessment of acceptability of consignments for safe shipment

#### 4.1 Identification and classification

4.1.1 Each solid bulk cargo in this Code has been assigned a Bulk Cargo Shipping Name (BCSN). When a solid bulk cargo is carried by sea it shall be identified in the transport documentation by the BCSN. The BCSN shall be supplemented with the United Nations (UN) number when the cargo is dangerous goods.

4.1.2 If waste cargoes are being transported for disposal, or for processing for disposal, the name of the cargoes shall be preceded by the word "WASTE".

4.1.3 Correct identification of a solid bulk cargo facilitates identification of the conditions necessary to safely carry the cargo and the emergency procedures, if applicable.

4.1.4 Solid bulk cargoes shall be classified, where appropriate, in accordance with the UN Manual of Tests and Criteria, part III. The various properties of a solid bulk cargo required by this Code shall be determined, as appropriate to that cargo, in accordance with the test procedures approved by a competent authority in the country of origin, when such test procedures exist. In the absence of such test procedures, those properties of a solid bulk cargo shall be determined, as appropriate to that cargo, in accordance with the test procedures prescribed in appendix 2 to this Code.

#### 4.2 Provision of information

4.2.1 The shipper shall provide the master or his representative with appropriate information on the cargo sufficiently in advance of loading to enable the precautions which may be necessary for proper stowage and safe carriage of the cargo to be put into effect.

4.2.2 Cargo information shall be confirmed in writing and by appropriate shipping documents prior to loading. The cargo information shall include:

- .1 the BCSN when the cargo is listed in this Code. Secondary names may be used in addition to the BCSN;
- .2 the cargo group (A and B, A, B or C);
- .3 the IMO Class of the cargo, if applicable;
- .4 the UN number preceded by letters UN for the cargo, if applicable;
- .5 the total quantity of the cargo offered;
- .6 the stowage factor;
- .7 the need for trimming and the trimming procedures, as necessary;

- .8 the likelihood of shifting, including angle of repose, if applicable;
- .9 additional information in the form of a certificate on the moisture content of the cargo and its transportable moisture limit in the case of a concentrate or other cargo which may liquefy;
- .10 likelihood of formation of a wet base (see subsection 7.2.3 of this Code);
- .11 toxic or flammable gases which may be generated by cargo, if applicable;
- .12 flammability, toxicity, corrosiveness and propensity to oxygen depletion of the cargo, if applicable;
- .13 self-heating properties of the cargo, and the need for trimming, if applicable;
- .14 properties on emission of flammable gases in contact with water, if applicable;
- .15 radioactive properties, if applicable; and
- .16 any other information required by national authorities.

4.2.3 Information provided by the shipper shall be accompanied by a declaration. An example of a cargo declaration form is set out in the next page. Another form may be used for cargo declaration. As an aid to paper documentation, Electronic Data Processing (EDP) or Electronic Data Interchange (EDI) techniques may be used.

**FORM FOR CARGO INFORMATION  
for Solid Bulk Cargoes**

BCSN	
Shipper	Transport document number
Consignee	Carrier
Name/means of transport	Instructions or other matters
Port/place of departure	
Port/place of destination	
General description of the cargo (Type of material/particle size)	Gross mass (kg/tonnes)
Specifications of bulk cargo, if applicable: Stowage factor: Angle of repose, if applicable: Trimming procedures: Chemical properties if potential hazard* : * e.g., Class & UN No. or "MHB"	
Group of the cargo  <input type="checkbox"/> Group A and B* <input type="checkbox"/> Group A* <input type="checkbox"/> Group B <input type="checkbox"/> Group C * For cargoes which may liquefy (Group A and Group A and B cargoes)	Transportable moisture limit  Moisture content at shipment
Relevant special properties of the cargo (e.g., highly soluble in water)	Additional certificate(s)* <input type="checkbox"/> Certificate of moisture content and transportable moisture limit <input type="checkbox"/> Weathering certificate <input type="checkbox"/> Exemption certificate <input type="checkbox"/> Other (specify) * If required
<b>DECLARATION</b> I hereby declare that the consignment is fully and accurately described and that the given test results and other specifications are correct to the best of my knowledge and belief and can be considered as representative for the cargo to be loaded.	Name/status, company/organization of signatory  Place and date  Signature on behalf of shipper

### **4.3 Certificates of test**

4.3.1 To obtain the information required in 4.2.1 the shipper shall arrange for the cargo to be properly sampled and tested. The shipper shall provide the ship's master or his representative with the appropriate certificates of test, if required in this Code.

4.3.2 When a concentrate or other cargo which may liquefy is carried, the shipper shall provide the ship's master or his representative with a signed certificate of the TML, and a signed certificate or declaration of the moisture content. The certificate of TML shall contain, or be accompanied by the result of the test for determining the TML. The declaration of moisture content shall contain, or be accompanied by, a statement by the shipper that the moisture content is, to the best of his knowledge and belief, the average moisture content of the cargo at the time the declaration is presented to the master.

4.3.3 When a concentrate or other cargo which may liquefy is to be loaded into more than one cargo space of a ship, the certificate or the declaration of moisture content shall certify the moisture content of each type of finely grained material loaded into each cargo space. Notwithstanding this requirement, if sampling according to internationally or nationally accepted standard procedures indicates that the moisture content is uniform throughout the consignment, then one certificate or declaration of average moisture content for all cargo spaces is acceptable.

4.3.4 Where certification is required by the individual schedules for cargoes possessing chemical hazards, the certificate shall contain, or be accompanied by, a statement from the shipper that the chemical characteristics of the cargo are, to the best of his knowledge, those present at the time of the ship's loading.

### **4.4 Sampling procedures**

4.4.1 Physical property tests on the consignment are meaningless unless they are conducted prior to loading on truly representative test samples.

4.4.2 Sampling shall be conducted only by persons who have been suitably trained in sampling procedures and who are under the supervision of someone who is fully aware of the properties of the consignment and also the applicable principles and practices of sampling.

4.4.3 Prior to taking samples, and within the limits of practicability, a visual inspection of the consignment which is to form the ship's cargo shall be carried out. Any substantial portions of material which appear to be contaminated or significantly different in characteristics or moisture content from the bulk of the consignment shall be sampled and analysed separately. Depending upon the results obtained in these tests, it may be necessary to reject those particular portions as unfit for shipment.

4.4.4 Representative samples shall be obtained by employing techniques which take the following factors into account:

- .1 the type of material;
- .2 the particle size distribution;

- .3 composition of the material and its variability;
- .4 the manner in which the material is stored, in stockpiles, rail wagons or other containers, and transferred or loaded by material-handling systems such as conveyors, loading chutes, crane grabs, etc.;
- .5 the chemical hazards (toxicity, corrosivity, etc.);
- .6 the characteristics which have to be determined: moisture content, TML, bulk density/stowage factor, angle of repose, etc.;
- .7 variations in moisture distribution throughout the consignment which may occur due to weather conditions, natural drainage, e.g., to lower levels of stockpiles or containers, or other forms of moisture migration; and
- .8 variations which may occur following freezing of the material.

4.4.5 Throughout the sampling procedures, utmost care shall be taken to prevent changes in quality and characteristics. Samples shall be immediately placed in suitable sealed containers which are properly marked.

4.4.6 Unless expressly provided otherwise, sampling for the test required by this Code shall follow an internationally or nationally accepted standard procedure.

#### **4.5 Interval between sampling/testing and loading for TML and moisture content determination**

4.5.1 A test to determine the TML of a solid bulk cargo shall be conducted within six months to the date of loading the cargo. Notwithstanding this provision, where the composition or characteristics of the cargo are variable for any reason, a test to determine the TML shall be conducted again after it is reasonably assumed that such variation has taken place.

4.5.2 Sampling and testing for moisture content shall be conducted as near as practicable to the time of loading. If there has been significant rain or snow between the time of testing and loading, check tests shall be conducted to ensure that the moisture content of the cargo is still less than its TML. The interval between sampling/testing and loading shall never be more than seven days.

4.5.3 Samples of frozen cargo shall be tested for the TML or the moisture content after the free moisture has completely thawed.

#### **4.6 Sampling procedures for concentrate stockpiles**

4.6.1 It is not practicable to specify a single method of sampling for all consignments since the character of the material and the form in which it is available will affect the selection of the procedure to be used. In the absence of internationally or nationally accepted standard sampling procedures, the following sampling procedures for concentrate stockpiles may be used to determine the moisture content and the TML of mineral concentrates. These procedures are not intended to replace sampling procedures, such as the use of automatic sampling, that achieve equal or superior accuracy of either moisture content or TML.

4.6.2 Sub-samples are taken in a reasonably uniform pattern, where possible from a levelled stockpile.

4.6.3 A plan of the stockpile is drawn and divided into areas, each of which contains approximately 125 t, 250 t or 500 t depending on the amount of concentrate to be shipped. Such a plan will indicate the number of sub-samples required and where each is to be taken. Each sub-sample taken is drawn from approximately 50 cm below the surface of the designated area.

4.6.4 The number of sub-samples and sample size are given by the competent authority or determined in accordance with the following scale:

Consignments of not more than 15,000 t:

One 200 g sub-sample is taken for each 125 t to be shipped.

Consignments of more than 15,000 but not more than 60,000 t:

One 200 g sub-sample is taken for each 250 t to be shipped.

Consignments of more than 60,000 t:

One 200 g sub-sample is taken for each 500 t to be shipped.

4.6.5 Sub-samples for moisture content determination are placed in sealed containers (such as plastic bags, cans or small metallic drums) immediately on withdrawal for conveyance to the testing laboratory, where they are thoroughly mixed in order to obtain a fully representative sample. Where testing facilities are not available at the testing site, such mixing is done under controlled conditions at the stockpile and the representative sample placed in a sealed container and shipped to the test laboratory.

4.6.6 Basic procedural steps include:

- .1 identification of consignment to be sampled;
- .2 determination of the number of individual sub-samples and representative samples, as described in 4.6.4, which are required;
- .3 determination of the positions from which to obtain sub-samples and the method of combining such sub-samples to arrive at a representative sample;
- .4 gathering of individual sub-samples and placing them in sealed containers;
- .5 thorough mixing of sub-samples to obtain the representative sample; and
- .6 placing the representative sample in a sealed container if it has to be shipped to a test laboratory.



#### 4.7 Examples of standardized sampling procedures, for information

- ISO 3082: 1998 - Iron ores – Sampling and sample preparation procedures
  - ISO 1988: 1975 - Hard coal – Sampling
  - ASTMD 2234-99 - Standard Practice for Collection of a Gross Sample of Coal
- Australian Standards
- AS 4264.1 - Coal and Coke-Sampling
    - Part 1: Higher rank coal – Sampling Procedures
  - AS 1141 – Series - Methods of sampling and testing aggregates
  - BS.1017:1989 - Methods of sampling coal and coke
  - BS 1017 - British Standard Part 1: 1989 methods of sampling of coal
  - BS 1017 - British Standard Part 2: 1994 methods of sampling of coal

Canadian Standard Sampling Procedure for Concentrate Stockpiles

European Communities Method of Sampling for the Control of Fertilizers

- JIS M 8100 - Japanese General Rules for Methods of Sampling Bulk Materials
- JIS M 8100: 1992 - Particulate cargoes – General Rules for Methods of Sampling

Polish Standard Sampling Procedure for:

Iron and Manganese Ores – Ref. No. PN-67/H-04000

Non-ferrous Metals – Ref. No. PN-70/H-04900

Russian Federation Standard Sampling Procedure for the Determination of Moisture Content in Ore Concentrates.

#### 4.8 Documentation required on board the ship carrying dangerous goods

4.8.1 Each ship carrying dangerous goods in solid form in bulk shall have a special list or manifest setting forth the dangerous goods on board and the location thereof, in accordance with SOLAS regulation VII/7-2.2. A detailed stowage plan, which identifies by class and sets out the location of all dangerous goods on board, may be used in place of such a special list or manifest.

4.8.2 When dangerous goods in solid form in bulk are carried appropriate instructions on emergency response to incidents involving the cargoes shall be on board.

4.8.3 Cargo ships of 500 gross tonnage and over constructed on or after 1 September 1984 and cargo ships of less than 500 gross tonnage constructed on or after 1 February 1992, subject to SOLAS regulation II-2/19.4 (or II-2/54.3), shall have a Document of compliance when carrying dangerous goods in solid form in bulk except class 6.2 and class 7.

## Section 5

### Trimming procedures

#### 5.1 General provisions for trimming

5.1.1 Trimming a cargo reduces the likelihood of the cargo shifting and minimizes the air entering the cargo. Air entering the cargo could lead to spontaneous heating. To minimize these risks, cargoes shall be trimmed reasonably level, as necessary.

5.1.2 Cargo spaces shall be as full as practicable without resulting in excessive loading on the bottom structure or 'tween-deck to prevent sliding of a solid bulk cargo. Due consideration shall be given to the amount of a solid bulk cargo in each cargo space, taking into account the possibility of shifting and longitudinal moments and forces of the ship. Cargo shall be spread as widely as practicable to the boundary of the cargo space. Alternate hold loading restrictions, as required by SOLAS chapter XII, may also need to be taken into account.

5.1.3 The master has the right to require that the cargo be trimmed level, where there is any concern regarding stability based upon the information available, taking into account the characteristics of the ship and the intended voyage.

#### 5.2 Special provisions for multi-deck ships

5.2.1 When a solid bulk cargo is loaded only in lower cargo spaces, it shall be trimmed sufficiently to equalize the mass distribution on the bottom structure.

5.2.2 When solid bulk cargoes are carried in 'tween-decks, the hatchways of such 'tween-decks shall be closed in those cases where the loading information indicates an unacceptable level of stress of the bottom structure if the hatchways are left open. The cargo shall be trimmed reasonably level and shall either extend from side to side or be secured by additional longitudinal divisions of sufficient strength. The safe load-carrying capacity of the 'tween-decks shall be observed to ensure that the deck structure is not overloaded.

5.2.3 If coal cargoes are carried in 'tween decks, the hatchways of such 'tween-decks shall be tightly sealed to prevent air moving up through the body of the cargo in the 'tween decks.

#### 5.3 Special provisions for cohesive bulk cargoes

5.3.1 All damp cargoes and some dry ones possess cohesion. For cohesive cargoes, the general provisions in subsection 5.1 shall apply.

5.3.2 The angle of repose is not an indicator of the stability of a cohesive bulk cargo and it is not included in the individual schedules for cohesive cargoes.

#### 5.4 Special provisions for non-cohesive bulk cargoes

5.4.1 Non-cohesive bulk cargoes are those listed in paragraph 1 in appendix 3 and any other cargo not listed in the appendix, exhibiting the properties of a non-cohesive material.

5.4.2 For trimming purposes, solid bulk cargoes can be categorized as cohesive or non-cohesive. The angle of repose is a characteristic of non-cohesive bulk cargoes which is indicative of cargo stability and has been included in the individual schedules for non-cohesive cargoes. The angle of repose of the cargoes shall establish which provisions of this section apply. Methods for determining the angle of repose are given in section 6.

#### **5.4.3 Non-cohesive bulk cargoes having an angle of repose less than or equal to 30°**

These cargoes, which flow freely like grain, shall be carried according to the provisions applicable to the stowage of grain cargoes\*. The bulk density of the cargo shall be taken into account when determining:

- .1 the scantlings and securing arrangements of divisions and bin bulkheads; and
- .2 the stability effect of free cargo surfaces.

#### **5.4.4 Non-cohesive bulk cargoes having an angle of repose greater than 30° to 35° inclusive**

These cargoes shall be trimmed according to the following criteria:

- .1 the unevenness of the cargo surface measured as the vertical distance ( $\Delta h$ ) between the highest and lowest levels of the cargo surface shall not exceed  $B/10$ , where  $B$  is the beam of the ship in metres, with a maximum allowable  $\Delta h = 1.5$  m; or
- .2 loading is carried out using trimming equipment approved by the competent authority.

#### **5.4.5 Non-cohesive bulk cargoes having an angle of repose greater than 35°**

These cargoes shall be trimmed according to the following criteria:

- .1 the unevenness of the cargo surface measured as the vertical distance ( $\Delta h$ ) between the highest and lowest levels of the cargo surface shall not exceed  $B/10$ , where  $B$  is the beam of the ship in metres, with a maximum allowable  $\Delta h = 2$  m; or
- .2 loading is carried out using trimming equipment approved by the competent authority.

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\* Reference is made to chapter VI of the SOLAS Convention, and the International Code for the Safe Carriage of Grain in Bulk adopted by the Maritime Safety Committee of the Organization by resolution MSC.23(59).

## Section 7

### Cargoes which may liquefy

#### 7.1 Introduction

7.1.1 The purpose of this section is to bring to the attention of masters and others with responsibilities for the loading and carriage of bulk cargoes, the risks associated with liquefaction and the precautions to minimize the risk. Such cargoes may appear to be in a relatively dry granular state when loaded, and yet may contain sufficient moisture to become fluid under the stimulus of compaction and the vibration which occurs during a voyage.

7.1.2 A ship's motion may cause a cargo to shift sufficiently to capsize the vessel. Cargo shift can be divided into two types, namely, sliding failure or liquefaction consequence. Trimming the cargo in accordance with section 5 can prevent sliding failure.

7.1.3 Some cargoes which may liquefy may also heat spontaneously.

#### 7.2 Conditions for hazards

7.2.1 Group A cargoes contain a certain proportion of small particles and a certain amount of moisture. Group A cargoes may liquefy during a voyage even when they are cohesive and trimmed level. Liquefaction can result in cargo shift. This phenomenon may be described as follows:

- .1 the volume of the spaces between the particles reduces as the cargo is compacted owing to the ship motion, etc.;
- .2 the reduction in space between cargo particles causes an increase in water pressure in the space; and
- .3 the increase in water pressure reduces the friction between cargo particles resulting in a reduction in the shear strength of the cargo.

7.2.2 Liquefaction does not occur when one of the following conditions is satisfied:

- .1 the cargo contains very small particles. In this case particle movement is restricted by cohesion and the water pressure in spaces between cargo particles does not increase;
- .2 the cargo consists of large particles or lumps. Water passes through the spaces between the particles and there is no increase in the water pressure. Cargoes which consist entirely of large particles will not liquefy;
- .3 the cargo contains a high percentage of air and low moisture content. Any increase in the water pressure is inhibited. Dry cargoes are not liable to liquefy.

7.2.3 A cargo shift caused by liquefaction may occur when the moisture content exceeds the TML. Some cargoes are susceptible to moisture migration and may develop a dangerous wet base even if the average moisture content is less than the TML. Although the cargo surface may appear dry, undetected liquefaction may take place resulting in shifting of the cargo. Cargoes with high moisture content are prone to sliding, particularly when the cargo is shallow and subject to large heel angles.

7.2.4 In the resulting viscous fluid state cargo may flow to one side of the ship with a roll but not completely return with a roll the other way. Consequently the ship may progressively reach a dangerous heel and capsize quite suddenly.

### **7.3 Provisions for cargoes which may liquefy**

#### **7.3.1 General**

7.3.1.1 Concentrates or other cargoes which may liquefy shall only be accepted for loading when the actual moisture content of the cargo is less than its TML. Notwithstanding this provision, such cargoes may be accepted for loading on specially constructed or fitted cargo ships even when their moisture content exceeds the TML.

7.3.1.2 Cargoes which contain liquids other than packaged canned goods or the like shall not be stowed in the same cargo space above or adjacent to these solid bulk cargoes.

7.3.1.3 Adequate measures shall be taken to prevent liquids entering the cargo space in which these solid bulk cargoes are stowed during the voyage.

7.3.1.4 Masters shall be cautioned about the possible danger of using water to cool these cargoes while the ship is at sea. Introducing water may bring the moisture content of these cargoes to a flow state. When necessary, due regard shall be paid to apply water in the form of a spray.

#### **7.3.2 Specially constructed or fitted cargo ships**

7.3.2.1 Cargoes having a moisture content in excess of the TML shall only be carried in specially constructed cargo ships or in specially fitted cargo ships.

7.3.2.2 Specially constructed cargo ships shall have permanent structural boundaries, so arranged as to confine any shift of cargo to an acceptable limit. The ship concerned shall carry evidence of approval by the Administration.

7.3.2.3 Specially fitted cargo ships shall be fitted with specially designed portable divisions to confine any shift of cargo to an acceptable limit. Specially fitted cargo ships shall be in compliance with the following requirements:

- .1 The design and positioning of such special arrangements shall adequately provide not only the restraint of the immense forces generated by the flow movement of high-density bulk cargoes, but also for the need to reduce to an acceptable safe level the potential heeling movements arising out of a transverse cargo flow across the cargo space. Divisions provided to meet these requirements shall not be constructed of wood.

- .2 The elements of the ship's structure bounding such cargo shall be strengthened, as necessary.
- .3 The plan of special arrangements and details of the stability conditions on which the design has been based shall have been approved by the Administration. The ship concerned shall carry evidence of approval by the Administration.

7.3.2.4 A submission made to an Administration for approval of such a ship shall include:

- .1 relevant structural drawings, including scaled longitudinal and transverse sections;
- .2 stability calculations, taking into account loading arrangements and possible cargo shift, showing the distribution of cargo and liquids in tanks, and of cargo which may become fluid; and
- .3 any other information which may assist the Administration in the assessment of the submission.

## **Section 8**

### **Test procedures for cargoes which may liquefy**

#### **8.1 General**

For a Group A cargo, the actual moisture content and transportable moisture limit shall be determined in accordance with a procedure determined by the appropriate authority as required by section 4.1.4 of this Code, unless the cargo is carried in a specially constructed or fitted ship.

#### **8.2 Test procedures for measurement of moisture content**

There are recognized international and national methods for determining moisture content for various materials. Reference is made to paragraph 1.1.4.4 of appendix 2.

#### **8.3 Methods for determining transportable moisture limit**

The recommended methods for determining transportable moisture limit are given in appendix 2.

#### **8.4 Complementary test procedure for determining the possibility of liquefaction**

A ship's master may carry out a check test for approximately determining the possibility of flow on board ship or at the dockside by the following auxiliary method:

Half fill a cylindrical can or similar container (0.5 to 1 litre capacity) with a sample of the material. Take the can in one hand and bring it down sharply to strike a hard surface such as a solid table from a height of about 0.2 m. Repeat the procedure 25 times at one- or two-second intervals. Examine the surface for free moisture or fluid conditions. If free moisture or a fluid condition appears, arrangements should be made to have additional laboratory tests conducted on the material before it is accepted for loading.

## APPENDIX 2

### LABORATORY TEST PROCEDURES, ASSOCIATED APPARATUS AND STANDARDS

#### 1 Test procedures for materials which may liquefy and associated apparatus

Three methods of testing for the transportable moisture limit are currently in general use:

- .1 flow table test;
- .2 penetration test;
- .3 Proctor/Fagerberg test.

As each method has its advantages, the selection of the test method should be determined by local practices or by the appropriate authorities.

##### 1.1 *Flow table test procedure*

###### 1.1.1 *Scope*

The flow table is generally suitable for mineral concentrates or other fine material with a maximum grain size of 1 mm. It may also be applicable to materials with a maximum grain size up to 7 mm. It will not be suitable for materials coarser than this and may also not give satisfactory results for some materials with high clay content. If the flow table test is not suitable for the material in question, the procedures to be adopted should be those approved by the authority of the port State.

The test described below provides for determination of:

- .1 the moisture content of a sample of cargo, hereinafter referred to as the test material;
- .2 the flow moisture point (FMP) of the test material under impact or cyclic forces of the flow table apparatus; and
- .3 the transportable moisture limit of the test material.



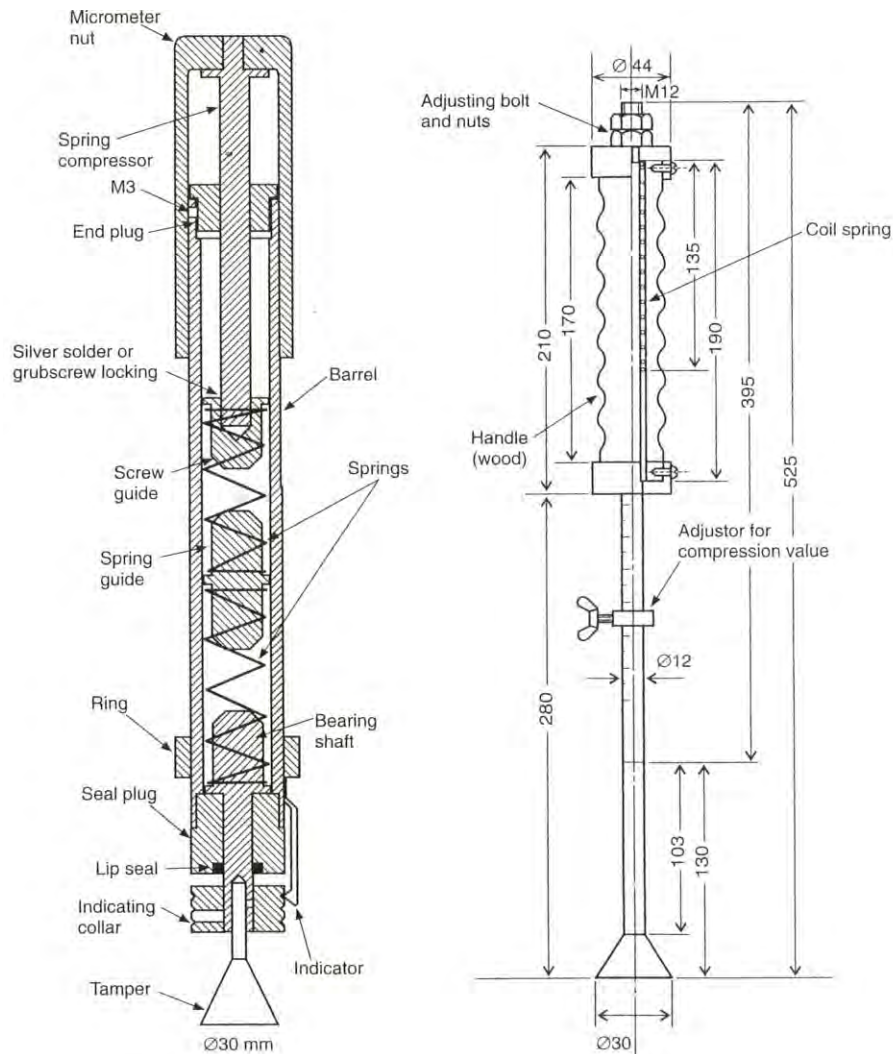
1.1.2 *Apparatus (see figure 1.1.2)*

- .1 Standard flow table and frame (ASTM Designation (C230-68) – see 3).



**Figure 1.1.2 – Flow table and accessory apparatus**

- .2 Flow table mounting (ASTM Designation (C230-68) – see 3).
- .3 Mould (ASTM Designation (C230-68) – see 3).
- .4 Tamper (see figure 1.1.2.4): the required tamping pressure may be achieved by using calibrated, spring-loaded tampers (examples are included in figure 1.1.2.4) or some other suitable design of tamper that allows a controlled pressure to be applied via a 30 mm diameter tamper head.
- .5 Scales and weights (ASTM Designation (C109-73) – see 3) and suitable sample containers.
- .6 Glass graduated measuring cylinder and burette having capacities of 100-200 ml and 10 ml, respectively.
- .7 A hemispherical mixing bowl approximately 30 cm diameter, rubber gloves and drying dishes or pans. Alternatively, an automatic mixer of similar capacity can be used for the mixing operations. In this case, care should be exercised to ensure that the use of such a mechanical mixer does not reduce the particle size or consistency of the test material.
- .8 A drying oven with controlled temperature up to approximately 110°C. This oven should be without air circulation.



**Figure 1.1.2.4 – Examples of spring-loaded tampers**

### 1.1.3 Temperature and humidity

It is preferable to work in a room where the samples will be protected from excessive temperatures, air currents and humidity variations. All phases of the material preparation and testing procedure should be accomplished in a reasonable space of time to minimize moisture losses and, in any event, within the day of commencement. Where possible, sample containers should be covered with plastic film or other suitable cover.

### 1.1.4 Procedure

The quantity of material required for a flow moisture test will vary according to the specific gravity of the material to be tested. It will range from approximately 2 kg for coal to 3 kg for mineral concentrates. It should be collected as a representative sample of the cargo being shipped. Experience has shown that more accurate test results will be obtained by ensuring that the moisture content of the test sample is increased rather than decreased towards the FMP.

Consequently, it is recommended that a preliminary flow moisture test should be conducted, generally in accordance with the following, to indicate the condition of the test sample, i.e. the quantity of water and the rate at which it is to be added or whether the sample should be air-dried to reduce its moisture content before commencing the main flow moisture test.

#### 1.1.4.1 Preparation of the test sample

The representative sample of test material is placed in the mixing bowl and thoroughly mixed. Three subsamples (A), (B) and (C) are removed from the mixing bowl as follows: about one fifth of the sample (A) should be immediately weighed and placed in the drying oven to determine the moisture content of the sample “as received”. Two further subsamples, each of about two fifths of the gross weight, should then be taken, one (B) for the preliminary FMP test and the other (C) for the main FMP determination:

- .1 *Filling the mould.* The mould is placed on the centre of the flow table and filled in three stages with the material from the mixing bowl. The first charge, after tamping, should aim to fill the mould to approximately one third of its depth. The quantity of sample required to achieve this will vary from one material to another, but can readily be established after some experience has been gained of the packing characteristics of the material being tested.

The second charge, after tamping, should fill the mould to about two thirds of its depth and the third and final charge, after tamping, should reach to just below the top of the mould (see figure 1.1.4.2).

- .2 *Tamping procedure.* The aim of tamping is to attain a degree of compaction similar to that prevailing at the bottom of a shipboard cargo of the material being tested. The correct pressure to be applied is calculated from:

$$\text{Tamping pressure (Pa)} = \begin{array}{l} \text{Bulk density of cargo (kg/m}^3\text{)} \\ \times \text{Maximum depth of cargo (m)} \\ \times \text{Gravity acceleration (m/s}^2\text{)} \end{array}$$

Bulk density can be measured by a single test, using the Proctor C apparatus described in ASTM Standard D-698 or JIS-A-1210, on a sample of the cargo at the proposed moisture content of loading.

When calculating the tamping pressure, if no information concerning cargo depth is available the maximum likely depth should be used.

Alternatively, the pressure may be estimated from table 1.1.4.1.

The number of tamping actions (applying the correct, steady pressure each time) should be about 35 for the bottom layer, 25 for the middle and 20 for the top layer, tamping successively over the area completely to the edges of the sample to achieve a uniformly flat surface for each layer.

- .3 *Removal of the mould.* The mould is tapped on its side until it becomes loose, leaving the sample in the shape of a truncated cone on the table.

**Table 1.1.4.1**

Typical cargo	Bulk density (kg/m <sup>3</sup> )	Maximum cargo depth			
		2 m	5 m	10 m	20 m
		← Tamper pressure (kPa) →			
Coal	1000	20 (1.4)	50 (3.5)	100 (7.1)	200 (14.1)
	2000	40 (2.8)	100 (7.1)	200 (14.1)	400 (28.3)
Metal ore	3000	60 (4.2)	150 (10.6)	300 (21.2)	600 (42.4)
	4000	80 (5.7)	200 (14.1)	400 (28.3)	800 (56.5)
Lead ore conc.	5000	100 (7.1)	250 (17.7)	500 (35.3)	1000 (70.7)

(values in parenthesis are equivalent kgf when applied via a 30 mm diameter tamper head)

1.1.4.2 The preliminary flow moisture test:

- .1 Immediately after removing the mould, the flow table is raised and dropped up to 50 times through a height of 12.5 mm at a rate of 25 times per minute. If the material is below the FMP, it usually crumbles and bumps off in fragments with successive drops of the table (see figure 1.1.4-3).
- .2 At this stage, the flow table is stopped and the material returned to the mixing bowl, where 5-10 ml of water, or possibly more, is sprinkled over the surface and thoroughly mixed into the material, either with rubber-gloved fingers or an automatic mixer.

The mould is again filled and the flow table is operated as described in 1.1.4.2.1 for up to 50 drops. If a flow state is not developed, the process is repeated with further additions of water until a flow state has been reached.

- .3 *Identification of a flow state.* The impacting action of the flow table causes the grains to rearrange themselves to produce compaction of the mass. As a result, the fixed volume of moisture contained in the material at any given level increases as a percentage of the total volume. A flow state is considered to have been reached when the moisture content and compaction of the sample produce a level of saturation such that plastic deformation occurs\*. At this stage, the moulded sides of the sample may deform, giving a convex or concave profile (see figure 1.1.4-4).

\* In certain conditions, the diameter of the cone may increase before the flow moisture point is reached, due to low friction between the grains rather than to plastic flow. This must not be mistaken for a flow state.

With repeated action of the flow table, the sample continues to slump and to flow outwards. In certain materials, cracks may also develop on the top surface. Cracking, with the appearance of free moisture, is not, however, an indication of development of a flow state. In most cases, measurement of the deformation is helpful in deciding whether or not plastic flow has occurred. A template which, for example, will indicate an increase in diameter of up to 3 mm in any part of the cone is a useful guide for this purpose. Some additional observations may be useful. For example: when the (increasing) moisture content is approaching the FMP, the sample cone begins to show a tendency to stick to the mould. Further, when the sample is pushed off the table, the sample may leave tracks (stripes) of moisture on the table. If such stripes are seen, the moisture content may be above the FMP: the absence of tracks (stripes) is not necessarily an indication of being below the FMP.

Measuring the diameter of the cone, at the base or at half height, will always be useful. By addition of water in increments of 0.4% to 0.5% and applying 25 drops of the flow table, the first diameter increase will generally be between 1 and 5 mm and after a further increment of water the base diameter will have expanded by between 5 and 10 mm.

- .4 As an alternative to the procedure described above, for many concentrates a fast way of finding the approximate FMP is as follows:

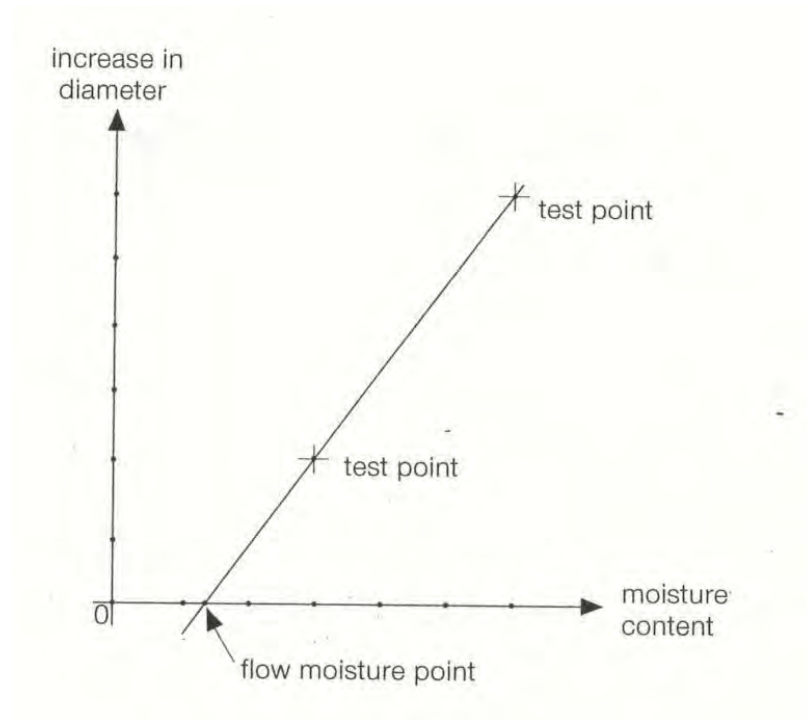
When the moisture content is definitely beyond the FMP, measure the diameter after 25 drops, repeat the test after adding a further increment of water, measure the diameter and draw a diagram as illustrated in figure 1.1.4-1, showing increase in diameter plotted against moisture content. A straight line drawn through the two points will cross the moisture content axis close to the FMP.

Having completed the preliminary FMP test, the sample for the main test is adjusted to the required level of moisture content (about 1% to 2%) below the flow point.

#### 1.1.4.3 Main flow moisture test

When a flow state has been reached in the preliminary test, the moisture content of sub-sample (C) is adjusted to about 1% to 2% less than the last value which did not cause flow in the preliminary test (this is suggested simply to avoid starting the main test too close to the FMP and then having to waste time air-drying it and starting again). The final test is then carried out on this adjusted sample in the same manner as for the preliminary test, but in this case with the addition of water in increments of no more than 0.5% of the mass of the test material (the lower the "preliminary" FMP, the smaller the increments should be). After each stage, the whole moulded sample should be placed in a container, weighed immediately and retained for moisture determination if required. This will be necessary if the sample flowed or if the next, slightly wetter, sample flows. If not required it may be returned to the mixing bowl.

When a flow state has been reached, the moisture content should be determined on two samples, one with moisture content just above the FMP and the other with moisture content just below the FMP. The difference between the two values should then be 0.5% or less, and the FMP is taken as the mean of these two values.



**Figure 1.1.4-1**



**Figure 1.1.4-2**



**Figure 1.1.4-3**



**Figure 1.1.4-4**

#### 1.1.4.4 Determination of moisture content

##### *Introduction*

It should be noted that, for many materials, there are recognized international and national methods for determining moisture content. These methods, or ones that have been established to give equivalent results, should be followed.

### Concentrates and similar materials

It is clearly important that the samples should be dried to a constant mass. In practice, this is ascertained after a suitable drying period at 105°C by weighing the sample successively with an interval of several hours elapsing. If the mass remains constant, drying has been completed, whereas if the mass is still decreasing, drying should be continued.

The length of the drying period depends upon many variables, such as the disposition of the material in the oven, the type of container used, the particle size, the rate of heat transfer, etc. It may be that a period of five hours is ample for one concentrate sample, whereas it is not sufficient for another. Sulphide concentrates tend to oxidize, and therefore the use of drying ovens with air circulation systems is not recommended for these materials, nor should the test sample be left in the drying oven for more than four hours.

### Coal

The recommended methods for determination of the moisture content are those described in ISO 589-1974, "Hard Coal – Determination of Total Moisture". This method, or ones that have been established to give equivalent results, should be followed.

*Calculation of moisture content, FMP and transportable moisture limit:*

Taking  $m_1$  as the exact mass of the subsample "as received" (see 1.1.4.1),

Taking  $m_2$  as the exact mass of the "as received" subsample, after drying,

Taking  $m_3$  as the exact mass of the sample just above the flow state (see 1.1.4.3),

Taking  $m_4$  as the exact mass of the sample just above the flow state, after drying,

Taking  $m_5$  as the exact mass of the sample just below the flow state (see 1.1.4.3),

Taking  $m_6$  as the exact mass of the sample just below the flow state, after drying,

Then:

- .1 The moisture content of the concentrate "as received" is:

$$\frac{(m_1 - m_2)}{m_1} \times 100, \text{ in per cent} \quad (1.1.4.4.1)$$

- .2 The FMP of the material is:

$$\frac{\frac{(m_3 - m_4)}{m_3} + \frac{m_5 - m_6}{m_5}}{2} \times 100, \text{ in per cent} \quad (1.1.4.4.2)$$

- .3 The transportable moisture limit of the material is 90% of the FMP.



## **Peat Moss**

For all Peat Moss, determine the bulk density, using either the ASTM or CEN (20 litres) method.

Peat should be above or below 90kg/cubic metre on a dry weight basis in order to obtain the correct TML.

As indicated in 1.1.1, the following should be determined:

- .1 the moisture content of a sample of cargo (MC);
- .2 the flow moisture point (FMP);
- .3 the transportable moisture limit (TML). The TML will be determined as follows:
  - .3.1 for peat with a bulk density of greater than 90 kg/cubic metre on a dry weight is 85% of the FMP; and
  - .3.2 for peat with a bulk density of 90 kg/cubic metre or less on a dry weight, the TML is 90% of the FMP.

### **1.2 Penetration test procedure**

The penetration test constitutes a procedure whereby a material in a cylindrical vessel is vibrated. The flow moisture point is determined on the basis of the penetration depth of an indicator.

#### **1.2.1 Scope**

- .1 The penetration test is generally suitable for mineral concentrates, similar materials, and coals up to a top size of 25 mm.
- .2 In this procedure, the sample, in a cylindrical vessel, is subjected to vertical vibration of  $2g \text{ rms} \pm 10\%$  ( $g$  = gravity acceleration) for 6 minutes. When the penetration depth of a bit put on the surface exceeds 50 mm, it is judged that the sample contains a moisture greater than the flow moisture point.
- .3 This procedure consists of a preliminary test to get an approximate value of the flow moisture point and a main test to determine the accurate flow moisture point. When the approximate value of the flow moisture point is known, the preliminary test can be omitted.
- .4 The room where the samples are tested should be prepared as mentioned in 1.1.3.

#### **1.2.2 Apparatus (see figure 1.2.2)**

- .1 The test apparatus consists of:
  - .1 a vibrating table;

- .2 cylindrical vessels;
  - .3 indicators (penetration bits and a holder);
  - .4 a tamper (see 1.1.2.4); and
  - .5 ancillary equipment (see 1.1.2.5 to .8).
- .2 The vibrator (see figure 1.2.2.2), with a table on which a cylindrical vessel can be clamped, should be capable of exciting a mass of 30 kg at a frequency of either 50 Hz or 60 Hz with an acceleration of 3g rms or more, and it can be controlled to adjust the acceleration level.
- .3 Dimensions of cylindrical vessels (see figures 1.2.2.3-1 and 1.2.2.3-2) are as follows:

Cylinder size	Inner diameter	Depth	Wall thickness
small	146 mm	202 mm	9.6 mm or more
large	194 mm	252 mm	10.3 mm or more

The vessels should be made of reasonably rigid, non-magnetic, impermeable and lightweight material such as acrylics or vinyl chloride.

The small cylindrical vessel is selected for the materials having a maximum particle size of 10 mm or less. The large cylindrical vessel is for those having a maximum particle size of 25 mm or less.

- .4 Penetration bits (see figure 1.2.2.4) are made of brass. The mass of the bit for coal should be adjusted to 88 g (5 kPa), and that for concentrates to 177 g (10 kPa). When the sample contains coarse particles, it is recommended that two bits of the same pressure are put on the surface to avoid misjudgment.
- .5 A holder (see figure 1.2.2.5) should be made to guide the rod of a bit with minimum friction to the centre of a cylindrical vessel. When two bits are used, they should be positioned in accordance with figure 1.2.2.
- .6 A cylindrical vessel and penetration indicators should be selected in accordance with the nature and condition of the test sample, viz. size of particles and bulk density.

### 1.2.3 Procedure

#### 1.2.3.1 Preparation of the test sample and the vibrating table:

- .1 The quantity of the sample required is approximately six times or more the capacity of the selected cylindrical vessel. The amount of representative test sample with which each container is filled should be as follows: approximately 1,700 cm<sup>3</sup> for the small container, and 4,700 cm<sup>3</sup> for the large container.
- .2 Mix the sample well and divide into three approximately equal sub-samples, namely (A), (B) and (C). The sub-sample (A) should be immediately weighed and placed in the drying oven to determine the moisture content of the sample "as received".

The sub-samples (B) and (C) are used for the preliminary test and the main test, respectively.

- .3 The vibration level of the vibrating table should be calibrated, using an acceleration meter, prior to carrying out testing. The acceleration of the table should be adjusted to 2g rms  $\pm$  10% with a container filled with a sample mounted on the table.

#### 1.2.3.2 Preliminary flow moisture test

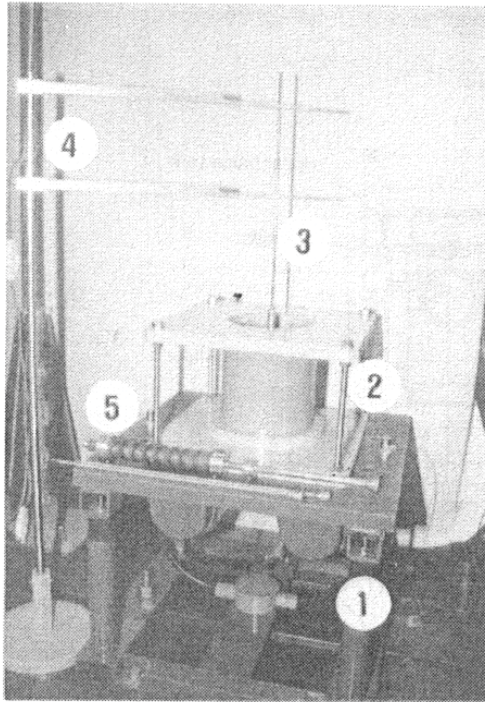
This test is intended to measure quickly the approximate flow moisture point, using sub-sample (B). Water is added in increments after every penetration test. When a flow state has been reached, the moisture content of the sample just above the flow state is measured. The moisture content of the sample just below the flow state can be calculated by deducting the increment of water last added from the gross mass of the sample.

- .1 Fill the appropriate cylindrical vessel with sub-sample (B) in four distinct stages and tamp after the addition of each layer using a specified tamper. Tamp to a pressure denoted in 1.1.4.1 for mineral concentrates or to 40 kPa for coals, and apply the pressure evenly over the whole surface area of the material until a uniformly flat surface is obtained.
- .2 Place the penetration bit on the surface of the material through the holder.
- .3 Operate the vibrator at a frequency of 50 Hz or 60 Hz with an acceleration of 2g rms  $\pm$  10% for 6 minutes. If necessary, the acceleration level should be checked by referring to the output of the acceleration meter attached to the vibrating table.
- .4 After 6 minutes of vibration, read the depth of penetration.

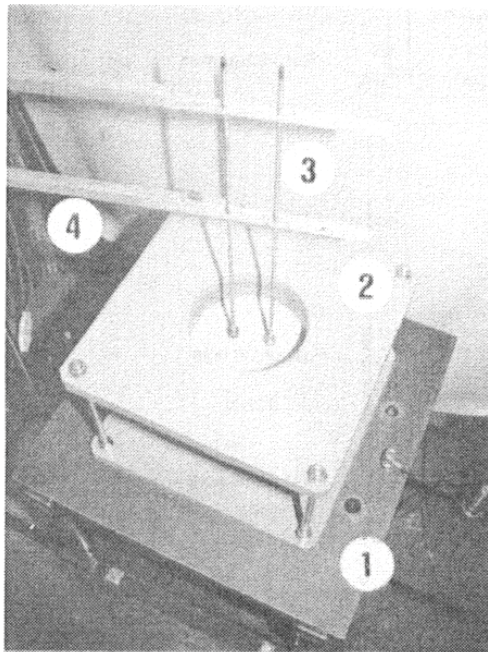
- .5 When the depth of penetration is less than 50 mm, it is judged that liquefaction did not take place. Then:
  - .1 Remove the material from the cylindrical vessel and replace in the mixing bowl with the remainder of the sample.
  - .2 Mix well and weigh the contents of the mixing bowl.
  - .3 Sprinkle an increment of water of not more than 1% of the mass of the material in the bowl and mix well.
  - .4 Repeat the procedure described in 1.2.3.2.1 to 1.2.3.2.5.
- .6 When the depth of penetration is greater than 50 mm, it is judged that liquefaction took place. Then:
  - .1 Remove the material from the cylindrical vessel and replace in the mixing bowl.
  - .2 Measure the moisture content in accordance with the procedure described in 1.1.4.4.
  - .3 Calculate the moisture content of the sample just below the flow moisture point on the basis of the amount of water added.
- .7 If the penetration depth in the first attempt exceeds 50 mm, i.e. the sample as received liquefied, mix sub-samples (B) and (C) and dry at room temperature to reduce the moisture. Then, divide the material into two sub-samples (B) and (C), and repeat the preliminary test.

#### 1.2.3.3 The main flow moisture test

- .1 On the basis of the preliminary test, the main test should be carried out to determine the flow moisture point more accurately.
- .2 Adjust the moisture content of the sub-sample (C) to the last value, which did not cause flow in the preliminary flow moisture test.
- .3 The first test of the main flow moisture test is carried out on this adjusted sample in the same manner as described in 1.2.3.2. In this case, however, the addition of water in increments should not be more than 0.5% of the mass of the test material.
- .4 When the approximate value of the flow moisture point is known in advance, the moisture content of the sub-sample (C) is adjusted to approximately 90% of this value.
- .5 When a flow state has been reached, the flow moisture point is determined as described in 1.1.4.3.



- ① Vibration table
- ② Cylindrical vessel (150 mm diameter)
- ③ Penetration bit (10 kPa)
- ④ Bit holder
- ⑤ Tamper

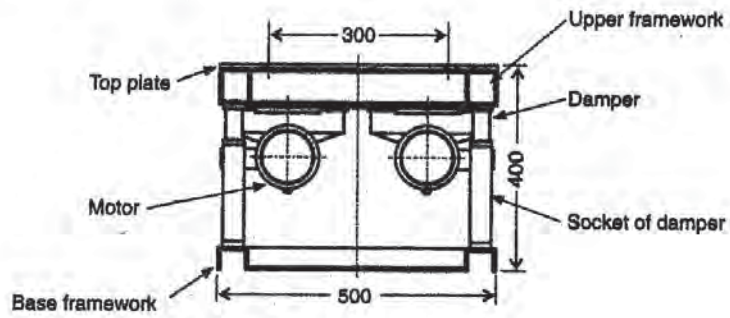


- ① Vibration table
- ② Cylindrical vessel (150 mm diameter)
- ③ Penetration bit (5 kPa)
- ④ Bit holder

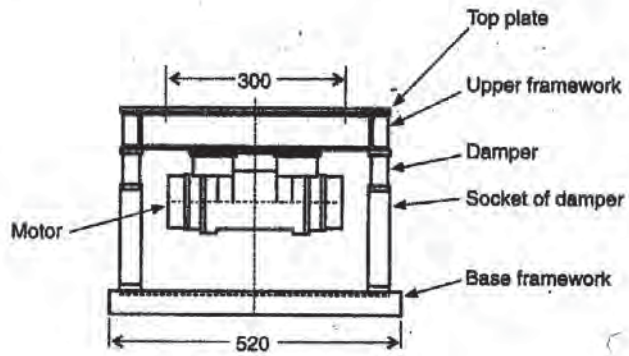
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**Figure 1.2.2 – Test apparatus**

FRONT VIEW



SIDE VIEW



VIEW FROM BASE

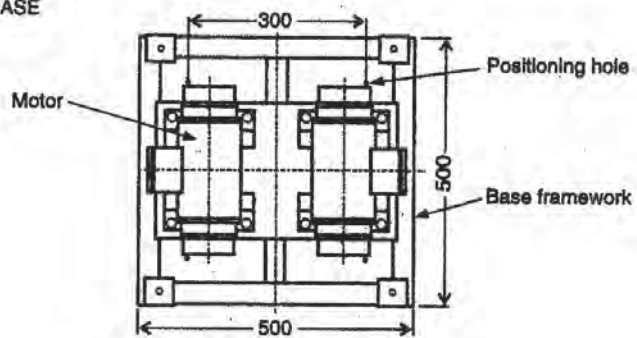


Figure 1.2.2.2 – *Vibration table*

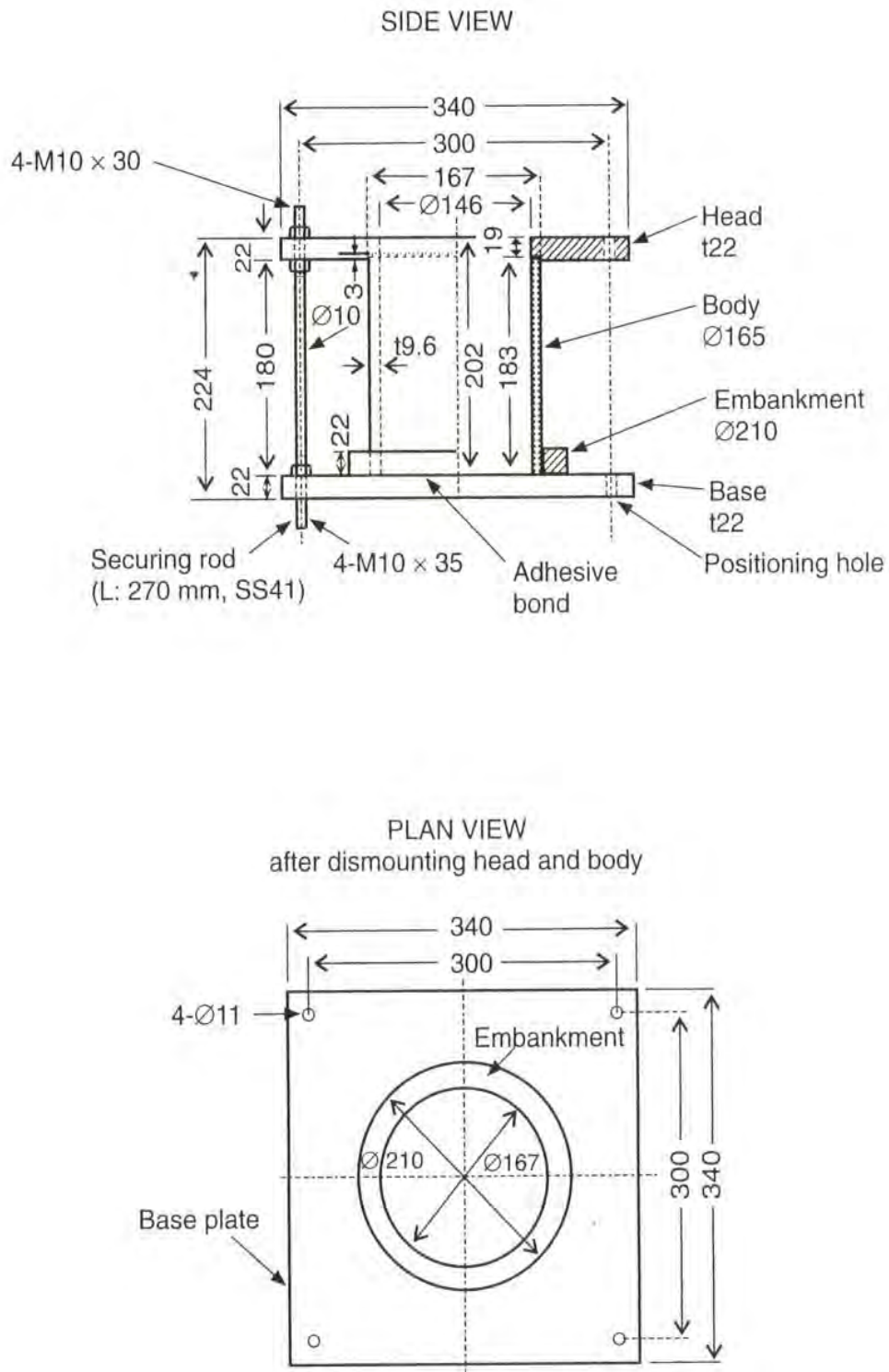
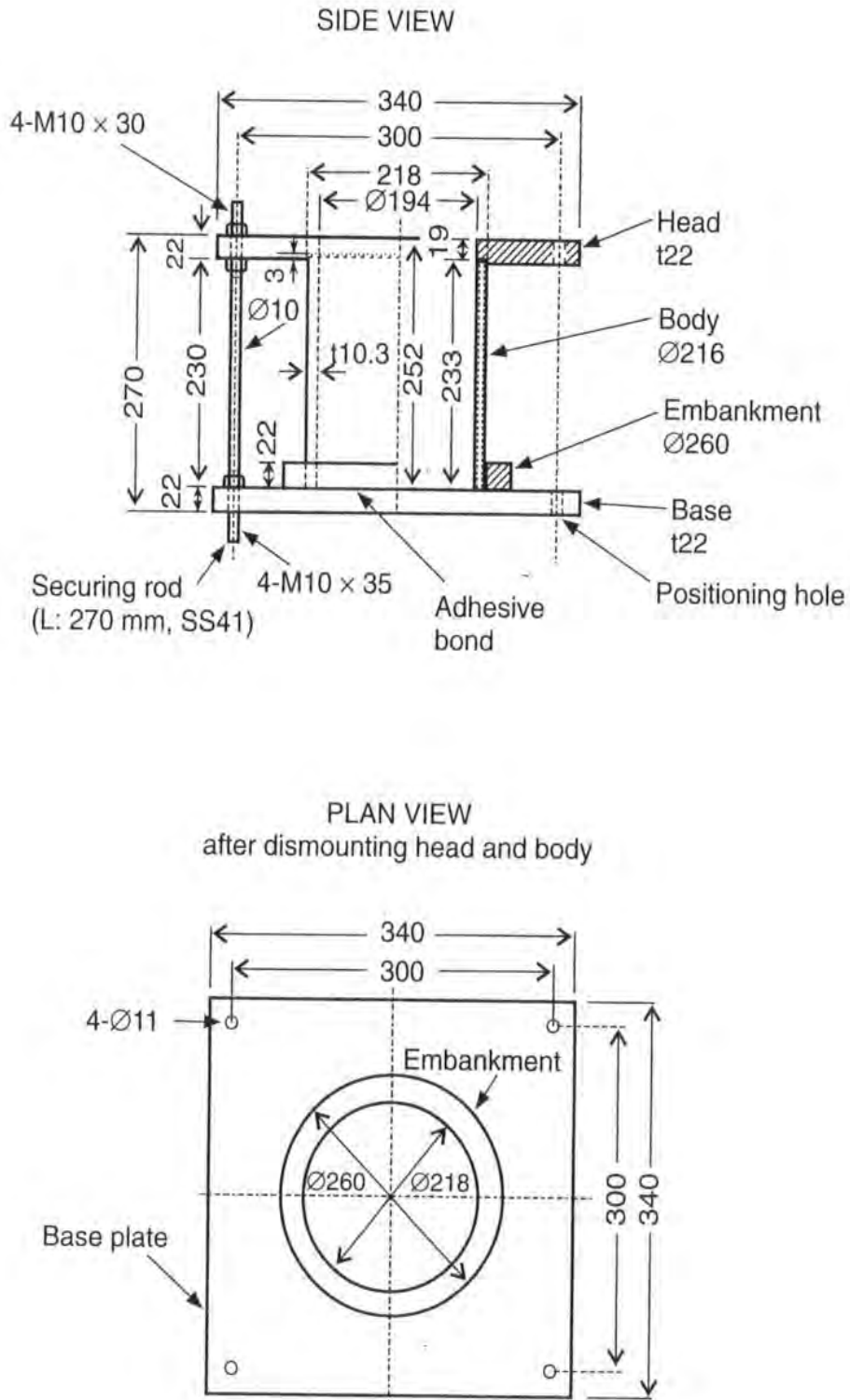
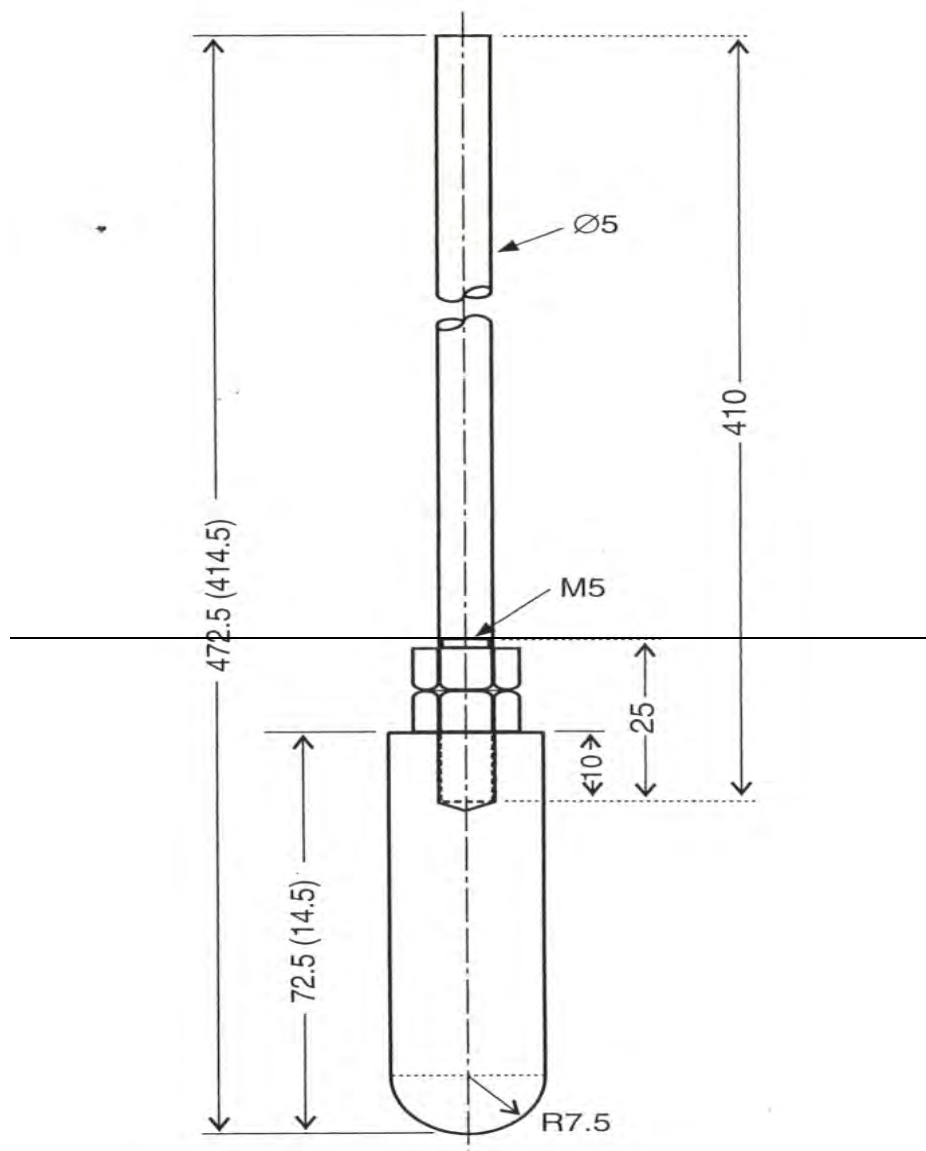


Figure 1.2.2.3-1 – *Cylindrical vessel, 150 mm diameter*



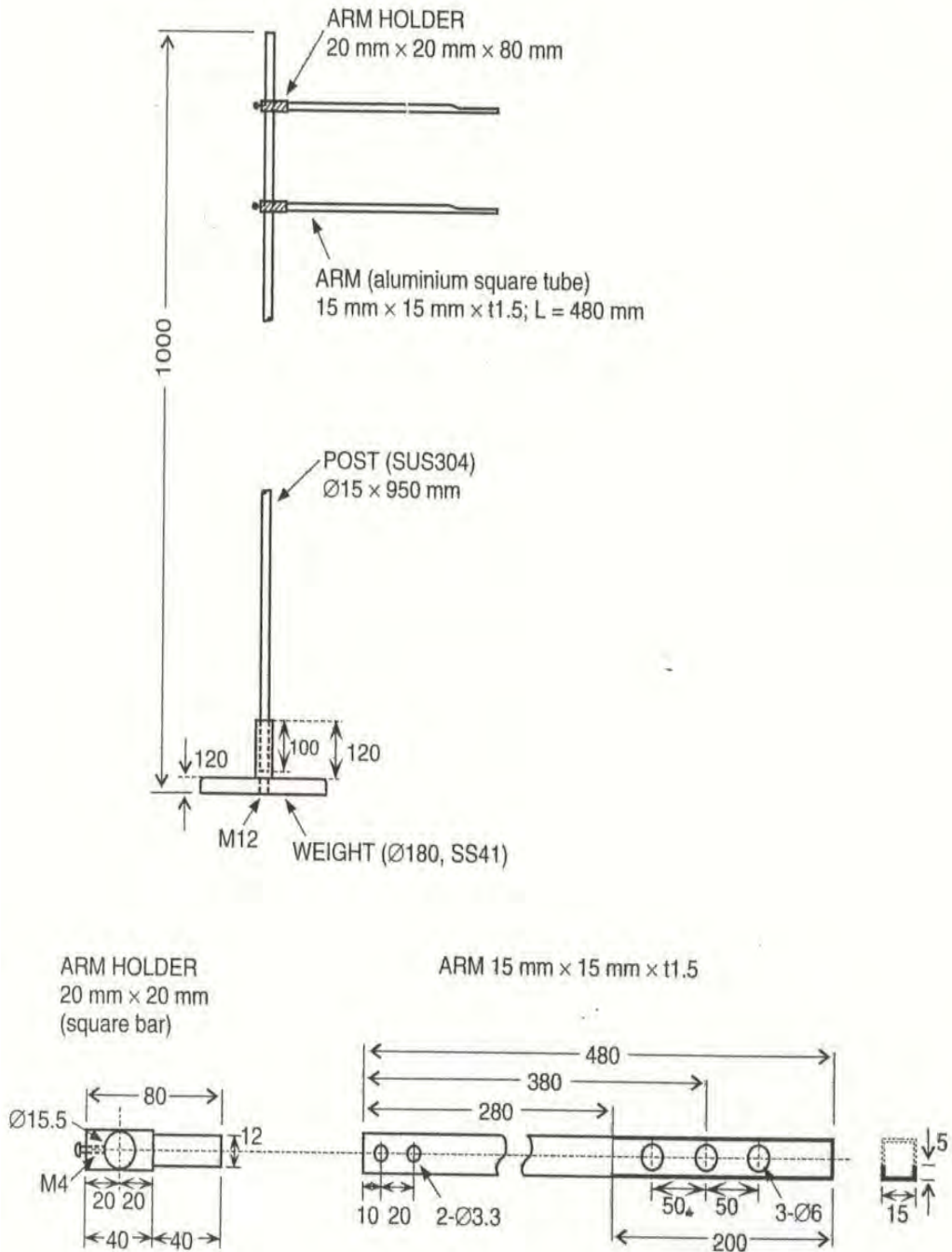
**Figure 1.2.2.3-2 – Cylindrical vessel, 200 mm diameter**





(Dimensions indicated in brackets are of the 5 kPa bit)  
(unit: mm)

**Figure 1.2.2.4 – Penetration bit**



**Figure 1.2.2.5 – Bit holder**

### 1.3 *Proctor/Fagerberg test procedure*

#### 1.3.1 *Scope*

- .1 Test method for both fine and relatively coarse-grained ore concentrates or similar materials up to a top size of 5 mm. This method should not be used for coal or other porous materials.
- .2 Before the Proctor/Fagerberg test is applied to coarser materials with a top size greater than 5 mm, an extensive investigation for adoption and improvement is required.
- .3 The transportable moisture limit (TML) of a cargo is taken as equal to the critical moisture content at 70% degree of saturation according to the Proctor/Fagerberg method test.

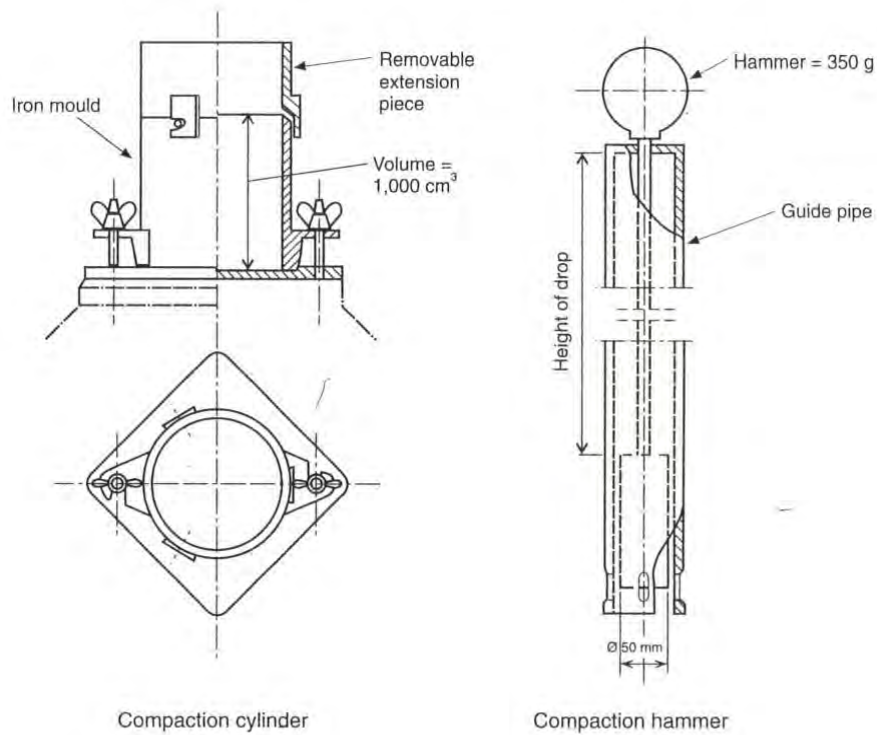
#### 1.3.2 *Proctor/Fagerberg test equipment*

- .1 The Proctor apparatus (see figure 1.3.2) consists of a cylindrical iron mould with a removable extension piece (the compaction cylinder) and a compaction tool guided by a pipe open at its lower end (the compaction hammer).
- .2 Scales and weights (see 3.2) and suitable sample containers.
- .3 A drying oven with a controlled temperature interval from 100°C to maximum 105°C. This oven should be without air circulation.
- .4 A suitable mixer. Care should be taken to ensure that the use of the mixer does not reduce the particle size or consistency of the test material.
- .5 Equipment to determine the density of the solid material, for example a pycnometer.

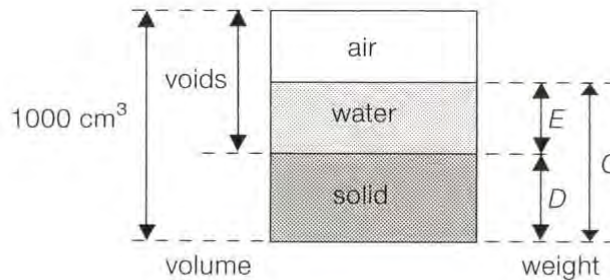
#### 1.3.3.3 Temperature and humidity (see 1.1.3)

#### 1.3.4 *Procedure*

- .1 *Establishment of a complete compaction curve.* A representative sample according to a relevant standard (see section 4.7, page 20) of the test material is dried at a temperature of approximately 100°C. The total quantity of the test material should be at least three times as big as required for the complete test sequence. Compaction tests are executed for five to ten different moisture contents (five to ten separate tests). The samples are adjusted in order that dry to almost saturated (plastic) samples are obtained. The required quantity per compaction test is about 2000 cm<sup>3</sup>.



**Figure 1.3.2 – Proctor apparatus**



**Figure 1.3.4.2**

At each compaction test a suitable amount of water is added to the sample of the dried test material and mixed thoroughly for 5 minutes. Approximately one fifth of the mixed sample is filled into the mould and levelled and then the increment is tamped uniformly over the surface of the increment. Tamping is executed by dropping the hammer 25 times through the guide pipe, 0.2 m each time. The performance is repeated for all five layers. When the last layer has been tamped the extension piece is removed and the sample is levelled off along the brim of the mould. When the weight of the cylinder with the tamped sample has been determined, the cylinder is emptied, the sample is dried and the weight is determined.

The test then is repeated for the other samples with different moisture contents.

.2 *Definitions and data for calculations (see figure 1.3.4.2)*

- empty cylinder, mass in grams: A
  - cylinder with tamped sample, mass in grams: B
  - wet sample, mass in grams: C
- $$C = B - A$$
- dry sample, mass in grams: D
  - water, mass in grams (equivalent to volume in cm<sup>3</sup>): E

$$E = C - D$$

Volume of cylinder: 1000 cm<sup>3</sup>

.3 *Calculation of main characteristics*

- density of solid material, g/cm<sup>3</sup> (t/m<sup>3</sup>): d
- dry bulk density, g/cm<sup>3</sup> (t/m<sup>3</sup>):  $\gamma$

$$\gamma = \frac{D}{1000}$$

- net water content, volume %:  $e_v$

$$e_v = \frac{E}{D} \times 100 \times d$$

- void ratio: e (volume of voids divided by volume of solids)

$$e = \frac{1000 - D}{D} = \frac{d}{\lambda} = - 1$$

- degree of saturation, percentage by volume: S

$$S = \frac{e_v}{e}$$

- gross water content, percentage by mass:  $W^1$

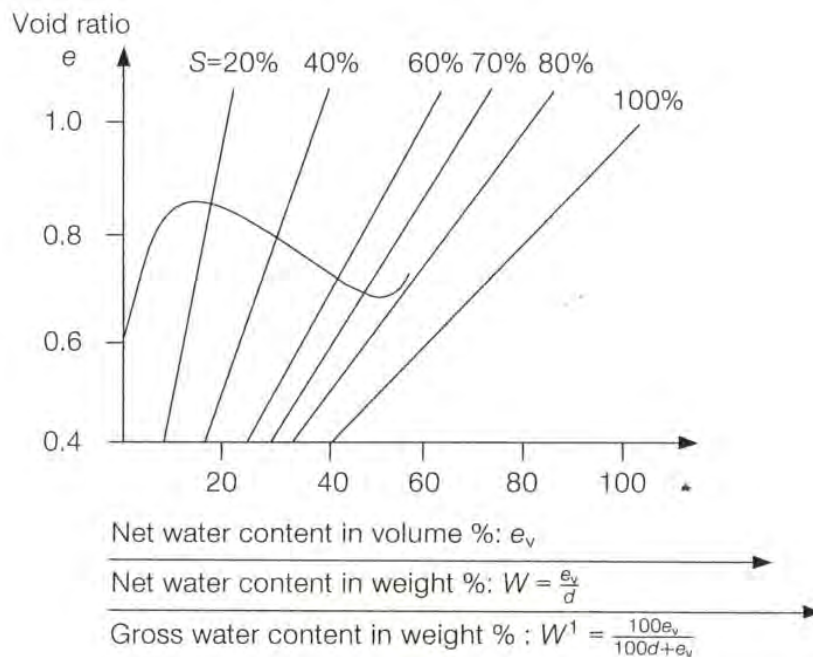
$$W^1 = \frac{E}{C} \times 100$$

- net water content, percentage by mass:  $W$

$$W = \frac{E}{D} \times 100$$

.4 *Presentation of the compaction tests*

For each compaction test the calculated void ratio ( $e$ ) value is plotted as the ordinate in a diagram with net water content ( $e_v$ ) and degree of saturation ( $S$ ) as the respective abscissa parameters.



**Figure 1.3.4.5**

.5 *Compaction curve*

The test sequence results in a specific compaction curve (see figure 1.3.4.5).

The critical moisture content is indicated by the intersection of the compaction curve and the line  $S = 70\%$  degree of saturation. The transportable moisture limit (TML) is the critical moisture content.

## 2 Test procedures to determine the angle of repose and associated apparatus

### 2.1 *Determination of angle of repose of fine-grained materials (size less than 10 mm): “tilting box test”. For use in laboratory or port of loading*

#### 2.1.1 *Scope*

The test provides for the determination of the angle of repose of fine-grained non-cohesive materials (size less than 10 mm). The results so obtained may be used when interpreting sections 5 and 6 of this Code for the materials in question.

#### 2.1.2 *Definition*

The angle of repose obtained by this test is the angle formed between the horizontal and the top of the testbox when the material in the box just begins to slide in bulk.

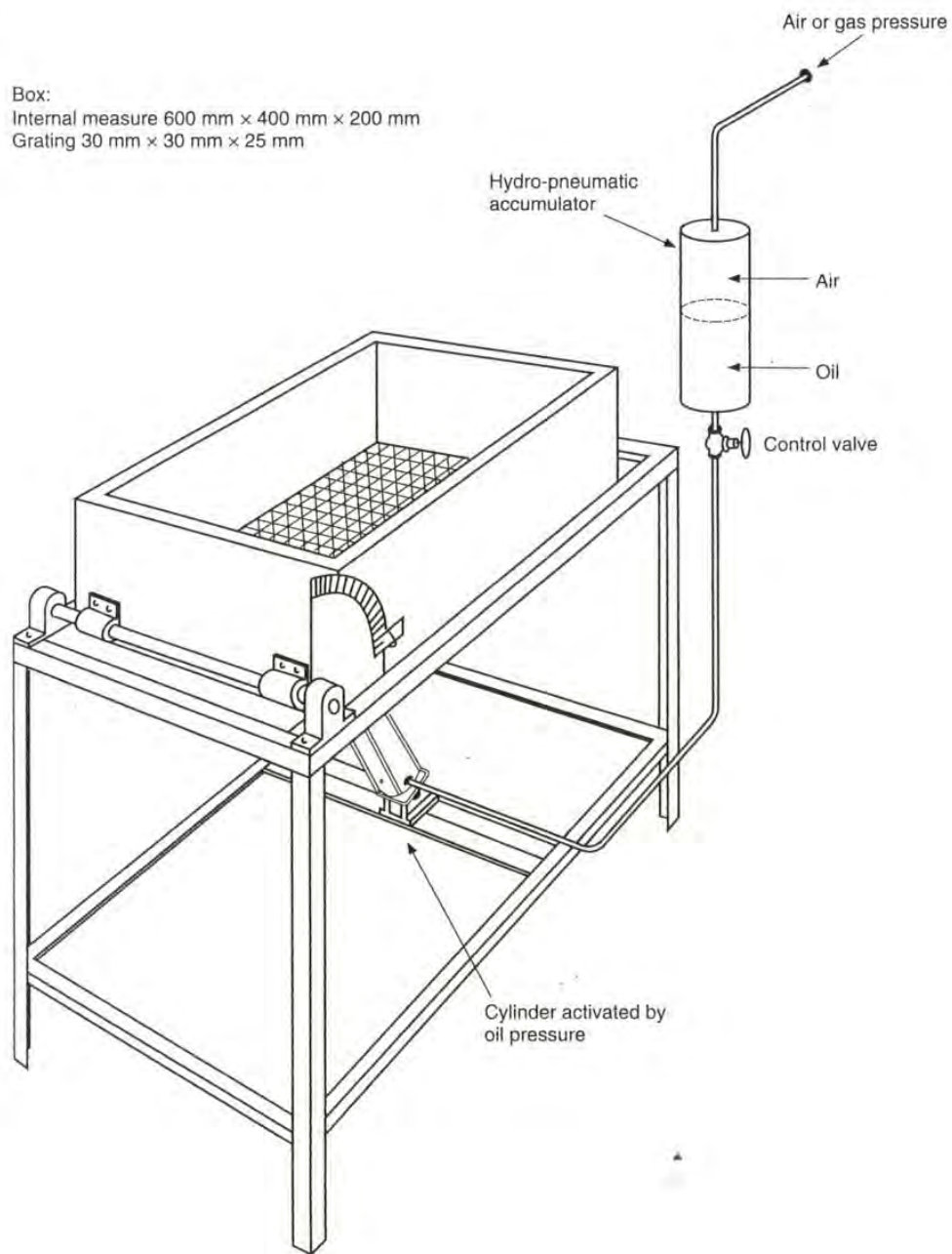
#### 2.1.3 *Principle of test*

When measuring the angle of repose by this method, the material surface should initially be level and parallel to the testbox base. The box is tilted without vibration and tilted without vibration and tilting is stopped when the product just begins to slide in bulk.

#### 2.1.4 *Apparatus (see figure 2.1.4)*

Apparatus is as follows:

- .1 A framework, on top of which is attached an open box. Attachment of the box to the frame is by means of a shaft passing through bearings affixed to both the frame and the end of the box, enabling the box to be subjected to a controlled tilt.
- .2 The dimensions of the box are 600 mm long, 400 mm wide and 200 mm high.
- .3 To prevent sliding of the material along the bottom of the box during tilting, a tightly fitting grating (openings 30 mm x 30 mm x 25 mm) is placed on the bottom of the box before filling.
- .4 Tilting of the box is effected by a hydraulic cylinder fitted between the frame and the bottom of the box. Other means may be used to obtain the required tilting but in all cases vibration must be eliminated.
- .5 To pressurize the hydraulic cylinder, a hydropneumatic accumulator may be used, pressurized by air or gas at a pressure of about 5 kp/cm<sup>2</sup>.
- .6 The rate of tilting should be approximately 0.3°/s.
- .7 Range of tilt should be at least 50°.



**Figure 2.1.4 – Basic sketch of tilting box**

- .8 A protractor is fitted to the end of the shaft. One lever of the protractor is fitted so that it may be screw-adjusted to the horizontal.
- .9 The protractor should measure the angle of the top of the box to the horizontal to within an accuracy of  $0.5^\circ$ .
- .10 A spirit level or some other levelling device should be available to zero the protractor.



### 2.1.5 *Procedure*

The box is filled with the material to be tested by pouring it slowly and carefully from the lowest practical height into the box in order to obtain uniformity of loading.

The excess material is scraped off with the aid of a straight edge, inclined at about 45° towards the direction of scraping.

The tilting system is then activated and stopped when the material just begins to slide in bulk.

The angle of the top of the box to the horizontal is measured by the protractor and recorded.

### 2.1.6 *Evaluation*

The angle of repose is calculated as the mean of three measurements and is reported to within half a degree.

**Notes:** Preferably the test should be carried out with three independent samples.

Care should be taken to ensure that the shaft is adjusted to be horizontal before testing.

## 2.2 *Alternative or shipboard test method to be used for the determination of the angle of repose when the tilting box is not available*

### 2.2.1 *Definition*

According to this method the angle of repose is the angle between the cone slope and the horizontal measured at half height.

### 2.2.2 *Principle of test*

To determine the angle of repose, a quantity of the material to be tested is poured very carefully out of a flask onto a sheet of rough-textured paper, in such a way that a symmetrical cone is formed.

### 2.2.3 *Equipment*

The necessary equipment to carry out this test is as follows:

- a horizontal table free from vibrations;
- a sheet of rough-textured paper onto which the material should be poured;
- a protractor; and
- a 3-litre conical flask.

#### 2.2.4 Procedure

Put the sheet of paper on the table. Split 10l of the material to be tested into three sub-samples and test each in the following way:

Pour two thirds of the sub-sample (i.e. 2 l) onto the sheet, producing a starting cone. The remainder of this sub-sample is then poured very carefully from a height of a few millimetres on top of the cone. Care should be taken that the cone will be built up symmetrically. This may be achieved by revolving the flask slowly close around the top of the cone when pouring.

When measuring, care should be taken that the protractor does not touch the cone; otherwise this may result in sliding of the material and spoil the test.

The angle has to be measured at four places around the cone, about 90 degrees apart.

This test should be repeated on the other two sub-samples.

#### 2.2.5 Calculations

The angle of repose is taken as the mean of the 12 measurements and is reported to half a degree. This figure can be converted to the tilting box value as follows:

$$a_t = a_s + 3^\circ \quad (2.2.5)$$

Where  $a_t$  = angle of repose according to the tilting box text

$a_s$  = angle of repose according to the survey test

### 3 Standards used in test procedures

#### 3.1 *Standard flow table and frame*\*

##### 3.1.1 *Flow table and frame*

3.1.1.1 The flow table apparatus shall be constructed in accordance with figure 3. The apparatus shall consist of an integrally cast rigid iron frame and a circular rigid table top, 10 inches  $\pm$  0.1 inch (254 mm  $\pm$  2.5 mm) in diameter, with a shaft attached perpendicular to the table top by means of a screw thread. The table top, to which the shaft with its integral contact shoulder is attached, shall be mounted on a frame in such a manner that it can be raised and dropped vertically through the specified height, with a tolerance in height of  $\pm$ 0.005 inches (0.13 mm) for new tables and  $\pm$  0.015 inches (0.39 mm) for tables in use, by means of a rotated cam. The table top shall have a fine-machined plane surface, free of blowholes and surface defects, and shall be scribed as shown in figure 3. The table top shall be of cast brass or bronze having a Rockwell hardness number not less than HRB 25 with an edge thickness of 0.3 inches (8 mm), and shall have six integral radial stiffening ribs. The table top and attached shaft shall weigh 9 lb  $\pm$  0.1 lb (4 kg  $\pm$  0.05 kg) and the weight shall be symmetrical around the centre of the shaft.

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\* Source: "Standard Specification for Flow Table for Use in Tests of Hydraulic Cement", Designation C230-68. Reprinted by permission of American Society for Testing and Materials (ASTM), 1916 Race Street, Philadelphia, Penn., USA, copyright ASTM 1977.

3.1.1.2 The cam and vertical shaft shall be of medium-carbon machinery steel, hardened where indicated in figure 3. The shaft shall be straight and the difference between the diameter of the shaft and the diameter of the bore of the frame shall be not less than 0.002 inches (0.05) and not more than 0.003 inches (0.08 mm) for new tables and shall be maintained at from 0.002 inches to 0.010 inches (0.26 mm) for tables in use. The end of the shaft shall not fall upon the cam at the end of the drop, but shall make contact with the cam not less than 120° from the point of drop. The face of the cam shall be a smooth spiralled curve of uniformly increasing radius from ½ inch to 1¼ inches (13 mm to 32 mm) in 360° and there shall be no appreciable jar as the shaft comes into contact with the cam. The cam shall be so located and the contact faces of the cam and shaft shall be such that the table does not rotate more than one revolution in 25 drops. The surfaces of the frame and of the table which come into contact at the end of the drop shall be maintained smooth, plane, and horizontal and parallel with the upper surface of the table and shall make continuous contact over a full 360°.

3.1.1.3 The supporting frame of the flow table shall be integrally cast of fine-grained, high-grade cast iron. The frame casting shall have three integral stiffening ribs extending the full height of the frame and located 120° apart. The top of the frame shall be chilled to a depth of approximately ¼ inch (6.4 mm) and the face shall be ground and lapped square with the bore to give 360° contact with the shaft shoulder. The underside of the base of the frame shall be ground to secure a complete contact with the steel plate beneath.

3.1.1.4 The flow table may be driven by a motor,<sup>1</sup> connected to the camshaft through an enclosed worm gear speed reducer and flexible coupling. The speed of the camshaft shall be approximately 100 rpm. The motor drive mechanism shall not be fastened or mounted on the table base plate or frame.

The performance of a flow table shall be considered satisfactory if, in calibration tests, the table gives a flow value that does not differ by more than 5 percentage points from flow values obtained with a suitable calibration material.<sup>2</sup>

### 3.1.2 *Flow table mounting*

3.1.2.1 The flow table frame shall be tightly bolted to a cast iron or steel plate at least 1 inch (25 mm) thick and 10 inches (250 mm) square. The top surface of this plate shall be machined to a smooth plane surface. The plate shall be anchored to the top of a concrete pedestal by four ½ inch (13 mm) bolts that pass through the plate and are embedded at least 6 inches (150 mm) in the pedestal. The pedestal shall be cast inverted on the base plate. A positive contact between the base plate and the pedestal shall be obtained at all points. No nuts or other such levelling devices shall be used between the plate and the pedestal. Levelling shall be effected by suitable means under the base of the pedestal.

---

<sup>1</sup> A 1/20 hp (40 W) motor has been found adequate. The flow table may be driven by a hand-operated camshaft as shown in the illustration.

<sup>2</sup> Such a material may be obtained from the Cement and Concrete Reference Laboratory at the National Bureau of Standards, Washington, D.C. 20234, USA.

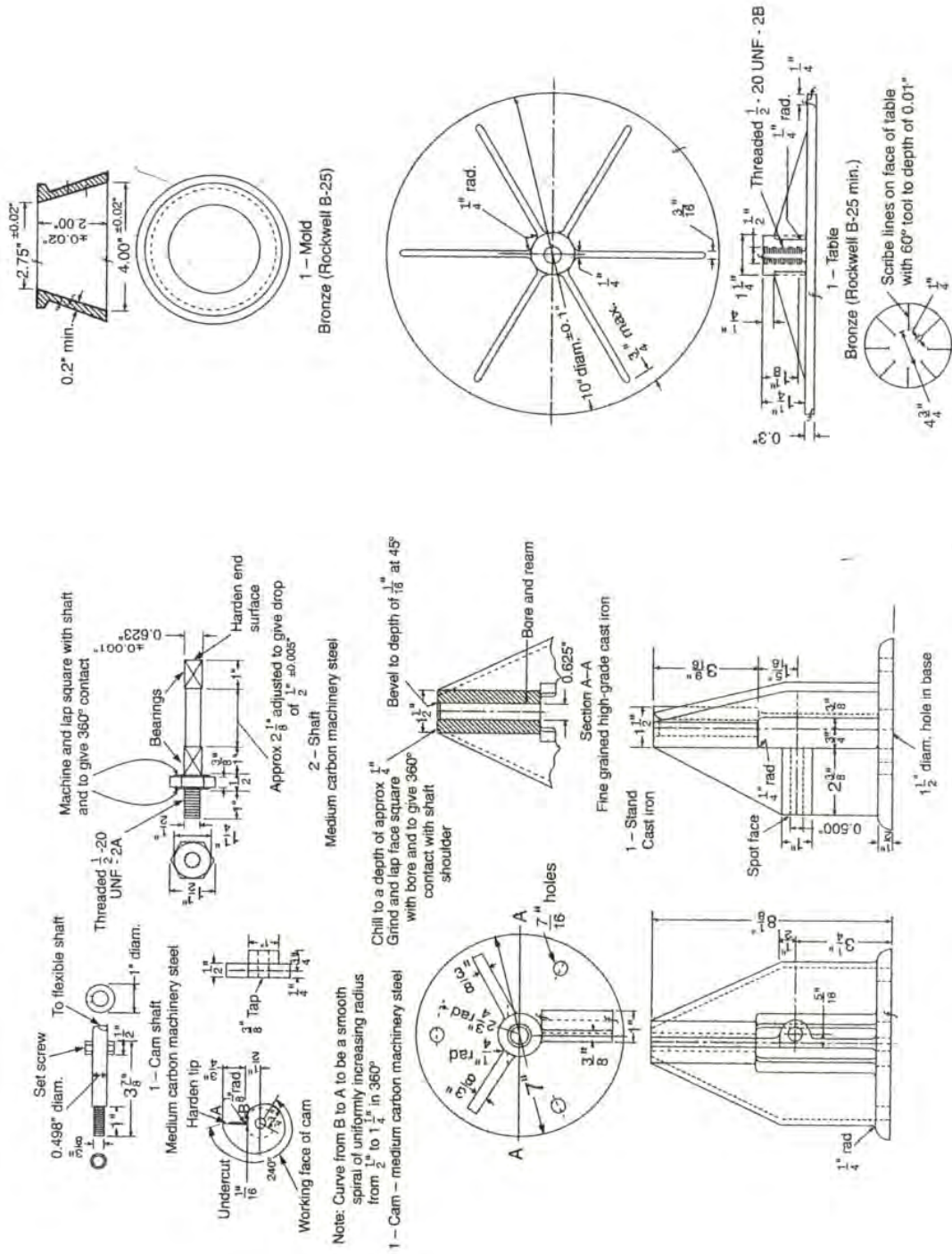


Figure 3

3.1.2.2 The pedestal shall be 10 inches to 11 inches (250 mm to 275 mm) square at the top, and 15 inches to 16 inches (375 mm to 400 mm) square at the bottom, 25 inches to 30 inches (625 mm to 750 mm) in height, and shall be of monolithic construction, cast from concrete weighing at least 140 lb/ft<sup>3</sup> (2,240 kg/m<sup>3</sup>). A stable gasket cork pad, ½ inch (13 mm) thick and approximately 4 inches (102 mm) square, shall be inserted under each corner of the pedestal. The flow table shall be checked frequently for levelness of the table top, stability of the pedestal, and tightness of the bolts and nuts in the table base and the pedestal plate. (A torque of 20 lb ft (27 Nm) is recommended when tightening those fastenings.)

3.1.2.3 The table top, after the frame has been mounted on the pedestal, shall be level along two diameters at right angles to each other, in both the raised and lowered positions.

### 3.1.3 *Flow table lubrication*

3.1.3.1 The vertical shaft of the table shall be kept clean and shall be lightly lubricated with a light oil (SAE-10). Oil shall not be present between the contact faces of the table top and the supporting frame. Oil on the cam face will lessen wear and promote smoothness of operation. The table should be raised and permitted to drop a dozen or more times just prior to use if it has not been operated for some time.

### 3.1.4 *Mould*

3.1.4.1 The mould for casting the flow specimen shall be of cast bronze or brass, constructed as shown in figure 3. The Rockwell hardness number of the metal shall be not less than HRB 25. The diameter of the top opening shall be 2.75 inches  $\pm$  0.02 inches (69.8 mm  $\pm$  0.5 mm) for new moulds and 2.75 inches + 0.05 inches (+ 1.3 mm) and – 0.02 inches for moulds in use. The surfaces of the base and top shall be parallel and at right angles to the vertical axis of the cone. The mould shall have a minimum wall thickness of 0.2 inches (5 mm). The outside of the top edge of the mould shall be shaped so as to provide an integral collar for convenient lifting of the mould. All surfaces shall be machined to a smooth finish. A circular shield approximately 10 inches (254 mm) in diameter, with a centre opening approximately 4 inches (102 mm) in diameter, made of non-absorbing material not attacked by the cement, shall be used with the flow mould to prevent mortar from spilling on the table top.

## 3.2 *Scales and weights*\*

### 3.2.1 *Scales*

3.2.1.1 The scales used shall conform to the following requirements. On scales in use, the permissible variation at a load of 2000 g shall be  $\pm$  2.0 g. The permissible variation on new scales shall be one half of this value. The sensibility reciprocal\*\* shall be not greater than twice the permissible variation.

---

\* Source: "Standard Method of Test for Compressive Strength of Hydraulic Cement Mortars", Designation C109-3. Reprinted by permission of American Society for Testing and Materials (ASTM), 1916 Race Street, Philadelphia, Penn., USA, copyright ASTM 1977.

\*\* Generally defined, the sensibility reciprocal is the change in load required to change the position of rest of the indicating element or elements of a non-automatic indicating scale a definite amount at any load. For a more complete definition, see "Specifications, Tolerances, and Regulations for Commercial Weighing and Measuring Devices", *Handbook H44*, National Bureau of Standards, Washington, D.C., USA, September 1949, pp. 92 and 93.

### 3.2.2 *Weights*

3.2.2.1 The permissible variations on weights shall be as prescribed in the table below. The permissible variations on news weights shall be one half of the values in the table below.

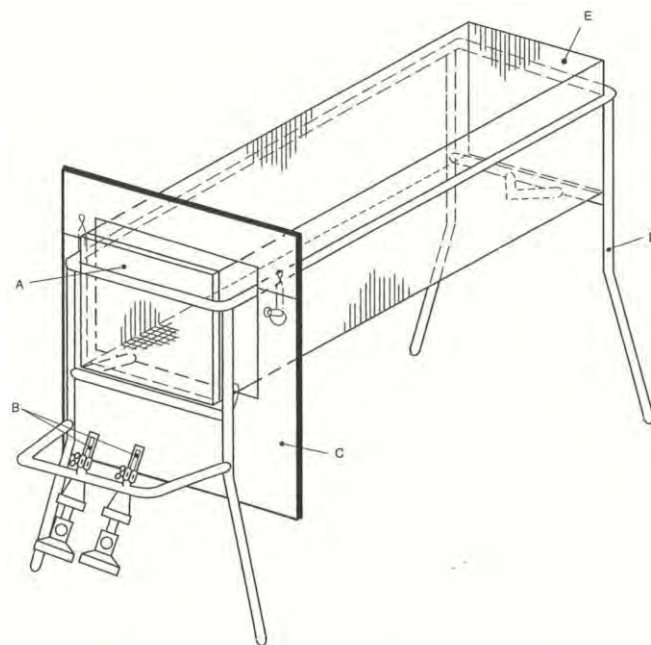
#### PERMISSIBLE VARIATIONS ON WEIGHTS

Weight (g)	Permissible variations on weights in use, plus or minus (g)
1000 .....	0.50
900 .....	0.45
750 .....	0.40
500 .....	0.35
300 .....	0.30
250 .....	0.25
200 .....	0.20
100 .....	0.15
50 .....	0.10
20 .....	0.05
10 .....	0.04
5 .....	0.03
2 .....	0.02
1 .....	0.01

## 4 Trough test for determination of the self-sustaining exothermic decomposition of fertilizers containing nitrates\*

### 4.1 Definition

A fertilizer capable of self-sustaining decomposition is defined as one in which decomposition initiated in a localized area will spread throughout the mass. The tendency of a fertilizer offered for transport to undergo this type of decomposition can be determined by means of the trough test. In this test localized decomposition is initiated in a bed of the fertilizer to be contained in a horizontally mounted trough. The amount of propagation, after removal of the initiating heat source, of decomposition through the mass is measured.



- A Steel plate (150 x 150 mm and 1 to 3 mm thick)
- B Gas burners (e.g., Teclu or Bunsen)
- C Heat shield (2 mm thick)
- D Stand (e.g., made from 15 mm wide, 2 mm thick steel bar)
- E Gauze trough (150 x 150 x 500 mm)

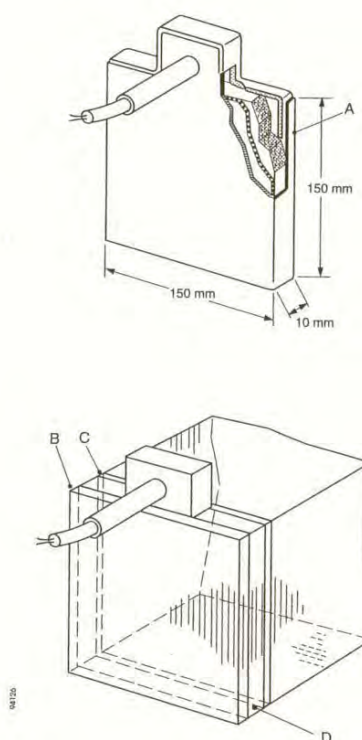
**Figure 4-1 – Gauze trough with support and burners**

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\* Source: Section 38 of the United Nations Recommendation on the Transport of Dangerous Goods, Manual of Tests and Criteria.

## 4.2 Apparatus and materials

The apparatus (figure 4-1) consists of a trough of internal dimensions 150 mm x 150 mm x 500 mm, open at the top. The trough is constructed of square-meshed gauze (preferably stainless steel) with a mesh width of about 1.5 mm and a wire thickness of 1.0 mm supported on a frame made from, for example, 15 mm wide, 2 mm thick steel bars. The gauze at each end of the trough may be replaced by 1.5 mm thick, 150 mm x 150 mm stainless steel plates. The trough should be rested on a suitable support. Fertilizers with a particle size distribution such that a significant amount falls through the mesh of the trough should be tested in a trough of smaller mesh gauze, or alternatively in a trough lined with gauze of a smaller mesh. During initiation sufficient heat should be provided and maintained to establish a uniform decomposition front. Two alternative heating methods are recommended, viz:



- A Aluminium or stainless steel sheathing (thickness 3 mm)
- B Insulating plate (thickness 5 mm)
- C Aluminium foil or stainless steel plate (thickness 3 mm)
- D Position of heating device in trough

**Figure 4-2 – Electrical heating device (capacity 250 W)**

### 4.2.1 Electrical heating

An electrical heating element (capacity 250 W) enclosed in a stainless steel box is placed inside and at one end of the trough (figure 4-2). The dimensions of the stainless steel box are 145 mm x 145 mm x 10 mm, and the wall thickness is 3 mm. The side of the box which is not in contact with the fertilizer should be protected with a heat shield (insulation plate 5 mm thick). The heating side of the box may be protected with aluminium foil or a stainless steel plate.



#### 4.2.2 *Gas burners*

A steel plate (thickness 1 mm to 3 mm) is placed inside one end of the trough and in contact with the wire gauze (figure 4-1). The plate is heated by means of two burners which are fixed to the trough support and are capable of maintaining the plate at temperatures between 400°C and 600°C, i.e. dull red heat.

4.2.3 To prevent heat transport along the outside of the trough, a heat shield consisting of a steel plate (2 mm thick) should be installed at about 50 mm from the end of the trough where the heating takes place.

4.2.4 The life of the apparatus may be prolonged if it is constructed of stainless steel throughout. This is particularly important in the case of the gauze trough.

4.2.5 Propagation may be measured using thermocouples in the substance and recording the time at which a sudden temperature rise occurs as the reaction front reaches the thermocouple.

#### 4.3 *Procedure*

4.3.1 The apparatus should be set up under a fume hood to remove toxic decomposition gases or in an open area where the fumes can be readily dispersed. Although there is no explosion risk, when performing the test it is advisable to have a protective shield, e.g., of suitable transparent plastics, between the observer and the apparatus.

4.3.2 The trough is filled with the fertilizer in the form to be offered for shipment and decomposition is initiated at one end, either electrically or by means of gas burners as described above. Heating should be continued until decomposition of the fertilizer is well established and propagation of the front (over approximately 30 mm to 50 mm) has been observed. In the case of products with high thermal stability, it may be necessary to continue heating for two hours. If fertilizers show a tendency to melt, the heating should be done with care, i.e. using a small flame.

4.3.3 About 20 minutes after the heating has been discontinued, the position of the decomposition front is noted. The position of the reaction front can be determined by difference in colour, e.g., brown (undecomposed fertilizer) to white (decomposed fertilizer), or by the temperature indicated by adjacent pairs of thermocouples which bracket the reaction front. The rate of propagation may be determined by observation and timing or from thermocouple records. It should be noted whether there is no propagation after heating is discontinued or whether propagation occurs throughout the substance.

#### 4.4 *Test criteria and method of assessing results*

4.4.1 If propagation of the decomposition continues throughout the substance the fertilizer is considered capable of showing self-sustaining decomposition.

4.4.2 If propagation does not continue throughout the substance, the fertilizer is considered to be free from the hazard of self-sustaining decomposition.

## 5 Description of the Test of Resistance to Detonation

### 5.1 Principle

5.1.1 The test sample is confined in a steel tube and subjected to detonation shock from an explosive booster charge. Propagation of the detonation is determined from the degree of compression of lead cylinders on which the tube rests horizontally during the test.

### 5.2 Sample preparation

5.2.1 The test must be carried out on a representative sample of cargo. Before being tested for resistance to detonation, the whole mass of the sample is to be thermally cycled five times between 25°C and 50°C ( $\pm 1^\circ\text{C}$ ) in sealed tubes. The sample shall be maintained at the extreme temperatures, measured at the centre of the sample, for at least 1 hour during each thermal cycle and at 20°C ( $\pm 3^\circ\text{C}$ ) after complete cycling until tested.

### 5.3 Materials

Seamless steel tube to ISO 65-1981-Heavy or equivalent

Tube length	1,000 mm
Nominal external diameter	114 mm
Nominal wall thickness	5 to 6.5 mm

Bottom plate (160 x 160 mm) of good weldable quality, thickness 5 to 6 mm to be butt-welded to one end of the tube around the entire circumference.

Initiation system and booster

Electrical blasting cap or detonating cord with non-metallic sleeve (10 to 13 g/m).

Compressed pellet of secondary explosive, such as hexogen/wax 95/5 or tetryl, with a central recess to take the detonator.

500  $\pm$  1 gramme plastic explosive containing 83 to 86 % penthrite, formed into a cylinder in a cardboard or plastic tube. Detonation velocity 7,300 to 7,700 m/s.

Six witness cylinders of refined, cast lead for detecting detonation.

50 mm diameter x 100 mm high, refined lead of at least 99.5% purity.

### 5.4 Procedure

Test Temperature: 15 to 20°C. Figures 1 and 2 show the test arrangement.

Fill the tube about one-third of its height with the test sample and drop it 10 cm vertically five times on the floor. Improve the compression by striking the side wall with a hammer between

drops. A further addition shall be made such that, after compaction or by raising and dropping the tube 20 times and a total of 20 intermittent hammer blows, the charge fills the tube to a distance of 70 mm from its orifice.

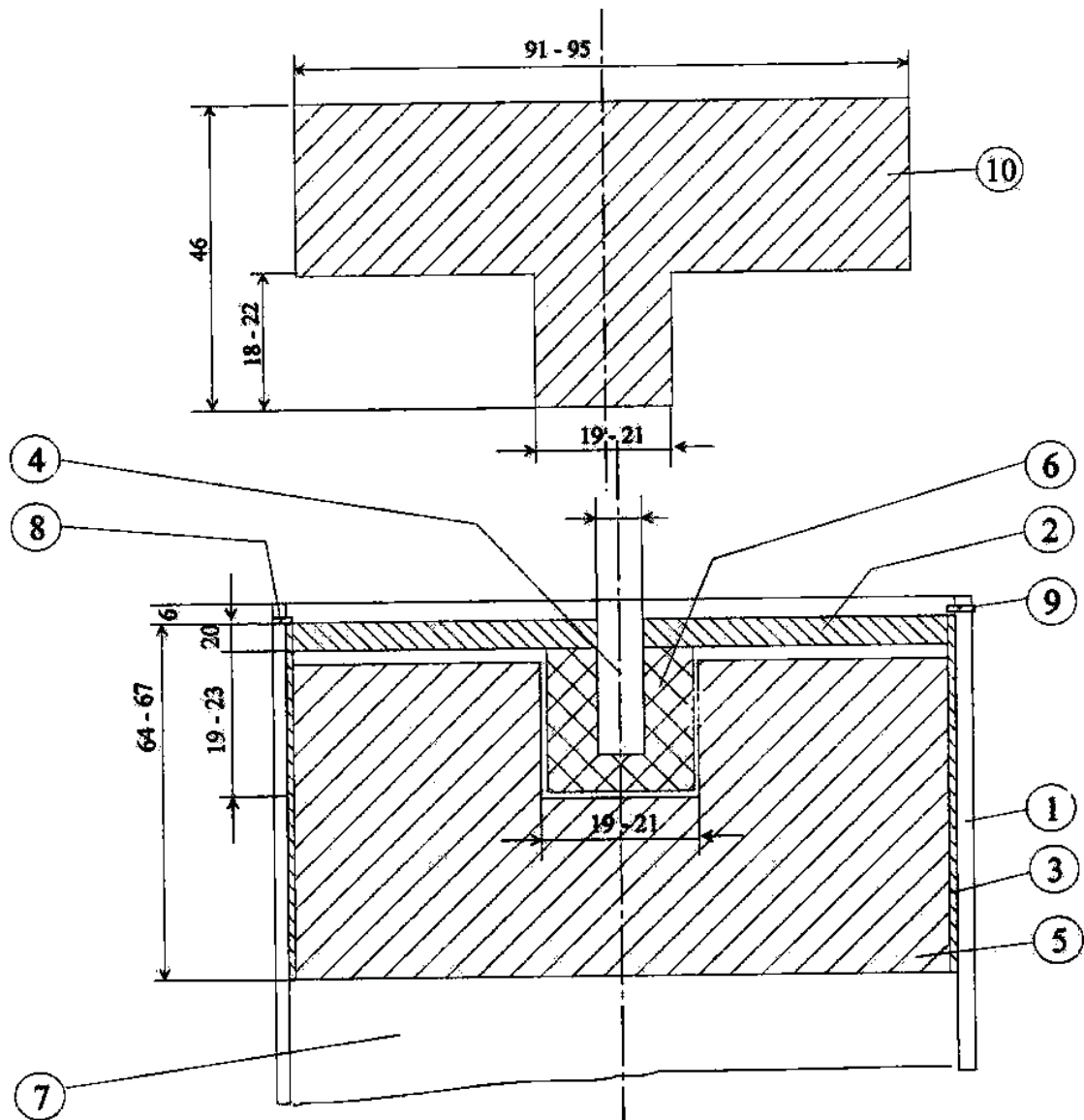
Insert the plastic explosive into the tube and press it down with a wooden die. Place the compressed pallet centrally in the recess within the plastic explosive. Close it with a wooden disc so that it remains in contact with the test sample. Lay the test tube horizontally on the 6 lead cylinders placed at 150 mm intervals (centric), with the centre of the last cylinder 75 mm from the bottom plate, on a firm, level, solid surface that is resistant to deformation or displacement. Insert the electrical blasting cap or the detonating cord.

Ensure that all necessary safety precautions are taken, connect and detonate the explosive.

Record, for each of the lead cylinders, the degree of compression expressed as a percentage of the original height of 100 mm. For oblique compression, the deformation is taken as the average of the maximum and minimum deformation.

## **5.5 Results**

The test is to be carried out twice. If in each test one or more of the supporting lead cylinders are crushed by less than 5%, the sample is deemed to satisfy the resistance to detonation requirements.



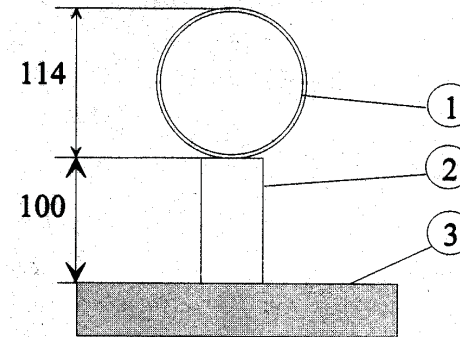
Dimensions in mm

- |   |                               |   |   |
|---|-------------------------------|---|---|
| ① | Steel tube                    | ⑥ | Compressed pellet                                   |
| ② | Wooden disc                   | ⑦ | Test sample   |
| ③ | Plastic or cardboard cylinder | ⑧ | 4-mm diameter hole drilled to receive split pin (9) |
| ④ | Wooden rod                    | ⑨ | Split pin   |
| ⑤ | Plastic explosive             | ⑩ | Wooden die for (5) diameter as for detonator        |

**Figure 1: *Booster charge***

- ① Steel tube
- ② Lead cylinders
- ③ Steel block
- ④ Bottom plate
- ⑤ Booster charge

1 to 6 numbers of lead cylinders



dimensions in mm

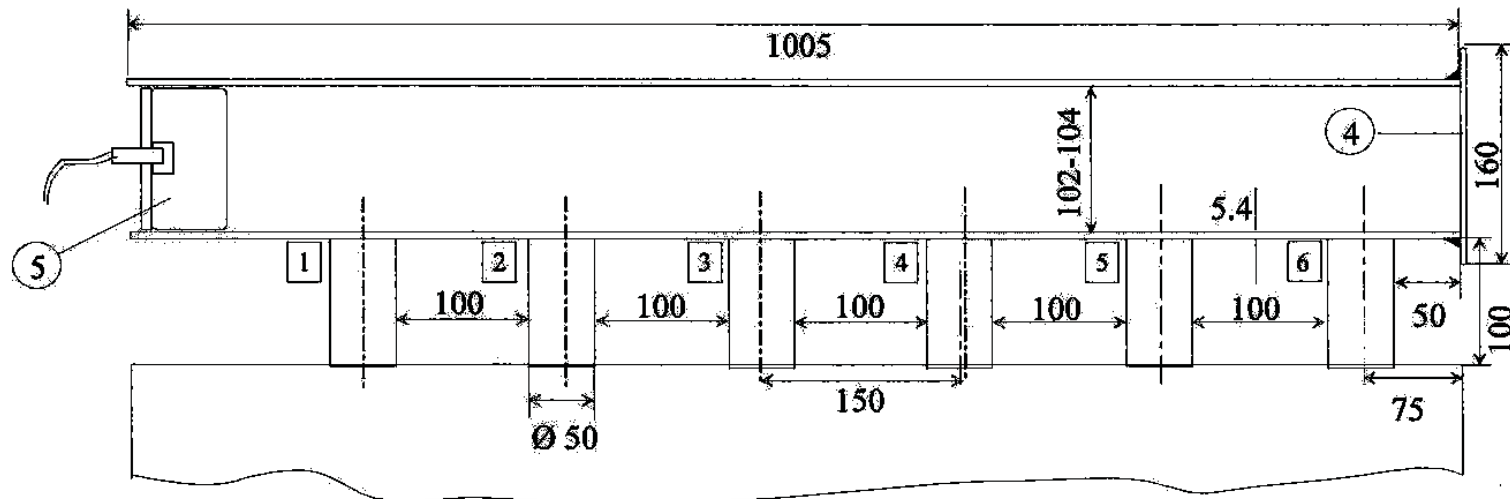


Figure 2: Positioning of the steel tube on the firing site

## **6 Self-heating test for charcoal**

### **6.1 Apparatus**

6.1.1 *Oven.* A laboratory oven fitted with internal air circulation and capable of being controlled at  $140^{\circ}\text{C} \pm 2^{\circ}\text{C}$ .

6.1.2 *Wire mesh cube.* Construct an open-top cube, 100 mm side, from phosphor bronze gauze 18,000 mesh per square centimetre (350 x 350 mesh). Insert it inside a slightly larger, well-fitting cube, made of phosphor bronze gauze 11 mesh per square centimetre (8 x 8 mesh). Fit the outer cube with a handle or hooks so that it can be suspended from above.

6.1.3 *Temperature measurement.* A suitable system to measure and record the temperature of the oven and in the centre of the cube. "Chromel-alumel" thermocouples, made from 0.27 mm diameter wire, are suitable for measuring the temperature range expected.

### **6.2 Procedure**

6.2.1 Fill the cube with carbon and tap down gently, adding carbon until the cube is full. Suspend the sample in the centre of the oven which has been preheated to  $140^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . Insert one of the thermocouples in the centre of the sample and the other between the cube and the oven wall. Maintain the temperature of the oven at  $140^{\circ}\text{C} \pm 2^{\circ}\text{C}$  for 12 hours and record the oven temperature and the sample temperature.

### **6.3 Results**

6.3.1 Non-activated carbon, non-activated charcoal, carbon black and lamp black fail the test if the temperature at any time during the 12 hours exceeded  $200^{\circ}\text{C}$ .

6.3.2 Activated carbon and activated charcoal fail the test if the temperature at any time during the 12 hours exceeded  $400^{\circ}\text{C}$ .