



BSI Standards Publication

Explosive atmospheres

Part 20-1: Material characteristics for gas and vapour
classification — Test methods and data

National foreword

This British Standard is the UK implementation of EN ISO 80079-20-1:2019. It is identical to ISO/IEC 80079-20-1:2017, incorporating corrigendum July 2018. It supersedes BS EN 60079-20-1:2010, which is withdrawn.

The start and finish of text introduced or altered by corrigendum is indicated in the text by tags. Text altered by ISO/IEC corrigendum July 2018 is indicated in the text by AC1 AC1.

The UK participation in its preparation was entrusted to Technical Committee EXL/23, Explosion and fire precautions in industrial and chemical plant.

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

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

© The British Standards Institution 2019
Published by BSI Standards Limited 2019

ISBN 978 0 539 12202 2

ICS 29.260.20

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 November 2019.

Amendments/corrigenda issued since publication

Date	Text affected
30 November 2019	Implementation of CEN correction notice 20 November 2019: Supersession details added, ISO reference corrected

English Version

**Explosive atmospheres - Part 20-1: Material
characteristics for gas and vapour classification - Test
methods and data (ISO/IEC 80079-20-1:2017, including
Cor 1:2018)**

Atmosphères explosives - Partie 20-1 : Caractéristiques
des produits pour le classement des gaz et des vapeurs
- Méthodes et données d'essai (ISO/CEI 80079-20-
1:2017, y compris Cor 1:2018)

Explosionsfähige Atmosphären - Stoffliche
Eigenschaften zur Klassifizierung von Gasen und
Dämpfen - Teil 20-1: Prüfverfahren und Daten
(ISO/IEC 80079-20-1:2017, einschließlich Cor 1:2018)

This European Standard was approved by CEN on 8 January 2018.

This European Standard was corrected and reissued by the CEN-CENELEC Management Centre on 20 November 2019.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

European foreword

This document (EN ISO/IEC 80079-20-1:2019) has been prepared by Technical Committee ISO/TMB "Technical Management Board - groups" in collaboration with Technical Committee CEN/TC 305 "Potentially explosive atmospheres - Explosion prevention and protection" the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2020, and conflicting national standards shall be withdrawn at the latest by April 2022.

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

This document supersedes EN 60079-20-1:2010.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

For the relationship with EU Directive(s) see informative Annex ZA, which is an integral part of this document.

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

Endorsement notice

The text of ISO/IEC 80079-20-1:2017, including Cor 1:2018 has been approved by CEN as EN ISO/IEC 80079-20-1:2019 without any modification.

Annex ZA

(informative)

Relationship between this European Standard and the essential requirements of Directive 2014/34/EU aimed to be covered

This European Standard has been prepared under a Commission's standardization request M/BC/CEN/92/46 to provide one voluntary means of conforming to essential requirements of Directive 2014/34/EU "Directive 2014/34/EU Of The European Parliament And Of The Council of 26 February 2014 on the harmonisation of the laws of the Member States relating to equipment and protective systems intended for use in potentially explosive atmospheres (recast)".

Once this standard is cited in the Official Journal of the European Union under that Directive, compliance with the normative clauses of this standard given in Table ZA.1 confers, within the limits of the scope of this standard, a presumption of conformity with the corresponding essential requirements of that Directive, and associated EFTA regulations.

Table ZA.1 — Correspondence between this European Standard and Directive 2014/34/EU

Essential Requirements of Directive 2014/34/EU	Clause(s)/sub-clause(s) of this EN	Remarks/Notes
1.0.1	All clauses	

WARNING 1 — Presumption of conformity stays valid only as long as a reference to this European Standard is maintained in the list published in the Official Journal of the European Union. Users of this standard should consult frequently the latest list published in the Official Journal of the European Union.

WARNING 2 — Other Union legislation may be applicable to the product(s) falling within the scope of this standard.

This page deliberately left blank

CONTENTS

FOREWORD.....	5
1 Scope.....	7
2 Normative references	7
3 Terms and definitions	7
4 Classification of gases and vapours.....	9
4.1 General.....	9
4.2 Classification according to the maximum experimental safe gap (MESG).....	9
4.3 Classification according to the minimum igniting current ratio (MIC ratio).....	10
4.4 Classification according to the similarity of chemical structure	10
4.5 Classification of mixtures of gases	10
5 Data for flammable gases and vapours, relating to the use of equipment.....	11
5.1 Determination of the properties	11
5.1.1 General	11
5.1.2 Equipment group	11
5.1.3 Flammable limits.....	11
5.1.4 Flash point (FP).....	11
5.1.5 Temperature class	12
5.1.6 Minimum igniting current (MIC)	12
5.1.7 Auto-ignition temperature (AIT).....	12
5.2 Properties of particular gases and vapours	12
5.2.1 Coke oven gas.....	12
5.2.2 Ethyl nitrite	12
5.2.3 MESG of carbon monoxide	12
5.2.4 Methane, Equipment Group IIA.....	13
6 Method of test for the maximum experimental safe gap (MESG)	13
6.1 Outline of method	13
6.2 Test apparatus.....	13
6.2.1 General	13
6.2.2 Material and mechanical strength	14
6.2.3 Exterior chamber	14
6.2.4 Interior chamber	14
6.2.5 Gap adjustment	14
6.2.6 Injection of mixture	14
6.2.7 Position of ignition source.....	14
6.3 Procedure	14
6.3.1 Preparation of gas mixtures	14
6.3.2 Temperature and pressure.....	14
6.3.3 Gap adjustment	15
6.3.4 Ignition	15
6.3.5 Observation of the ignition process.....	15
6.4 Determination of maximum experimental safe gap (MESG).....	15
6.4.1 General	15
6.4.2 Preliminary tests.....	15
6.4.3 Confirmatory tests	15
6.4.4 Reproducibility of maximum experimental safe gaps (MESG).....	15
6.4.5 Tabulated values	16

6.5	Verification of the MESG determination method	16
7	Method of test for auto-ignition temperature (AIT).....	16
7.1	Outline of method	16
7.2	Apparatus	16
7.2.1	General	16
7.2.2	Test vessel and support.....	17
7.2.3	Thermocouples	17
7.2.4	Oven	17
7.2.5	Metering devices	18
7.2.6	Mirror	18
7.2.7	Timer	18
7.2.8	Equipment for purging the test vessel with air	18
7.2.9	Automated apparatus.....	18
7.3	Sampling, preparation and preservation of test samples	19
7.3.1	Sampling	19
7.3.2	Preparation and preservation.....	19
7.4	Procedure	19
7.4.1	General	19
7.4.2	Sample injection	20
7.4.3	Determination of the auto-ignition temperature (AIT).....	20
7.5	Auto-ignition temperature (AIT)	21
7.6	Validity of results	21
7.6.1	Repeatability	21
7.6.2	Reproducibility.....	21
7.7	Data.....	22
7.8	Verification of the auto-ignition temperature determination method	22
Annex A (normative)	Ovens of test apparatus for the tests of auto-ignition temperature	23
A.1	General.....	23
A.2	“IEC oven”	23
A.3	“DIN oven”	23
Annex B (informative)	Tabulated values	30
Annex C (informative)	Determination of cool flames	84
Annex D (informative)	Volume dependence of auto-ignition temperature.....	86
Bibliography		87
Figure 1	– Test apparatus	13
Figure A.1	– Test apparatus: assembly	24
Figure A.2	– Section A-A (flask omitted)	25
Figure A.3	– Base heater (board made of refractory material)	25
Figure A.4	– Flask guide ring (board made of refractory material)	26
Figure A.5	– Neck heater (board made of refractory material)	26
Figure A.6	– Oven.....	27
Figure A.7	– Lid of steel cylinder.....	28
Figure A.8	– Lid of steel cylinder.....	29
Figure A.9	– Injection of gaseous sample.....	29
Figure C.1	– Additional thermocouple to detect cool flames	84

Figure C.2 – ‘Negative temperature coefficient’ shown for butyl butyrate as an example	85
Figure D.1 – Volume dependence of auto-ignition temperature	86
Table 1 – Classification of temperature class and range of auto-ignition temperatures.....	12
Table 2 – Values for verification of the apparatus	16
Table 3 – Values for verification of the apparatus	22
Table B.1 – Material data.....	32

INTERNATIONAL ELECTROTECHNICAL COMMISSION

EXPLOSIVE ATMOSPHERES –

Part 20-1: Material characteristics for gas and vapour classification – Test methods and data

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 itself does not provide any attestation of conformity. Independent certification bodies provide conformity assessment services and, in some areas, access to IEC marks of conformity. IEC is not responsible for any services carried out by independent certification bodies.
- 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 ISO/IEC 80079-20-1 has been prepared by subcommittee 31M: Non-electrical equipment and protective systems for explosive atmospheres, of IEC technical committee 31: Equipment for explosive atmospheres.

This first edition of ISO/IEC 80079-20-1 cancels and replaces IEC 60079-20-1:2010.  It constitutes a technical revision. This edition includes the following significant technical changes with respect to the previous edition:

The classifications for the temperature class or equipment group were changed in the informative Annex B for the following materials: CAS-No. 64-17-5; 78-93-3; 107-31-3:

- For dry Ethanol (CAS-No. 64-17-5), equipment group remains IIB, but for Ethanol in air with $\geq 4\%$ water vapour, as in common atmospheres, a second line has been added to indicate IIA;

- For 2-Butanone (CAS-No. 78-93-3), re-measurement indicated change from IIB to IIA was needed; and
- For Formic Acid Methyl Ester (CAS-No. 107-31-3), re-measurement indicated change from T2 to T1 was justified.

No significant changes were made with respect to the normative text of IEC 60079-20-1:2010. AC1

It is published as a double logo standard.

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

FDIS	Report on voting
31M/122/FDIS	31M/126/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.

A list of all parts in the IEC 60079 series, under the general title: *Explosive atmospheres*, as well as the International Standard 80079 series, can be found on the IEC website.

The committee has decided that the contents of this publication will remain unchanged until the stability date indicated on the IEC website 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.

The contents of the corrigendum of July 2018 have been included in this copy.

IMPORTANT – The 'colour inside' logo on the cover page of this publication indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

EXPLOSIVE ATMOSPHERES –

Part 20-1: Material characteristics for gas and vapour classification – Test methods and data

1 Scope

This part of ISO/IEC 80079 provides guidance on classification of gases and vapours. It describes a test method intended for the measurement of the maximum experimental safe gaps (MESG) for gas-air mixtures or vapour-air mixtures under normal conditions of temperature and pressure (20 °C, 101,3 kPa) so as to permit the selection of an appropriate group of equipment. This document also describes a test method intended for use in the determination of the auto-ignition temperature (AIT) of a vapour-air mixture or gas-air mixture at atmospheric pressure, so as to permit the selection of an appropriate temperature class of equipment.

Values of chemical properties of materials are provided to assist in the selection of equipment to be used in hazardous areas. Further data may be added as the results of validated tests become available.

The materials and the characteristics included in a table (see Annex B) have been selected with particular reference to the use of equipment in hazardous areas. The data in this document have been taken from a number of references which are given in the bibliography.

These methods for determining the MESG or the AIT may also be used for gas-air-inert mixtures or vapour-air-inert mixtures. However, data on air-inert mixtures are not tabulated.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 60050-426, *International Electrotechnical Vocabulary – Part 426: Electrical apparatus for explosive atmospheres* (available at <http://www.electropedia.org/>)

IEC 60079-11, *Explosive atmospheres – Part 11: Equipment protection by intrinsic safety "i"*

IEC 60079-14, *Explosive atmospheres – Part 14: Electrical installations design, selection and erection*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in IEC 60050-426 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

3.1

auto-ignition

reaction which is evidenced by a clearly perceptible flame and/or explosion, and for which the ignition delay time does not exceed 5 min

Note 1 to entry: See 7.2.2 for a test method.

3.2

ignition delay time

time between the completed injection of the flammable material and the ignition

3.3

auto-ignition temperature

AIT

lowest temperature (of a surface) at which under specified test conditions an ignition of a flammable gas or vapour in mixture with air or air-inert gas occurs

Note 1 to entry: See Clause 7 for a test method.

3.4

maximum experimental safe gap

MESG

maximum gap of a joint of 25 mm in width which prevents any transmission of an explosion during tests made under the conditions specified in this document

Note 1 to entry: See Clause 6 for a test method.

3.5

minimum igniting current $\langle AC_1 \rangle$

MIC

minimum current in a specified test circuit that causes the ignition of the explosive test mixture in the spark test apparatus according to IEC 60079-11

Note 1 to entry: See 5.1.6 for the test circuit.

3.6

flammable limits

lower flammable limit (LFL) and upper flammable limit (UFL) of gas in a gas-air mixture, between which a flammable mixture is formed

Note 1 to entry: The term “explosive limits” is used especially in European standardization and regulations interchangeably to describe these limits.

Note 2 to entry: The concentration can be expressed as either a volume fraction or a mass per unit volume.

3.6.1

lower flammable limit

LFL

concentration of flammable gas or vapour in air, below which an explosive gas atmosphere does not form

Note 1 to entry: For the purposes of Ex Equipment, this was previously referred to as the lower explosive limit (LEL).

Note 2 to entry: The concentration can be expressed as either a volume fraction or a mass per unit volume.

3.6.2

upper flammable limit

UFL

concentration of flammable gas or vapour in air, above which an explosive gas atmosphere does not form

Note 1 to entry: For the purposes of Ex Equipment, this was previously referred to as the upper explosive limit (UEL).

Note 2 to entry: The concentration can be expressed as either a volume fraction or a mass per unit volume.

3.7 equipment grouping

classification system of equipment related to the explosive atmosphere for which they are intended to be used

Note 1 to entry: IEC 60079-0 identifies three equipment groups:

Group I – equipment for mines susceptible to fire damp;

Group II, which is sub-divided into groups IIA, IIB and IIC – equipment for all places with an explosive gas atmosphere other than mines susceptible to fire damp;

Group III, which is sub-divided into groups IIIA, IIIB and IIIC – equipment for all places with an explosive dust atmosphere other than mines susceptible to fire damp.

3.8 flash point FP

lowest liquid temperature at which, under specified test conditions, a liquid gives off vapours in quantity such as to be capable of forming an ignitable vapour-air mixture

3.9 gas

gaseous phase of a substance that cannot reach equilibrium with its liquid or solid state in the temperature and pressure range of interest

Note 1 to entry: This is a simplification of the scientific definition, and merely requires that the substance is above its boiling point or sublimation point at the ambient temperature and pressure.

3.10 vapour

gaseous phase of a substance that can reach equilibrium with its liquid or solid state in the temperature and pressure range of interest

Note 1 to entry: This is a simplification of the scientific definition, and merely requires that the substance is below its boiling point or sublimation point at the ambient temperature and pressure.

4 Classification of gases and vapours

4.1 General

Equipment Group I addresses mines susceptible to firedamp.

NOTE Firedamp consists mainly of methane, but always contains small quantities of other gases, such as nitrogen, carbon dioxide, and hydrogen, and sometimes ethane and carbon monoxide. The terms firedamp and methane are used frequently in mining practice as synonyms.

Equipment Group II addresses flammable gases and vapours other than in mines susceptible to firedamp. Equipment Group II gases and vapours are classified according to their MESG and/or MIC ratio into equipment groups IIA, IIB and IIC.

All flammable materials are classified according to their AIT into temperature classes.

4.2 Classification according to the maximum experimental safe gap (MESG)

Gases and vapours may be classified according to their MESG into Equipment Groups IIA, IIB or IIC, based on the determination method described in this document. In order to ensure standardized results the MESG apparatus is dimensioned to avoid the possible effects of obstruction on the safe gaps.

NOTE 1 The standard method for determining MESG is described in 6.2, but where determinations have been undertaken only in an 8 l spherical vessel with ignition close to the flange gap these can be accepted provisionally.

NOTE 2 The design of the test apparatus for safe gap determination, other than that used for selecting the appropriate equipment group of enclosure for a particular gas, may need to be different to the one described in this document. For example, the volume of the enclosure, flange width, gas concentrations and the distance between the flanges and any external wall or obstruction may have to be varied. As the design depends on the particular investigation which is to be undertaken, it is impracticable to recommend specific design requirements, but for most applications the general principles and precautions indicated in this document will still apply.

NOTE 3 In IEC 60079-14 minimum distances of obstruction from the flameproof flange joints related to the equipment group of the hazardous area are given.

For the purpose of classification the MESG limits are:

Equipment Group IIA: $\text{MESG} \geq 0,90 \text{ mm}$.

Equipment Group IIB: $0,50 \text{ mm} < \text{MESG} < 0,90 \text{ mm}$.

Equipment Group IIC: $\text{MESG} \leq 0,50 \text{ mm}$.

Determination of both the MESG and MIC ratio is required when $0,50 < \text{MESG} < 0,55$. Then the equipment group is determined by MIC ratio,

NOTE 4 For gases and highly volatile liquids, the MESG is determined at 20 °C.

NOTE 5 If it was necessary to do the MESG determination at temperatures higher than ambient temperature a temperature 5 K above that needed to give the necessary vapour pressure or 50 K above the flash point is used and this value of MESG is given in the table and the classification of the equipment group is based on this result.

4.3 Classification according to the minimum igniting current ratio (MIC ratio)

Gases and vapours may be classified according to the ratio of their minimum igniting currents (MIC) to the ignition current of laboratory methane into Equipment Groups IIA, IIB or IIC. The purity of laboratory methane shall be not less than 99,9 % by volume.

NOTE The standard method of determining MIC ratios is with the apparatus described in IEC 60079-11, but where determinations have been undertaken in other apparatus these can be accepted provisionally.

For the purpose of classification the MIC ratios are:

Equipment Group IIA: $\text{MIC} > 0,80$.

Equipment Group IIB: $0,45 \leq \text{MIC} \leq 0,80$.

Equipment Group IIC: $\text{MIC} < 0,45$.

Determination of both the MESG and MIC ratio is required when $0,70 < \text{MIC} < 0,90$ or $0,40 < \text{MIC} < 0,50$. Then the equipment group is determined by MESG.

4.4 Classification according to the similarity of chemical structure

When a gas or vapour is a member of a homologous series of compounds, the classification of the gas or vapour can provisionally be inferred from the data of the neighbouring members of the series.

The classification according to the similarity of chemical structure is not allowed if the classification of one of the neighbouring members is based on MESG and the other on MIC ratio.

4.5 Classification of mixtures of gases

Mixtures of gases should generally be allocated to an equipment group only after a special determination of MESG or MIC ratio. One method to estimate the equipment group is to determine the MESG of the mixture by applying a form of Le Châtelier's principle:

$$MESG_{\text{mix}} = \frac{1}{\sum_i \left(\frac{X_i}{MESG_i} \right)}$$

Where X_i is the percentage by volume of material i and $MESG_i$ is the MESG of material i .

This method should not be applied in case of exceptions to the Le Châtelier's principle and to mixtures and/or streams that have:

- a) acetylene or its equivalent hazard (e.g. self-decomposition properties);
- b) oxygen or other strong oxidizer as one of the components;
- c) large concentrations (over 5 % by volume) of carbon monoxide. Because unrealistically high MESG values may result, caution should be exercised with two component mixtures where one of the components is an inert, such as nitrogen.

For mixtures containing an inert such as nitrogen in concentrations less than 5 % by volume, use an MESG of infinity. For mixtures containing an inert such as nitrogen in concentrations 5 % by volume and greater, use an MESG of 2.

NOTE An alternate method that includes stoichiometric ratios is presented in the essay "Maximum experimental safe gap of binary and ternary mixtures," by Brandes and Redeker.

5 Data for flammable gases and vapours, relating to the use of equipment

5.1 Determination of the properties

5.1.1 General

The compounds listed in this document are in accordance with Clause 4, or have physical properties similar to those of other compounds in that list.

5.1.2 Equipment group

The equipment groups are the result of MESG or MIC ratio determination except where there is no value listed for MESG or MIC ratio. For these, the equipment group is based on chemical similarity (see Clause 4).

NOTE If it was necessary to do the MESG determination at temperatures higher than ambient temperature, a temperature 5 K above that needed to give the necessary vapour pressure or 50 K above the Flash Point is used. This value of MESG is given in Table B.1 and the classification of the equipment group is based on this result.

5.1.3 Flammable limits

Determinations have been made by a number of different methods, but the preferred method is with a low energy ignition at the bottom of a vertical tube. The values (in percentage by volume and mass per volume) are listed in Table B.1.

If the flash point is high, the compound does not form a flammable vapour air/mixture at normal condition of temperature (20 °C). Where flammability data are presented for such compounds the determinations have been made at a temperature sufficiently elevated to allow the vapour to form a flammable mixture with air.

5.1.4 Flash point (FP)

The value given in Table B.1 is the "closed cup" measurement. When this data was not available, the "open cup" value is quoted and indicated by (oc). The symbol < (less than), indicates that the flash point is below the value (in degree Celsius) stated, this probably being the limit of the apparatus used.

5.1.5 Temperature class

The temperature class of a gas or vapour is given according to IEC 60079-14, as shown in Table 1:

Table 1 – Classification of temperature class and range of auto-ignition temperatures

Temperature class	Range of auto-ignition temperature (AIT) °C
T1	> 450
T2	$300 < \text{AIT} \leq 450$
T3	$200 < \text{AIT} \leq 300$
T4	$135 < \text{AIT} \leq 200$
T5	$100 < \text{AIT} \leq 135$
T6	$85 < \text{AIT} \leq 100$

5.1.6 Minimum igniting current (MIC)

The apparatus for the determination of minimum igniting current is defined in IEC 60079-11. The test apparatus shall be operated in a 24 V d.c. circuit containing a (95 ± 5) mH air-cored coil. The current in this circuit is varied to a minimum value until ignition of the most easily ignited concentration of the specific gas or vapour in air is obtained.

5.1.7 Auto-ignition temperature (AIT)

The value of auto-ignition temperature depends on the method of testing. The preferred method and data obtained is given in Clause 7 and in Annex A.

If the compound is not included in these data, the data obtained in similar apparatus, such as the apparatus described by ASTM E659, is listed.

NOTE Results from using the apparatus described in ASTM D2155 (now replaced by ASTM E659) were reported by C.J. Hilado and S.W. Clark. The apparatus is similar to the one used by Zabetakis. If there is no determination by either the IEC apparatus, nor similar apparatus, the lowest value obtained in other apparatus is listed. A more comprehensive list of data for auto-ignition temperature, with the reference to sources, is given by Hilado and Clark.

5.2 Properties of particular gases and vapours

5.2.1 Coke oven gas

Coke oven gas is a mixture of hydrogen, carbon monoxide and methane. If the sum of the concentrations (Vol. %) of hydrogen and carbon monoxide is less than 75 % by volume of the total, flameproof equipment of Equipment Group IIB is recommended; otherwise, equipment of Equipment Group IIC is recommended.

5.2.2 Ethyl nitrite

The auto-ignition temperature of ethyl nitrite is 95 °C, above which the gas suffers explosive decomposition.

NOTE Ethyl nitrite is not be confused with its isomer, nitroethane.

5.2.3 MESG of carbon monoxide

The MESG for carbon monoxide relates to a mixture with air saturated with moisture at normal ambient temperature. This determination indicates the use of Equipment Group IIB equipment.

in the presence of carbon monoxide. A larger MESG may be observed with less moisture. The lowest MESG (0,65 mm) is observed for a mixture of CO/H₂O near 7:1 mole ratio. Small quantities of hydrocarbon in the carbon monoxide-air mixture have a similar effect in reducing the MESG so that Equipment Group IIB equipment is required.

5.2.4 Methane, Equipment Group IIA

Industrial methane, such as natural gas, is classified as Equipment Group IIA, provided it does not contain more than 25 % by volume of hydrogen. A mixture of methane with other compounds from Equipment Group IIA, in any proportion, is classified as Equipment Group IIA.

6 Method of test for the maximum experimental safe gap (MESG)

6.1 Outline of method

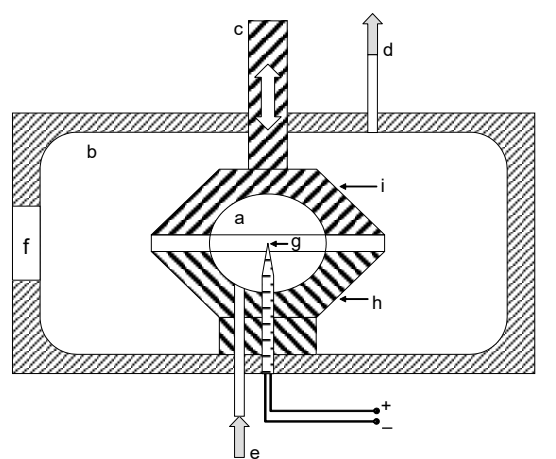
The interior and exterior chambers of the test apparatus are filled with a known mixture of the gas or vapour in air, under normal conditions of temperature and pressure (20 °C, 101,3 kPa) and with the circumferential gap between the two chambers accurately adjusted to the desired value. The internal mixture is ignited and the flame propagation, if any, is observed through the windows in the external chamber. The maximum experimental safe gap for the gas or vapour is determined by adjusting the gap in small steps to find the maximum value of gap which prevents ignition of the external mixture, for any concentration of the gas or vapour in air.

NOTE An exception is made for substances with vapour pressures which are too low to permit mixtures of the required concentrations to be prepared at normal ambient temperatures. For these substances, a temperature 5 K above that needed to give the necessary vapour pressure or 50 K above the flash point is used.

6.2 Test apparatus

6.2.1 General

The apparatus is described in the following subclauses and is shown schematically in Figure 1. It is also possible to use an automatic set-up when it is proven that the same results are obtained as with a manual apparatus.



IEC

Key

- | | | | |
|---|--------------------------------|---|-----------------------------|
| a | interior spherical chamber | f | observation windows |
| b | exterior cylindrical enclosure | g | spark electrode |
| c | adjustable part | h | lower gap plate, fixed |
| d | outlet of mixture | i | upper gap plate, adjustable |
| e | inlet of mixture | | |

Figure 1 – Test apparatus

6.2.2 Material and mechanical strength

The whole apparatus is constructed to withstand a maximum pressure of 1 500 kPa without significant expansion of the gap, so that no such expansion of the gap will occur during an explosion.

The main parts of the test apparatus, and in particular the walls and flanges of the inner chamber and the electrodes of the spark-gap, are normally of stainless steel. Other materials may have to be used with some gases or vapours, however, in order to avoid corrosion or other chemical affects. Light alloys should not be used for the spark-gap electrodes.

6.2.3 Exterior chamber

The exterior cylindrical enclosure "b" has a diameter of 200 mm and a height of 75 mm.

6.2.4 Interior chamber

The interior chamber "a" is a sphere with a volume measuring 20 cm³. The interior chamber is placed in the centre of the exterior chamber.

6.2.5 Gap adjustment

The two parts "i" and "h" of the internal chamber are so arranged that an adjustable 25 mm gap can be set up between the plane parallel faces of the opposing rims. The exact width of the gap can be adjusted by means of the micrometer (part "c").

6.2.6 Injection of mixture

The internal chamber is filled with the gas-air or vapour-air mixture through an inlet ("e"). The exterior chamber is filled with the mixture via the gap. The inlet and outlet should be protected by flame arresters.

6.2.7 Position of ignition source

The electrode "g" shall be mounted in such a way that the spark occurs in the centre of the internal chamber.

6.3 Procedure

6.3.1 Preparation of gas mixtures

As the consistency of the mixture concentration, for a particular test series, has a pronounced effect on the dispersion of the test results, it has to be carefully controlled. The flow of the mixture through the chamber is therefore maintained until the inlet and outlet concentrations are the same, or a method of equivalent reliability shall be used.

For the classification according to this document, the moisture content of the air used for the preparation of the mixture should not exceed 10 % relative humidity. Higher values of humidity could result for some substances in lower MESG values.

6.3.2 Temperature and pressure

The tests are made at an ambient temperature of $(20 \pm 5) ^\circ\text{C}$, except where otherwise permitted. The pressure within the test apparatus is adjusted to $(101,3 \pm 1) \text{ kPa}$.

NOTE An exception is made for substances with vapour pressures which are too low to permit mixtures of the required concentrations to be prepared at normal ambient temperatures. For these substances, a temperature 5 K above that needed to give the necessary vapour pressure or 50 K above the flash point is used.

6.3.3 Gap adjustment

The gap is first reduced to a very small value and examined to ensure that the flanges are parallel. The zero setting of the gap is checked but the value of torque applied should be low (e.g. a force of about 10^{-2} N applied at the circumference of the micrometer head).

6.3.4 Ignition

The internal mixture is ignited by an electrical spark generated by a high voltage transformer, with a root mean square of approximately 15 kV and a short circuit current of 30 mA. The spark discharge time shall be adjusted to 0,2 s.

6.3.5 Observation of the ignition process

Ignition of the internal mixture is confirmed by observation through the gap when the test is made. If no internal ignition occurs, the test is invalid. Ignition of the mixture in the external chamber is taken to occur when the whole volume of the chamber is seen to be filled by the flame of the explosion.

6.4 Determination of maximum experimental safe gap (MESG)

6.4.1 General

Mostly, the MESG is found at slightly rich mixtures, so it is recommended to start with the stoichiometric mixture.

6.4.2 Preliminary tests

With a defined mixture of the combustible vapour or gas with air, two ignition tests are carried out on a number of gaps, at 0,02 mm intervals, covering the range from a safe gap to an unsafe gap. From the results, the highest gap, g_0 , at which there is 0 % probability of ignition of the outer volume, and the lowest gap, g_{100} , giving 100 % probability of ignition of the outer volume, are determined.

The test series is repeated with a range of mixture concentrations, and the variation of the gap g_0 and g_{100} are obtained. The most incensive mixture is that for which these values are a minimum.

6.4.3 Confirmatory tests

The results are confirmed by repeating the tests, with 10 ignition tests for each step of gap adjustment, at different concentrations above and below the mixture found in the preliminary series. The minimum values of g_0 and g_{100} are then determined.

6.4.4 Reproducibility of maximum experimental safe gaps (MESG)

The highest acceptable difference between the values of $(g_0)_{\min}$ obtained from different test series is 0,04 mm.

If all values are within this range, the tabulated value of MESG will be equal to $(g_0)_{\min}$ where $(g_{100})_{\min} - (g_0)_{\min}$ is the smallest. For most substances, this difference will lie within one step of gap adjustment, i.e. within 0,02 mm.

If the difference between the values of $(g_0)_{\min}$ taken from different test series exceeds 0,04 mm, the laboratories concerned should repeat their tests after confirming that the test apparatus is able to reproduce the tabulated value for hydrogen.

6.4.5 Tabulated values

The values of the MESG, the difference $(g_{100})_{\min} - (g_0)_{\min}$ and the most incensive mixture determined in 6.4.1 are tabulated in Annex B.

The value of the MESG is used to determine the equipment group. The value $(g_{100})_{\min} - (g_0)_{\min}$ indicates the accuracy of the tabulated value of the MESG.

6.5 Verification of the MESG determination method

This verification procedure shall be used for new apparatus as well as for checking the performance of existing apparatus. Existing apparatus shall be checked at least every 12 months or whenever parts of the apparatus have been changed or renewed. For new apparatus, carry out experiments according to the instructions given in 6.3 with all the substances listed in Table 2. When renewing the test vessel it is in general sufficient to carry out the check test with methane and hydrogen.

Verification will be confirmed if the values obtained do not deviate more than $\pm 0,02$ mm from the values given in Table 2. The values are valid for an ambient temperature of (20 ± 2) °C and an ambient pressure of (100 ± 2) kPa.

If the results obtained by the test apparatus meet the required verification performance, record this fact in a permanent report.

Table 2 – Values for verification of the apparatus

Flammable substance	Concentration range Vol. %	MESG mm	Purity of substances mol %
Methane	8,0 to 10,0	1,16	99,995
Propane	3,5 to 4,5	0,92	99,95
Hydrogen	29,0 to 31,0	0,30	99,999

If the results obtained by the test apparatus do not meet the required verification performance, check the apparatus, especially the plane parallelism of the faces of the inner volume. The parallel offset of the faces has to be less than 0,01 mm for distances between 0,3 mm and 1,5 mm. If appropriate verify again.

7 Method of test for auto-ignition temperature (AIT)

7.1 Outline of method

A known volume of the product to be tested is injected into a heated open flask containing air. The contents of the flask are observed until ignition occurs. The test is repeated with different flask temperatures and different sample volumes. The lowest flask temperature at which ignition occurs is recorded as the auto-ignition temperature.

7.2 Apparatus

7.2.1 General

Historically, two apparatus types have been used: the IEC apparatus described in A.2 and the DIN apparatus described in A.3. The difference is that the IEC apparatus has an additional heater at the neck of the flask. Normally there is no impact on the test results. The principle of the test apparatus is described in the following subclauses. It is also possible to use an automatic set-up.

The test apparatus consists of:

- a test vessel;
- support for the test vessel;
- calibrated measuring thermocouple;
- an electrical hot-air oven;
- metering devices for metering the flammable substance;
- a mirror for observing the ignition;
- timer;
- equipment for cleaning the test vessel.

7.2.2 Test vessel and support

The test vessel shall be a 200 ml narrow-necked Erlenmeyer flask made of borosilicate glass according to ISO 1773. It shall be ensured that the inner surface of the bottom is flat. A clean flask shall be used for tests.

The support for the Erlenmeyer flask shall ensure that the heat dissipation via the support is as low as possible. If the support is mounted at the neck it shall be ensured that it does not use more than (5 ± 2) mm of the height of the neck of the Erlenmeyer flask (for example see Annex A).

If there is special need to know exactly the influence of volume on the auto-ignition temperature additional experiments in larger (respectively smaller) volumes of the same shape and material may be carried out.

NOTE Data from literature show a decrease of the auto-ignition temperature with increasing volume where at the same time the ignition delay time increases. See Annex D.

Where the auto-ignition temperature of the test sample exceeds the softening point of a borosilicate glass flask, or where the sample would cause deterioration of such a flask, i.e. by chemical attack, a quartz or metal flask may be used, provided this is declared in the test report.

7.2.3 Thermocouples

The vessel shall be equipped with at least one calibrated measuring thermocouple of 1,5 mm maximum diameter, having an accuracy of 1,5 K or better. The thermocouple(s) shall be mounted with intimate contact to the external surface of the flask at a distance of (25 ± 2) mm to the bottom of the flask (see Annex A).

7.2.4 Oven

The oven shall be of sufficient dimensions to heat up the test vessel in a uniform manner. It shall be designed in such a way that

- 1) when the oven is covered with a well-fitting lid and after having reached the respective temperature equilibrium
 - the temperature measured at the position of the measuring thermocouple and the temperature measured at the position of the centre of the bottom of the Erlenmeyer flask differ not more than 3 K over the whole temperature range and
 - the temperature measured at the position of the measuring thermocouple and the temperature measured at the position half of the height of the Erlenmeyer flask along the vertical axis differ not more than 15 K over the whole temperature range;
- 2) when the oven is equipped with the Erlenmeyer flask filled with air

- the temperature measured with the measuring thermocouple varies not more than 2 K over a period of 6 min over the whole temperature range.

Care shall be taken that there is no direct contact between the test vessel and the inner walls of the oven. The distance between the inner walls and the Erlenmeyer flask shall be at least 4 mm. The test vessel shall be mounted in such a way that

- it is totally immersed in the oven, whereas the oven should not overlap the Erlenmeyer flask more than 30 mm;
- it is uniformly heated;
- the mixture of air and flammable substance which is generated inside the Erlenmeyer flask by introducing the sample is not affected by the convection inside the oven;
- there is no possibility that the (explosive) mixture of air and flammable substance which is generated inside the Erlenmeyer flask by introducing the sample enters into the oven; and
- it is possible to meter the flammable substances and to observe the ignition.

Examples of ovens suitable for this purpose are described in Annex A.

7.2.5 Metering devices

For liquid samples, the metering device (e.g. pump, pipette, syringe) shall be designed in such a way that it is possible to meter droplets having a volume of $(25 \pm 10) \mu\text{l}$. This requirement can be fulfilled by, for example:

- a) a 0,25 ml or 1 ml hypodermic syringe equipped with a stainless steel needle of 0,15 mm maximum bore diameter, and calibrated in units not greater than 0,01 ml;
- b) a calibrated 1 ml pipette allowing 1 ml of distilled water at room temperature to be discharged in 35 to 40 droplets.

For gaseous samples, the metering device (e.g. flow meter, pump, syringe) shall be designed in such a way that it is possible to meter the gas with an accuracy of 10 % at a rate of $(25 \pm 5) \text{ ml/s}$. A filling tube that can be introduced into the test vessel shall be connected (movably) to the metering device.

Precaution against flash-back is taken for gaseous samples. One method which has been used is illustrated diagrammatically in Figure A.9.

7.2.6 Mirror

It is recommended that a mirror should be suitably positioned approximately 250 mm above the flask to permit convenient observation of the interior of the flask.

7.2.7 Timer

A timer subdivided at least in one-second intervals shall be used to determine the ignition delay time.

7.2.8 Equipment for purging the test vessel with air

The equipment (e.g. pump, air gun) shall allow the quick and complete purging of the test vessel by flushing it with clean, oil-free air.

7.2.9 Automated apparatus

If automated apparatus are used they shall fulfill all the requirements stated in 7.2.2 to 7.2.8. If the monitoring of the ignition is automated as well, it shall be ensured that all kinds of flames (even the very pale ones and the very small ones) are monitored, e.g. by thermocouple and photodiode. Additional visual observation of ignition shall be possible.

7.3 Sampling, preparation and preservation of test samples

7.3.1 Sampling

For sampling of liquid or gaseous products use the respective national/international standards.

NOTE Respective standards are, for example, ISO 3170, ISO 3171, ISO 15528.

Samples shall only be taken or stored in appropriate containers that prevent degradation or contamination of the samples. For liquid mixtures the free vapour space above the sample shall not exceed 20 % of the container volume.

The sample shall be stored at appropriate temperatures to avoid any change in the composition.

If the sample consists of a gas mixture that is removed from a container containing a liquid phase, take into account that the composition of the gas and the liquid phase can be different. Hence, it is recommended to take the test substance from the liquid phase.

7.3.2 Preparation and preservation

The components necessary for the test shall fulfill the following requirements:

- a) Air: The air shall be free of oil.
- b) Synthetic air (mixture of oxygen and nitrogen only): The concentration of the oxygen shall be within a range of 20,5 % to 21,0 % by volume. If synthetic air is used, it shall be stated in the test report.
- c) Inert gas: The purity of the inert gas, or the mixture of inert gases, shall be 99,8 % by volume or greater. If a mixture of inert gases is used, the composition of the mixture shall be stated in the test report.
- d) Sample: The sample may be:
 - 1) a single substance or
 - 2) a mixture of substances or
 - 3) a process sample (of known or unknown composition).

When a single substance or a mixture of substances is used, the purity of each substance shall be 99,8 % by volume or greater. In the case of a mixture of substances or a process sample of known composition the precision of the composition shall be stated in the test report. In the case of a process sample of unknown composition, the source of the sample or the sample shall be defined as well as possible (e.g. process conditions, other physical properties or safety characteristic data).

7.4 Procedure

7.4.1 General

The auto-ignition temperature is determined by varying the temperature of the test vessel and the amount of the sample.

It shall be ensured before each test that:

- the test vessel is clean, dry, without any residue and any visible alteration of the inner surface;
- the test vessel is flushed completely by air before each injection of the sample;
- before injecting the sample, the temperature of the test vessel is the intended one because cleaning and purging may lower the temperature.

If the auto-ignition temperature is to be determined in mixture with air/inert gas, purge the test vessel with the air/inert gas mixture after cleaning (with air) so that the atmosphere inside the Erlenmeyer flask is completely changed, or clean the test vessel with the air/inert gas mixture before each injection.

7.4.2 Sample injection

7.4.2.1 Liquid samples

When testing samples with boiling points at or near room temperature, care shall be taken to maintain the temperature of the sample injection system at a value which will ensure that no change of state occurs before the sample is injected into the test flask.

The required volume of the sample to be tested shall be injected as droplets (see 7.2.5) into the centre of the flask at a rate of 1 to 2 droplets per second. The metering device shall then be quickly withdrawn. Care shall be taken to avoid wetting the walls of the flask during injection.

7.4.2.2 Gaseous samples

The metering device and the connected filling tube are purged sufficiently (at least 10 times the volume) and then filled with the gas. The filling tube is introduced into the centre of the flask so that the outlet of the tube has a distance of (10 ± 2) mm to the bottom. The required volume shall then be injected into the test flask in portions of (10 ± 1) ml at a rate of about 25 ml per second, keeping the rate of injection as constant as possible. The filling tube shall then be quickly withdrawn from the flask.

7.4.3 Determination of the auto-ignition temperature (AIT)

7.4.3.1 Ignition criterion

Any flame detected within 5 min via the mirror or the photo-diode shall be taken as ignition.

Within 5 min a rapid temperature rise of at least 200 K with a rate of 10 K/s shall be met when using thermocouple(s) to detect an ignition.

NOTE With this criterion, normally hot flames are observed. Even if they are very pale ones (e.g. hydrogen, methane), these are hot flames. Some substances or mixtures of substances are able to form cool flames. For such cool flame phenomena see Annex C.

7.4.3.2 Testing procedure

The testing procedure to determine the auto-ignition temperature given in 7.4.3.3 to 7.4.3.8 shall be used.

7.4.3.3 Starting temperature

Starting from 80 °C the test vessel is heated up with a temperature rate of (5 ± 1) K/min. Whilst heating up inject every 20 K, (50 ± 5) ml in case of a gas or five droplets in case of a liquid till an ignition occurs. The test vessel shall be flushed completely by air before each injection. The temperature of the vessel at which this ignition occurs is the starting temperature.

The starting temperature shall be higher than the auto-ignition temperature.

7.4.3.4 Variation of temperature

Heat the test vessel to the starting temperature. Inject (50 ± 5) ml in the case of a gas or five droplets in the case of a liquid. If an ignition occurs within 5 min, lower the temperature of the test vessel in intervals of (5 ± 1) K till no ignition occurs within 5 min after introducing the

same amount of sample. The test vessel shall be flushed completely by air before each injection.

7.4.3.5 Variation of amount

At that temperature of 7.4.3.4 where just no ignition occurred vary the introduced amount of the sample (from the (50 ± 5) ml in the case of a gas or five droplets in the case of a liquid) stepwise in increments of (10 ± 1) ml in the case of gases or (25 ± 10) µl in the case of liquids in both directions till an ignition occurs or until it is ensured that no ignition will occur at this temperature with any amount of sample. The test vessel shall be flushed completely by air before each injection.

7.4.3.6 Second variation of temperature

Choose the temperature found in 7.4.3.4 and the amount found in 7.4.3.5 where an ignition occurred. Lower the temperature of the test vessel in intervals of (2 ± 1) K till no ignition occurs within 5 min after introducing every time the same amount of substance as found in 7.4.3.5.

7.4.3.7 Second variation of amount

At the temperature of 7.4.3.6 vary the introduced amount of the sample (starting with the amount of 7.4.3.5 respectively. 7.4.3.6 for repeated tests) stepwise in increments of (10 ± 1) ml in the case of gases or (25 ± 10) µl in the case of liquids in both directions till an ignition occurs or until it is ensured that no ignition will occur at this temperature with any amount of sample. The test vessel shall be flushed completely by air before each injection.

7.4.3.8 Repetition of tests

Repeat 7.4.3.6 and 7.4.3.7 until a temperature is found where no ignition occurs whatever the amount of substance will be.

NOTE For flammable liquids, having the (initial) boiling point clearly above the ignition temperature as determined, an additional injection of air, after having metered the flammable liquid, may lower the ignition temperature remarkably (F. Gutte: Erdöl, Erdgas, Kohle: 111 1995, 203-207). For taking this into account, introduce (30 ± 10) ml of air with high velocity to mix up the vapour layer of the flammable liquid that is formed in such cases at the bottom of the test vessel.

Record the lowest temperature of the test vessel at which an ignition occurred (whatever the introduced amount of sample was) and the corresponding ignition delay time.

The last repetition round shall be repeated twice.

7.5 Auto-ignition temperature (AIT)

The lowest temperature at which auto-ignition occurs in the tests described in 7.4.3.8 reduced by 3 % and rounded to the next 1 °C shall be recorded as the auto-ignition temperature, provided that the results satisfy the validity requirements of 7.6. The corresponding auto-ignition delay time and the barometric pressure shall be recorded.

7.6 Validity of results

7.6.1 Repeatability

Results of repeated tests on pure substances obtained by the same operator and fixture shall be considered suspect if they differ by more than ± 1 % of the mean value.

7.6.2 Reproducibility

Results of tests on pure substances obtained in different laboratories shall be considered suspect if they differ by more than ± 3 % of the mean value.

NOTE For substances able to change the surface of flask during testing the values for repeatability and reproducibility may be greater than those given above.

7.7 Data

A record shall be kept of the sample identification (name, purity, source and/or characterization in the case of mixtures or process samples), oxidizer identification (atmospheric or synthetic air; composition, purity and amount of added inert), test conditions (ambient temperature and ambient pressure), automated test set-up used or not, type of oven used, results (auto-ignition temperature and auto-ignition delay time; amount of sample at which the AIT is found).

7.8 Verification of the auto-ignition temperature determination method

This verification procedure shall be used for new apparatus as well as for checking the performance of existing apparatus. Existing apparatus has to be checked at least every 12 months or whenever parts of the apparatus have been changed or renewed. For new apparatus, carry out experiments according to the instructions given in 7.3 with all the substances listed in Table 3, starting the tests at the given starting temperature. When renewing the test vessel it is in general sufficient to carry out the check test with only one of the substances chosen according to the temperature range expected. The purity of the substances ethylene and acetone shall be 99,8 mol % or greater, and that of *n*-Heptane shall be 99,3 mol % or greater.

The values given in Table 3 are the respective mean values of the lowest temperatures reached by interlaboratory tests.

Verification will be confirmed if the values obtained for the lowest temperature for ignition do not deviate more than ± 3 % from the values given in Table 3. The values are valid for an ambient temperature of (20 ± 2) °C and an ambient pressure of (100 ± 2) kPa.

Table 3 – Values for verification of the apparatus

Flammable substance	Starting temperature °C	Measured lowest temperature for ignition °C
Acetone	560	528
Ethylene	455	436
<i>n</i> -Heptane	240	221

If the results obtained by the test apparatus meet the required verification performance, record this fact in a permanent report.

If the results obtained by the test apparatus do not meet the required verification performance, check the test vessel and the hot-air oven. If appropriate, change the test vessel and verify again.

Annex A (normative)

Ovens of test apparatus for the tests of auto-ignition temperature

A.1 General

Ovens constructed in accordance with A.2 and A.3 are suitable for the tests described in Clause 7.

A.2 “IEC oven”

The “IEC oven” is shown schematically in Figure A.1 to Figure A.5.

NOTE Historically this consists of a refractory cylinder, 127 mm in internal diameter and 127 mm long, circumferentially wound with a 1 200 W electric heater uniformly spaced along its length; a suitable refractory insulating material and retaining shell; a cover ring and flask guide ring made from a board of refractory material; a 300 W neck heater and a 300 W base heater.

Three thermocouples are used, positioned 25 mm and 50 mm below the bottom of the neck heater, and under the base of the flask near its centre.

A.3 “DIN oven”

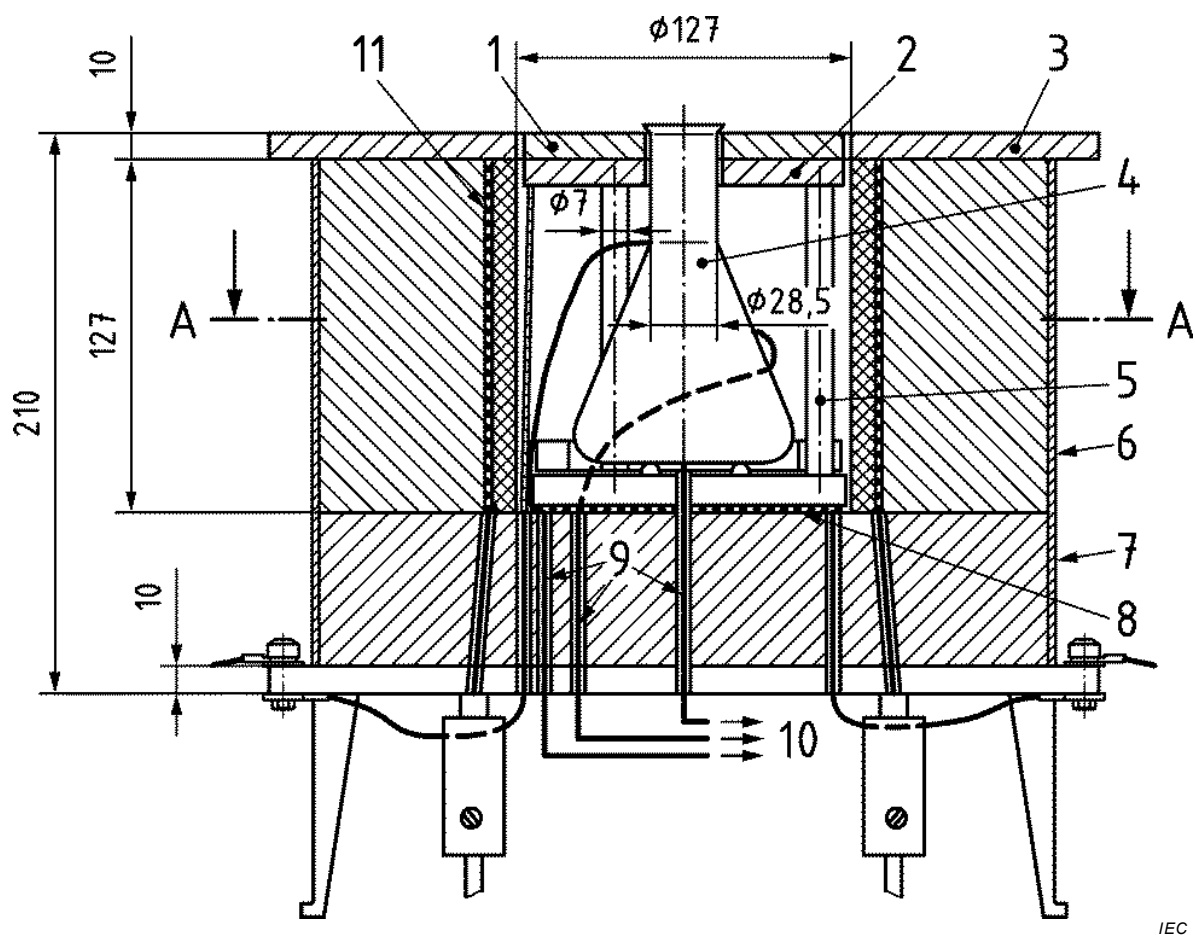
The “DIN oven” is shown schematically in Figure A.6 to Figure A.8. It consists of a resistance-heated oven of approximately 1 300 W, maximum heating current 6 A.

NOTE The heating wire, diameter 1,2 mm, length 35,8 m, of (Cr/Al 30/5) alloy is circumferentially wound round the full length of a ceramic cylinder, with a turn spacing of 1,2 mm. The heater is fixed in position with high temperature mastic and enclosed by a thermally insulating layer of aluminium oxide powder 20 mm thick. A stainless steel cylinder is inserted in the ceramic body with the smallest possible clearance. The lid, covering the whole oven, is also of stainless steel and holds the flask within the oven. For this purpose, the lid consists of a top disk, a split insulating gasket and a split lower disk. The neck of the flask is fitted into the lid with heat insulating packing and is held by the segments of the split gasket and the lower disk, which are squeezed against it and fixed to the top disk by means of two ring nuts.

The heater may be operated on a.c. or d.c. with appropriate means of voltage control. The maximum heating current of about 6 A should be used to attain the temperature required for the preliminary tests. If an automatic temperature control system is used, the heating and cooling periods should be of equal length and if possible only a part of the heater current should be so controlled.

Measurement thermocouples are positioned on the outer-surface of the wall of the flask, 25 mm ± 2 mm from its base, and at the centre of the under-surface of the base.

Dimensions in millimetres



Key

- | | |
|--------------------------------|-----------------------------|
| 1 cover ring | 7 electric crucible furnace |
| 2 neck heater | 8 base heater |
| 3 board of refractory material | 9 thermocouples |
| 4 200 ml Erlenmeyer flask | 10 to potentiometers |
| 5 ceramic support | 11 main heater |
| 6 retaining cylinder | |

Figure A.1 – Test apparatus: assembly