

INTERNATIONAL
STANDARD
NORME
INTERNATIONALE

IEC
CEI

60970

Second edition
Deuxième édition
2007-07

**Insulating liquids – Methods for counting and
sizing particles**

STANDARD PREVIEW
**Isolants liquides – Méthodes de détermination du
nombre et de la taille des particules**

[IEC 60970:2007](https://standards.iteh.ai/catalog/standards/sist/b768d81b-d063-478d-939f-3283d89cb64/iec-60970-2007)

<https://standards.iteh.ai/catalog/standards/sist/b768d81b-d063-478d-939f-3283d89cb64/iec-60970-2007>



Reference number
Numéro de référence
IEC/CEI 60970:2007



THIS PUBLICATION IS COPYRIGHT PROTECTED

Copyright © 2007 IEC, Geneva, Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either IEC or IEC's member National Committee in the country of the requester.

If you have any questions about IEC copyright or have an enquiry about obtaining additional rights to this publication, please contact the address below or your local IEC member National Committee for further information.

Droits de reproduction réservés. Sauf indication contraire, aucune partie de cette publication ne peut être reproduite ni utilisée sous quelque forme que ce soit et par aucun procédé, électronique ou mécanique, y compris la photocopie et les microfilms, sans l'accord écrit de la CEI ou du Comité national de la CEI du pays du demandeur.

Si vous avez des questions sur le copyright de la CEI ou si vous désirez obtenir des droits supplémentaires sur cette publication, utilisez les coordonnées ci-après ou contactez le Comité national de la CEI de votre pays de résidence.

IEC Central Office
3, rue de Varembe
CH-1211 Geneva 20
Switzerland
Email: inmail@iec.ch
Web: www.iec.ch

About the IEC

The International Electrotechnical Commission (IEC) is the leading global organization that prepares and publishes International Standards for all electrical, electronic and related technologies.

About IEC publications

The technical content of IEC publications is kept under constant review by the IEC. Please make sure that you have the latest edition, a corrigenda or an amendment might have been published.

- Catalogue of IEC publications: www.iec.ch/searchpub

The IEC on-line Catalogue enables you to search by a variety of criteria (reference number, text, technical committee,...). It also gives information on projects, withdrawn and replaced publications.

- IEC Just Published: www.iec.ch/online_news/justpub

Stay up to date on all new IEC publications. Just Published details twice a month all new publications released. Available on-line and also by email.

- Customer Service Centre: www.iec.ch/webstore/custserv

If you wish to give us your feedback on this publication or need further assistance, please visit the Customer Service Centre FAQ or contact us:

Email: csc@iec.ch
Tel.: +41 22 919 02 11
Fax: +41 22 919 03 00

A propos de la CEI

La Commission Electrotechnique Internationale (CEI) est la première organisation mondiale qui élabore et publie des normes internationales pour tout ce qui a trait à l'électricité, à l'électronique et aux technologies apparentées.

A propos des publications CEI

Le contenu technique des publications de la CEI est constamment revu. Veuillez vous assurer que vous possédez l'édition la plus récente, un corrigendum ou amendement peut avoir été publié.

- Catalogue des publications de la CEI: www.iec.ch/searchpub/cur_fut-f.htm

Le Catalogue en-ligne de la CEI vous permet d'effectuer des recherches en utilisant différents critères (numéro de référence, texte, comité d'études,...). Il donne aussi des informations sur les projets et les publications retirées ou remplacées.

- Just Published CEI: www.iec.ch/online_news/justpub

Restez informé sur les nouvelles publications de la CEI. Just Published détaille deux fois par mois les nouvelles publications parues. Disponible en-ligne et aussi par email.

- Service Clients: www.iec.ch/webstore/custserv/custserv_entry-f.htm

Si vous désirez nous donner des commentaires sur cette publication ou si vous avez des questions, visitez le FAQ du Service clients ou contactez-nous:

Email: csc@iec.ch
Tél.: +41 22 919 02 11
Fax: +41 22 919 03 00

INTERNATIONAL
STANDARD
NORME
INTERNATIONALE

IEC
CEI

60970

Second edition
Deuxième édition
2007-07

**Insulating liquids – Methods for counting and
sizing particles**

STANDARD PREVIEW
**Isolants liquides – Méthodes de détermination du
nombre et de la taille des particules**

IEC 60970:2007

<https://standards.iteh.ai/catalog/standards/sist/b768d81b-d063-478d-939f-328Bd89cb64/iec-60970-2007>



Commission Electrotechnique Internationale
International Electrotechnical Commission
Международная Электротехническая Комиссия

PRICE CODE
CODE PRIX

R

*For price, see current catalogue
Pour prix, voir catalogue en vigueur*

CONTENTS

FOREWORD.....	3
INTRODUCTION.....	5
1 Scope.....	6
2 Normative references	6
3 General caution, health, safety and environmental protection	6
4 Significance.....	7
5 Comparison and limitation of the methods	7
6 Types and identification of particles.....	8
7 Sampling	8
7.1 General remarks.....	8
7.2 Sampling vessels	9
7.3 Cleaning of sampling bottles	9
7.4 General directions for sampling	9
7.5 Sampling procedure	10
7.6 Labelling of samples.....	10
7.6.1 Samples from tanks.....	10
7.6.2 Samples from electrical equipment.....	10
8 Preparation of the samples for analysis.....	10
9 Method A – Automatic particle size analyzer.....	11
9.1 Summary of method	11
9.2 Apparatus and auxiliary materials.....	11
9.3 Calibration procedures	11
9.4 Preparation of the apparatus for counting.....	12
9.5 Preparation of sample before counting	12
9.6 Preparation of sample for counting.....	12
9.7 Counting procedures	12
9.8 Report.....	13
9.9 Precision	13
9.10 Repeatability	13
9.11 Reproducibility	13
10 Method B – Optical microscopy	14
10.1 Principle.....	14
10.2 Procedure by transmitted light.....	14
10.3 Procedure by incident light	14
Annex A (informative) Use of syringes as sampling vessels	15
Annex B (informative) Calibration of the automatic particle counters.....	17
Bibliography.....	18

INTERNATIONAL ELECTROTECHNICAL COMMISSION

INSULATING LIQUIDS – METHODS FOR COUNTING AND SIZING PARTICLES

FOREWORD

- 1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as “IEC Publication(s)”). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
- 2) The formal decisions or agreements of IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested IEC National Committees.
- 3) IEC Publications have the form of recommendations for international use and are accepted by IEC National Committees in that sense. While all reasonable efforts are made to ensure that the technical content of IEC Publications is accurate, IEC cannot be held responsible for the way in which they are used or for any misinterpretation by any end user.
- 4) In order to promote international uniformity, IEC National Committees undertake to apply IEC Publications transparently to the maximum extent possible in their national and regional publications. Any divergence between any IEC Publication and the corresponding national or regional publication shall be clearly indicated in the latter.
- 5) IEC provides no marking procedure to indicate its approval and cannot be rendered responsible for any equipment declared to be in conformity with an IEC Publication.
- 6) All users should ensure that they have the latest edition of this publication.
- 7) No liability shall attach to IEC or its directors, employees, servants or agents including individual experts and members of its technical committees and IEC National Committees for any personal injury, property damage or other damage of any nature whatsoever, whether direct or indirect, or for costs (including legal fees) and expenses arising out of the publication, use of, or reliance upon, this IEC Publication or any other IEC Publications.
- 8) Attention is drawn to the Normative references cited in this publication. Use of the referenced publications is indispensable for the correct application of this publication.
- 9) Attention is drawn to the possibility that some of the elements of this IEC Publication may be the subject of patent rights. IEC shall not be held responsible for identifying any or all such patent rights.

International Standard IEC 60970 has been prepared by IEC technical committee 10: Fluids for electrotechnical applications.

This second edition cancels and replaces the first edition published in 1989. This edition constitutes a technical revision.

The significant technical changes with respect to the previous edition are as follows:

- new calibration procedures for automated laser particle;
- three figures contamination code;
- new procedure of sample pre-treatment when automated laser counter method are used.

The text of this standard is based on the following documents:

FDIS	Report on voting
10/695/FDIS	10/714/RVD

Full information on the voting for the approval of this standard can be found in the report on voting indicated in the above table.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

The committee has decided that the contents of this publication will remain unchanged until the maintenance result date indicated on the IEC web site under "<http://webstore.iec.ch>" in the data related to the specific publication. At this date, the publication will be

- reconfirmed;
- withdrawn;
- replaced by a revised edition, or
- amended.

iTeh STANDARD PREVIEW **(standards.iteh.ai)**

[IEC 60970:2007](#)

<https://standards.iteh.ai/catalog/standards/sist/b768d81b-d063-478d-939f-328Bd89cb64/iec-60970-2007>

INTRODUCTION

The first edition of this standard was published in 1989, and confirmed in 1996. The present edition has been found necessary for consistency with the new ISO 4406:1999, in which calibration procedures for automated particles counters have been changed from ACFTD standard to ISO-MTD standard. Specific procedures for sample preparation are described in more detail when automated particle counters are used. Results and ISO Code reporting are consistent with ISO 4406:1999 standard. Repeatability and reproducibility data are reported.

It has been demonstrated that particle contamination of insulating liquids used in electrical equipment have been responsible for major faults [1]¹. Particle analysis is recommended (as complementary test) by IEC 60422[3] for power transformers with nominal voltage above 170 kV[2].

Particle counting and sizing is usually carried out using automated counters; the calibration standard for these counters was changed in 1999. The ISO reporting code has also been changed from a two-figure to a three-figure code. This code gives information on three classes of cumulative counting: particles/ml with $\varnothing > 4 \mu\text{m}$, particles/ml with $\varnothing > 6 \mu\text{m}$, particles/ml with $\varnothing > 14 \mu\text{m}$. Particle analysis with automated particle counters has been thoroughly investigated to verify factors influencing the results and to optimize the analysis procedure. Reference figures for repeatability and Reproducibility are reported, for particle counting and for ISO Class.

Annex A provides information about sampling with syringes. Annex B reports a reference for ISO MTD calibration procedure.

(standards.iteh.ai)

[IEC 60970:2007](https://standards.iteh.ai/catalog/standards/sist/b768d81b-d063-478d-939f-328Bd89cb64/iec-60970-2007)

<https://standards.iteh.ai/catalog/standards/sist/b768d81b-d063-478d-939f-328Bd89cb64/iec-60970-2007>

¹ Figures in square brackets refer to the bibliography.

INSULATING LIQUIDS – METHODS FOR COUNTING AND SIZING PARTICLES

1 Scope

This standard describes the sampling procedures and methods for the determination of particle concentration and size distribution.

Three methods are specified. One uses an automatic particle size analyser, working on the light interruption principle. The other two use an optical microscope, in either the transmitted light or incident light mode, to count particles collected on the surface of a membrane filter. The optical microscope methods are described in ISO 4407.

All three methods are applicable to both used and unused insulating liquids.

Annex A contains an alternative sampling procedure using a syringe and Annex B reports a reference for the calibration of automatic particle counters.

NOTE 1 The methods are not intended to measure particulate matter in liquids containing sludge. While analysing solid content on oils containing sludge refers to method for sediment and sludge determination in IEC 60422, Annex C.

NOTE 2 The methods specified are only applicable to measurements related to a limited range of size and number.

2 Normative references

[IEC 60970:2007](#)

<https://standards.iteh.ai/catalog/standards/sist/b768d81b-d063-478d-939f>

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

IEC 60475: *Method of sampling liquid dielectrics*

ISO 4406: *Hydraulic fluid power – Fluids – Method for coding the level of contamination by solid particles*

ISO 4407: *Hydraulic fluid power – Fluid contamination – Determination of particulate contamination by the counting method using an optical microscope*

ISO 5884: *Aerospace – Fluid systems and components – Methods for sampling and measuring the solid particle contamination of hydraulic fluids*

EN 50353: *Insulating oil – Determination of fibre contamination by the counting method using a microscope*

3 General caution, health, safety and environmental protection

This International Standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of the standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

The insulating liquids which are the subject of this standard should be handled with due regard to personal hygiene. Direct contact with the eyes may cause irritation. In the case of eye contact, irrigation with copious quantities of clean running water should be carried out

and medical advice sought. Some of the tests specified in this standard involve the use of processes that could lead to a hazardous situation. Attention is drawn to the relevant standard for guidance.

This standard is applicable to insulating liquids and used sample containers, the disposal or decontamination of which must be done according to local regulations. Every precaution should be taken to prevent release of mineral oil into the environment.

4 Significance

It is well known that particles have a detrimental effect on the dielectric strength of insulating liquids. It has long been the practice to include in specifications of insulating liquids the requirement that the fluid be clear and free of visible particulate matter. However, there has been no standard method for quantitative estimates, so that practices have differed. This standard gives standard procedures for the test.

Filtration of insulating liquids is an established practice in the electrical industry. The procedure described may serve to assess the performance of the filter system. The results obtained are dependent upon the method used. With the automatic counter the measured values also depend on the calibration procedure and in particular on the calibration material. It is therefore essential that the methods of analysis and the calibration standards are specified when quoting results.

The particle content of a sample may depend on different transformer parameters as well as the condition of the oil itself.

Storage may affect the sample, due to sedimentation and/or coalescence of particles. Shaking of the sample before analysis will be necessary.

5 Comparison and limitation of the methods

Automatic particle counters using the light interruption principle are quick and easy to use, but the following points should be borne in mind:

- With some liquids it may be necessary to modify their viscosity to comply with the operating parameters of the instrument.
- It is necessary to choose a sensor head suitable for counting in the size range required. No single head can count both very small particles (<2 µm) and very large particles (>200 µm).
- The instrument records the light interruption area of the particle and from this calculates the diameter of a sphere having the equivalent area or the longer axis of a specified ellipsoid with the same area, as established by ISO 4407. When measurements are carried out with automatic particle counters, the reported sizes are expressed as µm(c) to indicate that the particle size has been calculated from the observed cross-sectional area. Particle sizes from optical microscope counting are expressed as µm. The relationship between the two units is described in ISO 4406:
 - 6 µm(c) corresponds to 5 µm
 - 14 µm(c) corresponds to 15 µm.
- Automatic counters give no information as to the shape of the particles, and this constitutes a limitation with respect to the recognition of fibres. Their narrow and elongated shape results in a slight light obscuration and consequently in a very small equivalent sphere diameter. The results obtained can be different from those obtained by microscope counting. When it is important to evaluate the concentration of fibres, automatic counters cannot perform this task adequately.
- When air-saturated or over-saturated liquids are shaken manually or in a shaking machine, or given high-energy ultrasonic treatment, finely dispersed micro-bubbles may be formed

in the liquid. In the optical system of the automatic counter, these micro-bubbles will be counted as solid particles.

- These difficulties are avoided when using either of the optical microscopic methods. In addition, optical microscopy may give some information about the types of particles present. These methods are, however, much more time-consuming and operator dependent and may be very difficult to count particles of less than 5 µm.

6 Types and identification of particles

The origins of particles found in insulating liquids are manifold.

In new, unenergized, equipment the insulating liquid may contain cellulose fibres plus particles from the manufacturing process. These could include iron, aluminium, brass, welding cinder and sandblasting materials.

Insulating liquids in working transformers, at both normal and overload temperatures, slowly acquire soot and sludge particles. Localised overheating over 500 °C could be a source of carbon. The carbon particles produced in the OLTC diverter may migrate by leakage, accidents or human error into the bulk fluid compartment and contaminate the full charge.

A typical source of metallic particles is from pump bearing wear, although corrosion and arcing on metallic components may also produce particles.

Cellulose lint, sand, dust and particles of varnish, plastic or rubber can also be found in the fluid of transformers in service.

A knowledge of which particle types are present in the insulating liquid can, in certain cases, help in assessing the conditions of the equipment, in diagnosing a fault or indicating a risk of failure. The most dangerous particles are the conductive ones (metals, carbon, wet fibres, etc.). Particle identification and counting have been found to be necessary procedures of condition monitoring (CIGRE brochures 157[1] and 227[2]).

Certain particles may be identified by filtering a sample through a membrane filter and examining the residue under a microscope (EN 50353). At this stage some fibres can be identified using the dispersion staining technique and a number of metals by means of spot tests or micro chemical methods. Metallic particles can be better identified and quantified by instrumental analytical methods such as atomic absorption spectroscopy (AAS), induced coupled plasma (ICP-AES), and wet chemical analysis. A detailed description of methods for the identification of particles is, however, outside the scope of this standard.

7 Sampling

7.1 General remarks

The sample taken should only be used for the particle count determination. Further analysis may be done on the residual sample, but after the particle count determination.

The particle content of a sample is also dependant on the sampling point, time elapsed since the transformer was filled, the circulation rate and the time that the transformer has been left to stand prior to sampling.

With used liquids, oxidation products which are soluble at operating temperatures may precipitate when the sample is allowed to stand at room temperature for a prolonged period. This process, which is dependent upon the service age of the liquid, the time between sampling and analysis and the storage temperature, can affect the particle count.

For the above mentioned reasons, sampling is the main source of spreading of results.

7.2 Sampling vessels

The sampling vessels recommended in this standard are cylindrical, flat-bottomed, wide-necked, clear glass bottles fitted with a polypropylene threaded cap forming a seal with the bottle without the use of any inserts.

When an automatic particle size analyzer is used, the volume sampled shall be enough to allow a proper rinsing of the instrument's dead volume and measuring cell before the analysis.

For microscopy, the bottles shall have at least 100 cm³ capacity and be permanently marked to indicate 100 cm³ sample size.

An alternative method for insulating liquids in service using syringes as sampling vessels is given in Annex A.

7.3 Cleaning of sampling bottles

It is recommended that the bottles be cleaned to achieve a blank count of less than 200 particles above 5 µm per 100 cm³. The test should be performed on the filtered solvent used in the last stage of the cleaning process.

A cleaning method is given for guidance, but other methods can be used provided they achieve a similar or greater degree of cleanliness.

- a) Wash with warm water containing a detergent
- b) Rinse with warm water and drain
- c) Rinse thoroughly with 0,45 µm membrane filtered acetone to remove water
- d) Rinse with 0,45 µm membrane filtered petroleum ether 40 °C to 70 °C or with another suitable solvent. Leave 1 cm³ or 2 cm³ of solvent in the bottle and close the bottle.

If ultrasonic agitation is used before counting particles, the cleaning procedure must include ultrasonic treatment. Reject the procedure outlined in stage c) and instead place the sample bottle, filled with 0,45 µm membrane filtered acetone, in an ultrasonic bath for 1 min.

A residue of solvent in the bottle creates a positive pressure in the bottle helping to prevent contamination from the atmosphere when opening the bottle.

Warning: Attention is called to national regulations associated with the use of solvents.

The use of purchased sample bottles cleaned in accordance with ISO 5884 is allowed.

7.4 General directions for sampling

It is difficult to obtain representative samples from a drum. If sampling is found to be necessary, the procedure given in Annex A may be used or, alternatively, the procedures given in IEC 60475. In the case of sealed power transformers and instrument transformers or similar equipment with small liquid volume, the manufacturer's instructions on sampling procedure and quantity shall be followed. A sample from a transformer should preferably be taken during fluid circulation or immediately afterwards. The analysis obtained may depend on the sampling point selected. Confirmatory or follow-up samples should therefore always be taken from the same point.

Every precaution shall be taken when sampling not to contaminate the sample.

Outdoor sampling of insulating liquids in rain, fog, snowfall or high wind is only permitted if all precautions are taken to avoid contamination of the samples. In this special case the use of a cover is necessary.

Ensure that sampling is done by an experienced person.

7.5 Sampling procedure

Prior to taking the sample, the exterior of the sampling valve and the adjacent parts shall be thoroughly cleaned using lint-free wiping materials.

- Connect a length of plastic tubing, which is resistant to the liquid being sampled, to the sampling valve.
- If the sampling bottle contains filtered solvent, it may be necessary at low ambient temperature to warm the bottle with the hands, in order to create a positive pressure and thereby prevent the ingress of atmospheric particulate matter.

Warning: Petroleum ether, as well as many of its alternatives, is a highly flammable solvent, and appropriate safety measures must be observed when the bottle is opened.

- Thoroughly flush the valve and by draining a sufficient quantity of liquid into a waste container. The adequate draining volume depends on the total oil volume of the equipment (for big power units 5 l is recommended).
- Remove the cap from the bottle; do not empty out the solvent. Without interrupting the flow from the valve, substitute the bottle for the waste container and collect the required volume of sample as quickly as possible. Then remove the sample bottle and replace the waste container.
- Replace the bottle cap without over tightening.
- Close the sampling valve and replace any protection. Label the sample.

The sample shall be protected against light during transportation and storage.

7.6 Labelling of samples

7.6.1 Samples from tanks

[IEC 60970:2007](https://standards.iteh.ai/catalog/standards/sist/b768d81b-d063-478d-939f-328fd89cb64/iec-60970-2007)

[https://standards.iteh.ai/catalog/standards/sist/b768d81b-d063-478d-939f-](https://standards.iteh.ai/catalog/standards/sist/b768d81b-d063-478d-939f-328fd89cb64/iec-60970-2007)

[328fd89cb64/iec-60970-2007](https://standards.iteh.ai/catalog/standards/sist/b768d81b-d063-478d-939f-328fd89cb64/iec-60970-2007)

Labels shall carry the following markings:

- tank identification;
- sampling point;
- type of insulating liquid;
- date of sampling.

7.6.2 Samples from electrical equipment

Labels shall carry the following markings:

- equipment identification;
- sampling point;
- equipment in factory/in service;
- liquid identification;
- temperature of liquid;
- date of sampling.

8 Preparation of the samples for analysis

Samples should be processed as soon as possible after sampling, because their long storage will generally lead to sedimentation of particles. Fine particles may also coalesce to form larger particles. For the above mentioned reasons, a shaking procedure will be necessary.

When automatic particle analysers are used, a detailed procedure for sample treatment is described in Clause 9 (Method A – Automatic particle size analyzer).

If high-energy ultrasonic treatment or higher shear mixing is used, there is a risk that the particle count determined will be enhanced owing to breakdown of sludge and other large particles and also that finely dispersed micro bubbles are formed in the liquid. If too high a vacuum is applied to air-saturated liquids, more micro bubbles may be released from the liquid.

NOTE It is advisable that the agitation and vacuum procedure developed be tested for effectiveness. This can be done by taking several samples simultaneously. One sample is tested immediately after sampling, with no agitation or vacuum treatment at all, another after treatment and a third after some storage time. The test should be applied to liquids with a varying degree of particle content, state of ageing and air content.

For optical microscopy systems shaking can be done manually, in a shaking machine, or by ultrasonic treatment (see Clause 10: Method B – Optical microscopy).

9 Method A – Automatic particle size analyzer

9.1 Summary of method

The sample is agitated to suspend the particles, then passed at an optimum flow rate through the sensor unit of the particle counter. After the required fluid volume has been passed through the sensor the count is terminated and the results recorded.

9.2 Apparatus and auxiliary materials

- An automatic particle counter fitted with a sensor operating on the light interruption principle and suitable for counting within the range of 2,0 µm to greater than 200 µm. The automatic particle counter shall be capable of:
 - Sorting particles into at least > 4 µm, > 6 µm, > 14 µm size ranges.
 - Providing a specific particle count and distribution for a measured quantity of reference material to a precision of ±10 % of the total count.
 - Not saturating (i.e. providing a lower than expected count as a result of particle coincidence within the sensing zone) when analysing a suspension containing less than 2 000 particles per millilitre of fluid.

NOTE Alternative detectors may be used to extend the size range of the particles measured.

- Compressed gas – a pressurized source of air or nitrogen free from oil or water contamination filtered through a 0,45 µm membrane filter. The capacity, pressure range and constant pressure controls should meet the requirements of the particular equipment in use.
- Solvent dispenser fitted with a 0,45 µm membrane filter at the outlet.
- Solvent – petroleum ether with a boiling-range of 40 °C to 70 °C, or suitable alternative filtered through a 0,45 µm membrane filter.
- Detergent – liquid, water soluble, commercial grade.
- Calibration standard ISO MTD.
- Ultrasonic bath.

9.3 Calibration procedures

The calibration of the instrument shall be carried out using one of the procedures given in Annex B.

Calibration should be advisably carried out annually or when any repair/change is made to the sensor or whenever results are suspect. It is recommended to check instrument's performance at least each 6 months, by analysing a standard sample of standard reference material (SRM). The results must be consistent with the reproducibility in 9.11.