

BSI Standards Publication

Determination of the explosion limits and the limiting oxygen concentration(LOC) for flammable gases and vapours



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National foreword

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Determination of the explosion limits and the limiting oxygen concentration(LOC) for flammable gases and vapours

Détermination des limites d'explosivité des gaz et vapeurs et détermination de la concentration limite en oxygène (CLO) des gaz et des vapeurs inflammables

Bestimmung der Explosionsgrenzen und der Sauerstoffgrenzkonzentration (SGK) für brennbare Gase und Dämpfen

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European foreword

This document (EN 1839:2017) has been prepared by Technical Committee CEN/TC 305 "Potentially explosive atmospheres - Explosion prevention and protection", the secretariat of which is held by DIN.

This document supersedes EN 14756:2006, and EN 1839:2012.

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of an identical text or by endorsement, at the latest by July 2017, and conflicting national standards shall be withdrawn at the latest by January 2018.

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 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 relationship with EU Directives, see informative Annex ZA, which is an integral part of this document.

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Introduction

The hazard of an explosion can be avoided by preventing the formation of explosive mixtures of gases and/or vapours with air. To do so, the explosion limits (also known as "flammability limits") or the limiting oxygen concentration of the flammable substance need to be known. These limits depend mainly on:

- the properties of the flammable substance;
- temperature and pressure;
- size and shape of the test vessel;
- ignition source (type, energy);
- the criterion for self-propagating combustion;
- the inert gas (in case of the limiting oxygen concentration).

To obtain reliable and comparable results it is necessary to standardize the conditions for determining the explosion limits resp. the limiting oxygen concentration (i.e. apparatus and procedure). However, it is not possible to provide one single method that is suitable for all types of substances. For practical reasons, it is preferable to use apparatus that can also be used for the determination of other explosion characteristics. This European Standard, therefore, details two methods, namely, the tube method (method T) and the bomb method (method B). In general, the tube method gives a wider explosion range. Differences in the explosion limits and limiting oxygen concentration determined by the two methods can vary by up to 10 % relative.

For substances which are difficult to ignite, only a modified tube method is suitable. This is described in Annex A.

1 Scope

This European Standard specifies two test methods (method T and method B) to determine the explosion limits of gases, vapours and their mixtures, mixed with air or an air / inert gas mixture (volume fraction of the oxygen < 21 %) and the limiting oxygen concentration. This European Standard applies to gases, vapours and their mixtures at atmospheric pressure for temperatures up to $200 \,^{\circ}$ C.

2 Normative references

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

EN 13237:2012, Potentially explosive atmospheres - Terms and definitions for equipment and protective systems intended for use in potentially explosive atmospheres

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

flammable substance

substance in the form of gas, vapour or mixtures of these, able to undergo an exothermic reaction with air or air / inert gas mixture when ignited

[SOURCE: EN 13237:2012, 3.37, modified]

3.2

explosion range

range of the concentration of a flammable substance or mixture of substances in air, within which an explosion can occur, respectively range of the concentration of a flammable substance or mixture of substances in mixture with air / inert gas, within which an explosion can occur, determined under specified test conditions

[SOURCE: EN 13237:2012, 3.22, modified]

Note 1 to entry: The explosion limits are not part of the explosion range

3.3

lower explosion limit

LEL

lowest concentration of the explosion range

[SOURCE: EN 13237:2012, 3.19.1, modified]

Note 1 to entry: Those concentrations are given at which an explosion just fails during the tests.

3.4

upper explosion limit

IIEL.

highest concentration of the explosion range

[SOURCE: EN 13237:2012, 3.19.2, modified]

Note 1 to entry: Those concentrations are given at which an explosion just fails during the tests.

3.5

limiting air concentration

LAC

maximum air concentration in a mixture of a flammable substance, air and an inert gas, in which an explosion will not occur

Note 1 to entry: *LAC* is usually expressed as molar fraction in % or volume fraction in % (for conversion of units

see Annex B).

Note 2 to entry: The *LAC* does not depend only on the flammable gas or vapour, but also on the inert gas used.

3.6

limiting oxygen concentration

LOC

maximum oxygen concentration in a mixture of a flammable substance, air and an inert gas, in which an explosion will not occur

[SOURCE: EN 13237:2012, 3.49, modified]

Note 1 to entry: LOC is usually expressed as molar fraction in % or volume fraction in % (for conversion of units

see Annex B)

Note 2 to entry: The *LOC* does not depend only on the flammable gas or vapour, but also on the inert gas used.

Note 3 to entry: LOC is calculated from the measured LAC

3.7

inert gas

gas that does not react with the test substance or oxygen

3.8

explosion region

area inside the boundary curve formed by the explosion limits of a flammable substance in various mixtures with air and inert gas

[SOURCE: EN 13237:2012, 3.15, modified]

Note 1 to entry: In many cases the apex of the boundary curve corresponds to the limiting air concentration, LAC.

3.9

explosion criterion — flame detachment

in method T there are two alternate criteria for explosion (self-propagating combustion): i) the detachment and upward movement of the flame from the spark gap for at least 100 mm, or ii) the formation of a halo which either reaches the top of the tube, or reaches a minimum height of 240 mm

Note 1 to entry: Throughout the duration of the ignition spark, test mixtures, whose test substance content lies just outside the explosion range, may exhibit a luminous phenomenon (referred to as a "halo") above the spark gap which does not detach from the latter (see Annex C). For some test substances (e.g. halogenated hydrocarbons), this luminous phenomenon can occupy a large portion of the test vessel. The formation of a halo alone is not considered to count as an ignition of the test mixture unless it reaches the top of the tube or a minimum height of 240 mm.

3.10

explosion criterion — pressure rise

in method B, the criterion for an explosion (self-propagating combustion) is the generation of explosion overpressure which is equal to or greater than the overpressure created by the ignition source alone in air plus (5 ± 0.1) % of the initial pressure

3.11

vapour

gaseous phase emanating or being emanated from a liquid

Note 1 to entry: If not otherwise mentioned, the term "gas" in this standard also includes such vapours but not mists.

3.12

oxidizer

air or an air / inert gas mixture (volume fraction of the oxygen < 21 %)

3.13

sample

substance or mixture of substances for which explosion limits are to be determined

3.14

test substance

sample in the gaseous state; in the case of liquid samples, after complete evaporation

3.15

test mixture

mixture of test substance and air or air / inert gas

4 Test methods

4.1 General

The determination consists of a series of ignition tests which are carried out with test mixtures whose test substance content is varied when determining the explosion limits and with test mixtures whose test substance content and inert gas content is varied when determining the limiting oxygen concentration..

For organic substances which consist exclusively of carbon, hydrogen and oxygen (with the exception of peroxides), the LEL can be roughly estimated. At 20 °C, the LEL, in many cases, is approximately half the test substance content of the stoichiometric composition in air. The temperature dependence of the LEL

has to be taken into account. Up to 200 °C, the $\it LEL$ decreases more or less linearly between 30 % and 50 % of the value estimated for 20 °C.

There is currently no method which readily estimates the *UEL*.

When it is established that a given test mixture will not ignite, it is recommended that the quantitative composition of the non-ignited test mixture flowing out of the test vessel is analysed in order to determine whether any errors have arisen either with the metering devices or due to leakage.

4.2 Method T ("tube" method)

4.2.1 Detailed method

The test mixture flows through the cylindrical test vessel from the bottom upwards to the top until the contents previously in the test vessel have been completely replaced. Then, under quiescent conditions, an ignition is initiated using a series of induction sparks. It is observed whether or not flame detachment occurs. When determining the *LEL* or the *UEL* (explosion criterion — flame detachment) the test substance content of the test mixture is varied stepwise until an ignition of the mixture just fails or until it is established that there is no explosion range. When determining the LOC the test substance content of the test mixture as well as the inert gas content is varied stepwise until an ignition of the mixture just fails.

4.2.2 Reagents and materials

4.2.2.1 Air, which shall be free of water (\leq 0,1 mol% water vapour absolute) and oil (\leq 0,1 g / m³ oil)

If synthetic air is used, it has to be stated in the report.

4.2.2.2 Inert gases, the purity of the inert, or the mixture of inerts, shall be 99,8 % mol. or better

If a mixture of inerts is used, the composition of the mixture shall be stated in the test report.

4.2.2.3 Flammable substances, which may be either a single substance or a defined mixture of substances or a process sample (of known or unknown composition)

When a single substance or a defined mixture of substances is used, the purity of each substance shall be 99,8 % mol. or better. 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. For a process sample with unknown composition, the sample shall be defined as precisely as possible (e.g. process conditions).

If the flammable gas is derived from a liquid containing more than one component, the gas phase composition can differ from the composition of the liquid phase. When large volumes of the gas are removed, the composition of both the liquid and gas phases can change with time. For these reasons, the test sample shall be taken from the liquid phase.

4.2.2.4 Heat-resistant, chemically inert material for gaskets and adhesive mountings

Sample containers shall be kept closed before and after sampling to avoid changes in the sample composition within the container (e.g. loss of volatile components from mixtures). If a sample container contains a mixture with both gaseous and liquid phases present, the mixture composition of the two phases will be different. Under such conditions, it is recommended that the test substance sample be removed from the liquid phase. If the sample is taken from the gaseous phase, account shall be taken of the difference in composition.

4.2.3 Apparatus

4.2.3.1 Test vessel

The test vessel is an upright cylindrical vessel made of glass or other transparent material (e.g. polycarbonate) with an inner diameter of (80 ± 2) mm and a minimum length of 300 mm.

The vessel is equipped with an inlet pipe with a three-way valve for the test mixture, located at the bottom, and an outlet pipe and pressure vent in the upper part.

The bottom and top may be made of other material. However, the material shall be free of any catalytic effect and resistant to corrosion from the test mixture or the reaction products.

4.2.3.2 Ignition source

A series of induction sparks between two electrodes is used as the ignition source.

The electrodes shall end (60 ± 1) mm above the bottom of the test vessel.

Stainless steel is a suitable material for the electrodes. The electrodes shall be pointed rods with a diameter of maximum 4 mm. The angle of the tips shall be $(60 \pm 3)^{\circ}$. The distance between the tips shall be (5 ± 0.1) mm. The electrodes shall be mounted in the vessel so that they are gas tight at the highest explosion pressures generated during the test. The mounting shall be resistant to both heat and the test mixture, and also provide adequate electrical resistance from the test vessel body.

A high voltage transformer, with a root mean square of 13 kV to 16 kV and a short circuit current of 20 mA to 30 mA, shall be used for producing the ignition spark. The primary winding of the high voltage transformer shall be connected to the mains via a timer set to the required discharge time.

The spark discharge time shall be adjusted to 0,2 s.

The power of the induction sparks is dependent on the gas mixture and its pressure. In air at atmospheric conditions, according to calorimetric and electric measurements, such a source gives a spark with a power of approximately 10 W.

4.2.3.3 Equipment for preparing the test mixture

The test mixture is prepared by mixing flows of gaseous components. This requires the following equipment:

- metering device for air, gaseous samples, inert gases (e.g. mass flow controller, volume flow controller, metering pump for gases);
- metering device for liquid samples (e.g. volumetric metering pumps);
- evaporator equipment in the case of a liquid sample (for example see Annex D);
- mixing vessel for homogenizing the test mixture.

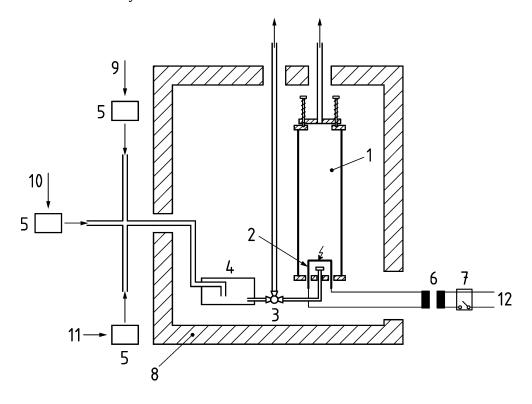
The metering devices and the equipment for preparing the test mixture have to be designed in such a way that the uncertainty of measurement of the test substance content in the test mixture is not higher than the data given in Table 1.

Table 1 — Maximum permissible uncertainty of measurement for the amount of test substance in the test mixture

molar amount of test substance %	maximum allowable uncertainty of measurement %	
	relative	absolute
≤ 2	±10	
> 2		±0,2

4.2.3.4 Temperature regulating system

For measurements at temperatures above ambient temperature, the apparatus requires a temperature regulating system. When this is used, it has to be ensured that the temperature difference inside the test vessel is not more than 10 K. This has to be checked when initially setting up the apparatus, whenever parts are renewed and at every verification.



Key

1 test vessel 7 timer

2 electrodes 8 facility for keeping the temperature

3 three-way valve 9 flammable substance

4 mixing vessel 10 air

5 metering devices 11 inert gas

6 high-voltage transformer 12 power supply

Figure 1 — Scheme of the 'tube' apparatus for determining the explosion limits respc. Limiting oxygen concentration

4.2.3.5 Safety equipment

The safety measures specified in Annex E shall be followed.

4.2.4 Preparation of the test mixture

When evaporating liquid samples, it is important to remember that the mixture composition of the gaseous phase in equilibrium with a liquid phase ("vapour") generally differs from the mixture composition of the liquid phase itself. Furthermore, the mixture compositions of the liquid and the vapour phases may change when removing material from the vapour phase. It is necessary to account for these possible changes in composition when determining explosion limits for flammable liquids, when handling liquid samples and when preparing test mixtures by evaporating liquid mixtures. To avoid error, the method of dynamic total evaporation is used. An example of a suitable evaporator set up is described in Annex D. When liquids are metered, it has to be ensured that bubbles are not formed in any component carrying the liquid (e.g. pipes). To achieve complete homogenization, the test mixture flows through a mixing vessel, preferably made of glass. For a mixing vessel with no built-in elements, a volume of at least 600 ml is recommended. It is expedient to introduce the test mixture tangentially. The mixing vessel is not necessary if homogenization is effectively achieved by the metering device. The temperature of the mixing vessel and of all parts carrying the test mixture is kept constant to prevent the test substance from condensing. It is recommended that the components carrying the test mixture are heated along with the test vessel.

4.2.5 Procedure

4.2.5.1 General

The characterization of explosion limits consists of determining the amount of test substance in the mixture with which the test mixture no longer ignites (according the explosion criterion in 3.10). Close to the explosion limits, the incremental change of test substance content is selected such that it is almost equal to the relative uncertainty of measurement given in Table 1.

If the explosion limits or the limiting oxygen concentration are to be determined at elevated temperature, preheat the test vessel and all parts carrying the test mixture to the required temperature. For liquid samples, the temperature of the test mixture shall be at least 25 K higher than the condensation temperature. Prior to each ignition attempt, it has to be ensured that the temperature in the test vessel differs by no more than 5 K from the required value.

4.2.5.2 Determination of explosion limits

For safety reasons, the initial ignition tests are carried out using a test mixture with test substance content which, if possible, lies outside the expected explosion range

Prior to each ignition attempt, the test vessel is purged with the test mixture. The purging volume has to be at least ten times the volume of the test vessel. When purging is complete, the inlet to the test vessel is sealed. The test mixture then by-passes the test vessel and flows directly into the exhaust system. An ignition is attempted using the induction spark under quiescent conditions (i.e. after a 6 s to 10 s delay). It is observed whether a flame detaches from the ignition source (see Annex C).

It is recommended that the ignition testing is carried out without interruption of the production of the test mixture. If restarting, it will take a finite time to produce a test mixture of constant composition even if the adjustment has not been changed.

If an ignition is observed, the test substance content in the test mixture is iteratively varied until no further flame detachment follows. Close to the explosion limits, the incremental change of test substance content is selected so that it is almost equal to the relative deviation given in Table 1. The test mixture concentration at which an ignition just fails (just no flame detachment) has to be confirmed with four additional tests. The determination is terminated when with all five tests a flame detachment is not observed. If flame detachment does occur, the test substance content has to be further changed,

i.e. for determination of the LEL, the test substance content has to be reduced by one increment; for the UEL, it has to be increased by one increment. Five tests are carried out at the new test substance content.

4.2.5.3 Determination of the limiting oxygen concentration

See 4.4

4.3 Method B ("bomb" method)

4.3.1 Principle

The quiescent test mixture in a closed vessel (the bomb) is subjected to an ignition source. The overpressure given by the ignition is measured and characterizes the explosivity of the test mixture. The amount of test substance in the test mixture is varied incrementally until the *LEL* or the *UEL* is determined, or until it is established that there is no explosion range.

4.3.2 Reagents and materials

See 4.2.2

4.3.3 Apparatus

4.3.3.1 Test vessel

The test vessel shall be cylindrical or spherical. The internal volume of the test vessel shall be equal to or greater than 0.005 m^3 . If a cylindrical vessel is used, the length to diameter ratio shall be between 1 and 1.5.

The test vessel and any equipment (valves, ignition source, transducer etc.) fitted to the vessel shall be designed to withstand a maximum overpressure of at least 15 bar.

The vessel shall be made of stainless steel or any material free of any catalytic effect and resistant to corrosion from the initial gas mixture and the products of combustion.

The test vessel shall be fitted with sufficient ports to allow filling, evacuating and purging.

If the test mixture is prepared inside the test vessel by partial pressures, it is recommended to disconnect the pressure measuring system used to prepare the test mixture, via a valve, to protect it during the ignition trials.

The components of the temperature measuring system located inside the test vessel (e.g. thermocouple) have to be mounted so that propagation of the flame is not hindered.

4.3.3.2 Ignition source

4.3.3.2.1 General

The ignition source shall be positioned in the centre of the test vessel. Suitable types of ignition source are either a series of induction sparks or a fuse wire. In the test report, the type of ignition source used shall be stated.

4.3.3.2.2 Induction spark

See 4.2.3.2

4.3.3.2.3 Fuse wire

An electric arc is generated by passing an electric charge along a straight length of fuse wire connected between two metal rods.

The electrical power required to melt the wire and generate the arc is supplied by an isolating transformer. The ignition energy delivered by the arc depends on its duration and on the power rating of the isolating transformer. The energy delivered shall be in the range of 10 J to 20 J as within this range of energies there is no significant variation in the explosion limits. This is achieved by limiting the power rating of the isolating transformer to between 0,7 kW and 3,5 kW and by the use of a phase control technique. This is a chopping technique that allows only part of the AC waveform from the transformer secondary windings to energize the wire.

Brass and stainless steel are suitable materials for the rods. The rods shall be parallel to each other with a separation distance of (5 ± 1) mm. For the fusing wire, a straight length of a NiCr wire (diameter 0,05 mm to 0,2 mm) shall be soldered to the tips of the of the metal rods. The rods shall be positioned in the test vessel so that the fuse wire is at the centre of the vessel. The electrodes shall be mounted in the vessel such that they are gas tight at the highest explosion pressures generated during the test. The mounting shall be resistant to heat, resistant to corrosion from the test mixture and combustion products and shall provide adequate electrical resistance from the test vessel body.

To reduce the time required for replacing the fusing wire after each test, the rods can be mounted in a plug that can be screwed into the test vessel wall.

The cross-section of the wires connecting the transformer to the rods shall be between 2,5 mm² and 7 mm². The length of the wires shall be less than 5 m. The diameter of the rods shall be between 1,5 mm and 5 mm.

If, for practical reasons, the diameter of the rods has to be less than 3 mm, additional mechanical support may be necessary.

4.3.3.3 Explosion overpressure measurement system

The pressure measurement system consists of:

- a pressure transducer;
- an amplifier;
- recording equipment.

The pressure transducers shall have a resonance frequency greater than 10 kHz.

The pressure measurement system shall have an accuracy that allows the explosion over pressure to be measured in accordance with the explosion criterion defined in 3.10. It shall have a time resolution of at least 1 ms.

The pressure transducer shall be fitted inside the test vessel, with the head flush with the internal wall.

4.3.3.4 Equipment for preparing the test mixture

The test mixture can be prepared either by partial pressures, or mixing gas flows of the substances. This can be done inside or outside the test vessel.

If the test mixture is prepared by partial pressures, the vessel used for the preparation of the mixture shall be fitted with:

- a vacuum pump and a vacuum gauge;
- pressure gauges or manometers;
- a means of homogenizing the test mixture (e.g. a stirrer).

The equipment used for measuring and preparing the test mixture has to be designed in such a way that the uncertainty of measurement of the test substance content in the test mixture is not higher than the data given in Table 1.

If the test mixture is prepared by mixing gas flows then, 4.2.3.3 and Table 1 apply.

4.3.3.5 Temperature regulating system

See 4.2.3.4

4.3.3.6 Safety equipment

The safety measures specified in Annex E shall be followed.

4.3.4 Preparation of the test mixture

4.3.4.1 General

If liquefied gases or liquids are used, it is necessary to ensure that there is no condensation.

Special care has to be taken when preparing test mixtures from samples of liquid mixtures (4.2.2.3 and 4.2.4).

NOTE Condensation can be prevented by checking the vapour pressure of the substances and by local heating to prevent cooling at certain parts of the apparatus (e.g. valves).

The test mixture can be prepared by partial pressures or mixing together flows of the component substances. This can be done inside or outside the test vessel.

It is also recommended to:

- determine the composition of the test mixture;
- check the metering devices;
- ensure that there are no metering errors or leaks.

4.3.4.2 Preparation of the test mixture by partial pressures

If the preparation of the test mixture involves evacuating the vessel, the amount of air remaining in the vessel has to be taken into account when calculating the necessary amounts of the flammable materials and air.

The mixture components are sequentially introduced into the vessel to give the required partial pressure. The partial pressure measuring system shall have a sensitivity of \pm 0,0005 bar, or better, and an uncertainty in measurement of 0,5 % of full scale.. The pressure sensors shall be checked in the range of 0 mbar to 20 mbar by a vacuum meter before starting the mixture preparation. It is necessary to ensure that the mixture in the vessel is thoroughly mixed during the introduction of each component. If the volume of the feed lines is not negligible compared to the volume of the vessel, they also need to be evacuated or purged.

NOTE For practical reasons, air is often introduced as the last component, especially if atmospheric air is used.

4.3.4.3 Preparation of the test mixture by mixing flows

The test mixture is prepared by thoroughly mixing metered flows of the gaseous substances.

When liquids are being tested, they shall be totally vaporized before mixing.

It is also recommended to:

- determine the composition of the test mixture;
- check the metering devices;
- ensure that there are no metering errors or leaks.

4.3.5 Procedure

The characterization of explosion limits consists of determining the amount of test substance in the test mixture with which the test mixture no longer ignites (according the explosion criterion in 3.10). Close to the explosion limits, the incremental change of test substance content is selected such that it is equal to the relative uncertainty of measurement given in Table 1.

If the explosion limits or the limiting oxygen concentration are determined at elevated temperature, preheat the test vessel and all components carrying the test mixture to the required temperature. For liquid samples, the temperature of the test mixture shall be at least 25 K higher than the condensation temperature. Prior to each ignition attempt, it has to be ensured that the temperature in the test vessel differs by not more than 5 K from the required value.

4.3.6 Determination of explosion limits

If the test mixture is prepared by the partial pressure method inside the test vessel, the procedure is as follows:

- a) preheat the test vessel and associated components to the required temperature;
- b) purge the vessel with inert gas (or an inert pre-mixture);
- c) evacuate the vessel and measure the residual pressure;
- d) charge the test vessel with each substance to the respective partial pressure (it is necessary to take into account the residual pressure measured beforehand);
- e) homogenize (e.g. stir) the mixture for a suitable period (3 min to 5 min);
- f) switch off the homogenizer, wait 1 min to 2 min till the mixture is quiescent;
- g) close the valve which protects the partial pressure transducer;
- h) turn on the explosion overpressure recording system;
- i) activate the ignition source and record the pressure-time-curve;
- i) return the vessel to atmospheric pressure;
- k) repeat steps a) to j) as necessary, changing the mixture composition iteratively

The test mixture concentration at which an ignition just fails has to be confirmed with four additional tests.

It is necessary to ensure that during operations d) and e) chemical decomposition reactions or slow oxidation of the test mixture do not occur. This is particularly relevant for tests carried out at elevated temperatures. Such reactions can usually be detected by an increase in pressure and / or temperature, and can lead to false test results.

If the test mixture is prepared by the method of mixing flows, or if it is prepared by partial pressure method in a vessel separate from the test vessel, the procedure is summarized as follows:

- 1) preheat the test vessel and associated components to the required temperature;
- 2) if the test mixture is prepared by mixing flows, purge the test vessel with the test mixture (the volume for purging has to be at least ten times the vessel volume);

or

if the test mixture is prepared by the partial pressure method using a separate vessel, evacuate the test vessel to a pressure < 5 mbar and fill with the test mixture;

- 3) close the test vessel isolation valves:
- 4) turn on the explosion overpressure recording system;
- 5) activate the ignition source and record the pressure-time-curve;
- 6) return the vessel to atmospheric pressure;
- 7) repeat step 1) to 6) as necessary, changing the mixture composition iteratively

The test mixture concentration at which an ignition just fails has to be confirmed with four additional tests.

For tests on mixtures with high test substance contents, combustion may produce a considerable amount of soot. In such cases, the interior of the vessel shall be inspected and cleaned to remove build-up of soot prior to subsequent tests.

4.3.7 Determination of the limiting oxygen concentration

See 4.4

4.4 Determination of the limiting oxygen concentration

4.4.1 Metering devices and additional equipment

The metering devices and the equipment for preparing the test mixture shall be designed in such a way that the uncertainty of measurement of the test substance content in the test mixture is not higher than given by the data in Table 1 and the uncertainty of measurement of the inert gas content in the test mixture is not higher than 0,1 % absolute.

A calibrated oxygen analyser with a precision of a molar fraction of $0.1\,\%$ oxygen (e.g. paramagnetic analyser, gas chromatography etc.) is necessary to check the result.

4.4.2 Procedure

4.4.2.1 General

Ignition trials with defined test mixtures are carried out using method (T) or method (B) in order to obtain the explosion region or a part of the explosion region for a ternary system of test substance, air and inert gas. By increasing incrementally the fraction of inert gas and varying the fraction of the test substance in the test mixtures the limiting air concentration, *LAC*, is obtained.

From the measured *LAC* the *LOC* is calculated according to the following equation:

$$LOC = 0,209 \times LAC \tag{1}$$

Two different methods are described for the determination of the *LAC*, an extended procedure and a short procedure.

In most cases the *LOC* corresponds to the apex of the explosion region. For these cases the short procedure may be used. However, in some cases the *LOC* will correspond to the upper explosion limit curve, and for these cases the extended procedure shall be used.

With substances that are difficult to ignite, or if the stoichiometric concentration in air is unknown, or the heat capacity of the inert component deviates considerably from that of air, then the extended procedure shall be used.

Otherwise the criterion for the selecting the "short procedure" over the "extended procedure" is given by Formula (2).

$$UEL \le 0.8 \times \left(100 - x_{air,L}\right) \tag{2}$$

where

 $\boldsymbol{x}_{\text{air,L}}$ is the air fraction at the explosion limit, as molar fraction in %, of the stoichiometric flammable substance / air mixture.

The following steps have to be carried out to decide on the procedure:

- 1) If the *UEL* (and *LEL*) are unknown, then they shall be determined according to 4.1 or 4.2.
- $x_{air,L}$

The explosion limit of the stoichiometric flammable substance / air mixture has to be determined. It is obtained by varying iteratively the inert gas fractions added. To start with, the added inert gas fraction shall be a molar fraction of 50 %. The first increment shall be a molar fraction of 5 %. The smallest increment shall be at least 0,5 %. $x_{air,L}$ is the air fraction of that flammable gas / air / inert gas mixture, with stoichiometric composition of flammable substance / air, in which an explosion just fails. Using Figure 2 as an example this corresponds to determinations along the line marked 2.

If the stoichiometric concentration in air is unknown, and the inert component is nitrogen, then the test substance concentration shall be fixed at $x_{TS} = 1.2$ *LEL* when determining $x_{air,L}$. Using Figure 2 as an example this corresponds to determinations along the line marked 3.

NOTE Violent reactions can occur in some mixtures with a molar fraction of 50 % inert gas.

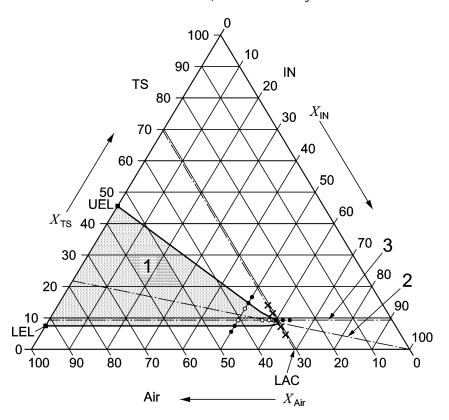
4.4.2.2 Short procedure

If Formula (2) is satisfied the short procedure can be applied (after carrying out steps 1 and 2) (see Figure 2). In this case only the area around the apex needs to be investigated.

The amount of inert gas added and the ratio of flammable substance to air are varied stepwise until LAC is determined starting from the mixture flammable substance / air / inert gas with which $x_{air,L}$ was found.

The following steps have to be carried out:

- 3a Based on the mixture of flammable substance / air / inert gas with which $x_{\text{air,L}}$ was found (in step 2) the ratio of flammable substance to air is increased stepwise until an ignition just fails. The lowest step size for the variation of the ratio flammable substance to air the amount shall be 0,001.
- 4a Starting with the mixture found in step 3a the amount of inert gas is varied while keeping constant the flammable substance / air ratio. The lowest increment for the variation of the inert gas added shall be $0.5\,\%$
- 5a Repeat step 3a and 4a until *LAC* is determined, and then verify with four additional tests.



Key

- 1 explosion region
- 2 line of stoichiometric concentration fuel / air
- 3 line $X_{TS} = 1.2 LEL$

LEL lower explosion limit in air

UEL upper explosion limit in air

LAC limiting air concentration

- *X* molar fraction in %
- IN inert gas
- TS test substance

Air air

- no explosion
- explosion
- additional tests at x_{air},L

Figure 2 — Short procedure scheme for the determination of the LAC

To confirm that the LAC does lie at the apex of the explosion region, and not along the upper boundary from the UEL, the explosion limits shall be determined with a fixed fraction of inert gas which is less than that at the LAC determined in steps 3a to 5a. This fixed amount of inert gas shall be 80 % of the inert fraction of the mixture composition at the osculation point corresponding to the LAC. Using Figure 2 as an example this corresponds to determinations at the points marked by dots along the 50 % inert gas line.

NOTE the osculation point is the point where the tangent line parallel to the inert gas axis touches the line bordering the explosion region.

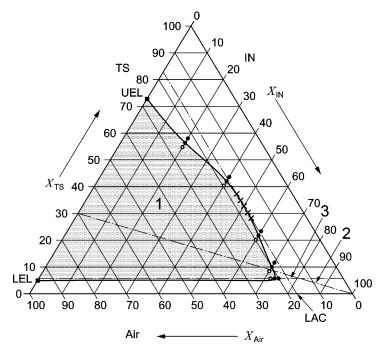
5b The *LAC* is the air concentration at the determined point. This can be read from the axis by drawing the tangent to the apex of the explosion region parallel to the inert axis

The *LOC* is then calculated by Formula (1)

For a typical example of the *LOC* determination, see Annex F.

4.4.2.3 Extended procedure

If Formula (2) is not fulfilled (the *LAC* does not correspond to the apex) the full explosion region, near the *UEL* branch, shall be investigated using the following procedures (after carrying out steps 1 and 2) (see Figure 3):



Key			
1	explosion region	X	molar fraction in %
2	line of stoichiometric concentration fuel / air	IN	inert gas
3	line $x_{TS} = 1,2 LEL$	TS	test substance
LEL	lower explosion limit in air	Air	air
UEL	upper explosion limit in air	•	additional tests at X_{Air} , L
LAC	limiting air concentration	0	no explosion
		×	explosion

Figure 3 — Extended procedure scheme for the determination of the LAC

- 3c) Explosion limits shall be determined at a minimum of four additional points spaced equally along the *UEL* branch. These limits shall be obtained by keeping the inert fraction constant and varying the air fraction with a step size of a molar fraction of 0,5 % (see Figure 3).
- 4c) The location of the *UEL* branch shall be determined by plotting the experimental points on a triangular diagram and drawing a curve through the points. This can be carried out mathematically using a spline interpolation procedure or graphically. If the former method is used a check shall be made that the calculated curve passes through the experimental points. The *LAC* is found by drawing a tangent to the *UEL* curve, which is parallel to the inert axis.
- 5c) The LAC obtained in step 4c shall be confirmed by five additional tests at constant air fraction $x_{air,LAC}$ by varying the amount of flammable substance (see Figure 3). The step size for these tests shall be a molar fraction of 2,5 % of the test substance. These additional tests shall be carried out within a concentration range of a molar fraction of 5 % below the osculation point of the tangent (see step 4c) to a molar fraction of 5 % above the osculation point. If ignition is found, an additional explosion limit shall be measured at the corresponding test substance content and the interpolation procedure shall be repeated.

The LOC is then calculated by Formula (1).

For a typical example of the *LOC* determination, see Annex F.

NOTE For chemically unstable test substances with high intra-molecular energies, e.g. acetylene, 1,2-butadiene, ethylene oxide etc., the LOC may be zero and the UEL 100 %.

4.4.2.4 Oxygen analysis

In both cases – short procedure and extended procedure - an oxygen analysis shall be carried out for at least one LAC mixture. The result of the oxygen analysis shall not deviate more than a molar fraction of \pm 0,2 % from the LOC calculated by Formula (1).

If the tube method (T) is used the test mixture sample for oxygen analysis shall be taken at the outlet of the ignition vessel before igniting the mixture. For the bomb method (B) the test mixture for oxygen analysis shall be prepared in the bomb with a slight overpressure, to allow a sample to be taken for analysis.

4.5 Recording of results

4.5.1 General

According to this European Standard, all details specified in Clause 6 shall be provided. The result shall be expressed as a molar fraction.

4.5.2 Determination of explosion limits

The evaluation of the test is based on the test mixture with which five tests showed that an explosion just failed. The uncertainty of measurement which is permissible according to this European standard shall be stated, this is the maximum value of ± 10 % relative, or ± 0.2 % absolute (see 4.2.3.3 and 4.3.3.4). A smaller uncertainty of measurement may be stated if it can be derived from the accuracies of the test mixture production. The absolute uncertainty of measurement is calculated from the relative uncertainty of measurement and the value confirmed by the five tests.

As the measurements are carried out to generate safety data, the results are recorded as follows:

LEL: value confirmed by five tests – absolute deviation.

UEL: value confirmed by five tests + absolute deviation.

In addition, the value for the last ignition shall be stated since it allows the step size to be calculated.

NOTE 1 As the values are obtained for safety purposes, for *LEL* the lowest value respectively for *UEL* the highest value is used instead of the mean values.

NOTE 2 The specific conditions and the objective of the method described in this standard do not permit the results to be evaluated by conventional statistical methods. Such methods are not applicable here as the conditions regarding the distributions of random deviations are not met and systematic deviations, caused by the influence of the conditions of measurement, cannot be separated from random deviations. Consequently, the details of the apparatus and procedure used are required to be noted in the test report (see Clause 6).

The uncertainty of measurement for the *LEL* and *UEL* is essentially determined by the error in the amount of the test substance in the test mixture (see Table 1) and the size of the incremental change in test substance content.

4.5.3 Determination of the limiting oxygen concentration

When 'short procedure' is used the evaluation of the test is based on the test mixture with which five tests showed that an explosion just failed.

When 'Extended procedure' is used the evaluation of the test is based on the graphically determined *LAC*.

The LOC as calculated from LAC is rounded to the nearest 0.1 %.

5 Verification

The verification of the apparatus and procedure shall be done according to Annex G.

The oxygen analyser has to be calibrated in the range between a molar fraction of 0 % and 21 % oxygen.

6 Test report

The test report shall give the following information:

- a) reference to this European Standard;
- b) laboratory name, operator and date;
- c) test conditions: test temperature and ambient pressure; humidity in case of difficult to ignite substances;
- d) sample identification: composition, purity and source;
- e) oxidizer identification:
 - 1) atmospheric or synthetic air;
 - 2) composition, purity and amount of added inert.
- f) test apparatus:
 - 1) in the case of method T:
 - i) preparation of test mixture.
 - 2) in the case of method B:
 - i) vessel shape and volume;

- ii) ignition system;
- iii) preparation of test mixture.
- g) what was determined (*UEL*, *LEL*, *LOC*):
 - 1) the values of the *LEL*, *UEL*, *LOC* according to the respective explosion criterion;
 - 2) the uncertainty of measurement of the test substance content in the test mixture;
 - 3) in case of *LEL* or *UEL* determination the test substance content in the test mixture closest to the *LEL* and / or the *UEL* at which ignition was observed.
 - 4) In case of *LOC* the amounts of test substance and inert gas at *LOC*
 - 5) a graphical representation of the explosion region including the point of tangency if the *LOC* was determined using the "combination of experiments and graphical evaluation" or the extended procedure.

A test mixture is considered non-explosive if, for five tests carried out under the same conditions, the explosive criterion is not achieved.

An example of an appropriate form that may be used for a test report is given in Annex H.

Annex A

(normative)

Method for determination of the explosion limits and limiting oxygen concentration of substances that are difficult to ignite

A.1 Background

This annex specifies the modifications to the tube method which are necessary when determining explosion limits in air or air / inert mixtures, and limiting oxygen concentration, at atmospheric pressure and at temperatures from ambient temperature up to 200° C for substances which are difficult to ignite.

These difficult-to-ignite substances have large quenching distances. They include for example ammonia, amines and partly halogenated compounds and mixtures containing high proportions of such substances.

The bomb method has been found to be unsuitable for these substances.

A.2 Explanation

A.2.1 Explosion criterion — flame detachment

Because difficult to ignite substances often show no clear flame detachment it is recommended to use a video camera to record and review the tests for method "T". Frame by frame analysis may be needed to make a final decision.

The criterion for an explosion (self-propagating combustion) is the upward movement of the flame from the spark gap for at least 100 mm. (For examples see Annex C).

Alternatively, if a halo forms it shall reach within 200 mm of the top of a 500 mm tube; this shall also count as an ignition.

A.2.2 Degree of halogenation

The degree of halogenations is the number of halogen atoms in the molecular structure divided by the number of H-atoms in the molecular structure.

A.3 Apparatus

A.3.1 Test vessel

The test vessel is an upright cylindrical test vessel made of glass or another transparent material (e.g. polycarbonate) with an inner diameter of (80 ± 2) mm and a length of 500 mm. An inlet pipe for the test mixture with a three-way valve has to be located at the bottom of the tube and an outlet pipe and pressure vent in the upper part.

The bottom and top of the vessel may be made of other material. Any material used, however, has to be free of catalytic effects, and special care has to be taken to ensure it is corrosion resistant to the test substance and to the reaction products which may be HF or HCl.

A.3.2 Reagents and materials

Special care has to be taken that the chemically inert materials used for gaskets and mountings are resistant to the reaction products.

The humidity of air can have an influence on the combustion reaction. Therefore, the humidity of air should be noted and reported.

A.3.3 Ignition source

The ignition source requirements are more stringent than for normally flammable materials.

A series of induction sparks between two electrodes is used as the ignition source.

The electrodes shall end (60 ± 1) mm above the bottom of the test vessel.

Stainless steel is a suitable material for the electrodes. The electrodes shall be pointed rods with a diameter of maximum 3 mm. The angle of the tips shall be $(60 \pm 3)^{\circ}$. The distance between the tips shall be $(5 \pm 0,1)$ mm. If the electrodes are mounted through the bottom of the tube, the distance between the fittings shall be at least 40 mm. The electrodes shall be mounted in the vessel so that they are gas tight at the highest pressures generated during the test. The mounting shall be resistant to heat and the test mixture, and provide adequate electrical resistance from the test vessel body.

A high voltage transformer, with a root mean square of 15 kV and a short circuit current of 30 mA shall be used for producing the ignition spark. The primary winding of the high voltage transformer shall be connected to the mains via a timer set to the required discharge time.

The spark discharge time shall be adjusted to 0,2 s.

The power of the induction sparks is dependent on the gas mixture and its pressure in air at atmospheric conditions. According to calorimetric and electric measurements, such a source gives a spark with a power of approximately 10 W.

Special care has to be taken that the tips of the electrodes are not corroded and thus enlarging the distance between the tips of the electrodes.

A.3.4 Equipment for preparing the test mixture

See 4.2.3.3.

The metering devices and the equipment for preparing the test mixture shall be designed in such a way that the uncertainty of measurement of the test substance content in the test mixture is not higher than \pm 0,2 % absolute.

A.4 Safety equipment

The safety measures described in Annex E shall be followed. Special care has to be taken to comply with E.2 b) and E.2 f).

A.5 Preparation of the test mixture

See 4.2.4. In addition to halogenated hydrocarbons with a degree of halogenation greater than 0,8, it is necessary to establish a relative humidity of air of 50 % (as determined at room temperature).

A.6 Procedure

A.6.1 Determination of LEL and UEL

See 4.2.4. For halogenated hydrocarbons with a degree of halogenations greater than 0,8, the explosion limits and limiting oxygen concentration shall be determined with moist air (RH $50\,\%$ at ambient temperature).

There is no 'rule of thumb' to estimate the explosion limits.

However, if the degree of halogenation is > 1,5 the compound is likely to have no explosion range at ambient pressure and temperature. At elevated temperatures and / or pressures, an explosion range may however exist. In such cases, a larger step width could be used to verify that no explosion range exists. This may not be valid for halogenated ethers e.g. anesthetics and halogenated hydrocarbons with double or multiple bonds. Although their degree of halogenation is > 1,5, they may have an explosion range at ambient conditions.

A.6.2 Determination of LOC

The extended procedure according to 4.4.2.3 has to be used (see also Annex F.2 of this standard). The short procedure according to 4.4.2.2 has been found to be unsuitable for these substances.

Annex B

(informative)

Conversion of the values for the explosion limits

B.1 Abbreviations and symbols

D density ratio

F molar mass ratio (ratio of molar masses)

M molar mass

m mass

 \dot{m} mass flow

n mole number (amount of substance)

Q compressibility factor ratio

V volume

 $V_{\rm mol}$ molar volume

 \dot{V} volume flow

 β mass concentration

w mass fraction

x mole fraction

Z compressibility factor

 φ volume fraction

 ρ real density

(A) air

at under atmospheric conditions, i.e. approximately 1 bar and approximately 20 °C

liq liquid state

(G) gas, i.e. sample or air

N standard conditions, i.e. at 1,013 bar and 0 $^{\circ}$ C

id for (test mixtures as) ideal gas

mol to characterize the molar volume

(S) sample

(TM) test mixture

B.2 Substance characteristics of air

 ρ_{at} 1,2 · 10³ g / m³

M(A) 29,0 g / mole

B.3 Definitions

Volume fraction in %:
$$\phi(S) = \frac{\dot{V}(S)}{\dot{V}(S) + \dot{V}(A)} \cdot 100$$
 (B.1)

When contents are stated as a volume fraction, the volume flows have to be related to the same pressure and to the same temperature and all components have to be gaseous under these conditions.

Mole fraction in %:
$$x(S) = \frac{n(S)}{n(S) + n(A)} \cdot 100$$
 (B.2)

The mole fraction is independent of pressure and of temperature.

Mass fraction in %:
$$w(S) = \frac{\dot{m}(S)}{\dot{m}(S) + \dot{m}(A)} \cdot 100$$
 (B.3)

The mass fraction is independent of pressure and of temperature.

Mass concentration at atmospheric conditions in g / m³:

real:
$$\beta_{at}(S) = \frac{m(S)}{V_{at}(TM)}$$
 (B.4)

or

ideal:
$$\beta_{at}^{id}(S) = \frac{m(S)}{V_{ot}^{id}(TM)}$$
 (B.5)

B.4 Mixture preparation

According to 4.2.4 and 4.3.4, it is possible to produce the test mixture in different ways:

a) Metering of air and gaseous samples using volumetric gas metering pumps:

The volume fraction of the sample can be directly adjusted on the gas metering pump. In this case, the volume fraction of the test substance is equal to the volume fraction of the sample. Volume flows metered with gas metering pumps are brought together under atmospheric conditions to form the test mixture.

b) Metering of air and gaseous samples using mass flow controllers:

If the mass flow controllers are calibrated in the volume flow in the standard state V_N (G), the mass flow will be obtained by multiplying this volume flow by the density of the gas in the standard state ρ_N (G) as follows:

$$\dot{m}(G) = \rho_N(G) \cdot \dot{V}_N(G) \tag{B.6}$$

The mass flow of the test substance is equal to the mass flow of the sample. Mass flows metered with mass flow controllers are brought together under atmospheric conditions to form the test mixture.

c) Metering of air with a mass flow controller and of liquid samples with a volumetric metering pump:

For the mass flow of air, the formula given in b) is valid.

The mass flow of the liquid sample is obtained from the volume flow of liquid \dot{V}_{liq} (S), which can be adjusted on the metering pump, and the density of the liquid ρ_{liq} (S):

$$\dot{m}(S) = \rho_{lia}(S) \cdot \dot{V}_{lia}(S) \tag{B.7}$$

The mass flow of the test substance is equal to the mass flow of the sample. Generally with this method, the test mixture is produced at increased temperature (i.e. the temperature in the evaporator tube). Only by working can the required mass concentration of test substance in the test mixture be produced. When mixed, both flows should have this temperature. The partial pressure of the test substance in the test mixture can approximate to the vapour pressure of the sample at this temperature. As air and test substance thus are not at the same pressure when mixed, the conditions for the conversion into volume fractions are not fulfilled.

d) By partial pressures:

In this case moles of gas, air and inert gas are mixed together.

B.5 Conversion

As the real volume of the test mixture is generally unknown, it is assumed for the conversions that the test mixture is an ideal gas.

The mass concentration calculated in this way is a quantity which can be used only for comparison purposes. In many cases, it is impossible to produce a mixture with this mass concentration of sample under atmospheric conditions. Therefore, it is necessary to check whether the conversion is possible and reasonable, taking into account the pressure and temperature conditions at which the measurements have been carried out.

The following substance characteristics are required for the conversion:

— the density ratio *D* of the densities of the gaseous sample $\rho(S)$ and of the air $\rho(A)$:

$$D = \frac{\rho(S)}{\rho(A)} \tag{B.8}$$

— the molar mass ratio F of the molar masses of the sample M(S) and of the air M(A):

$$F = \frac{M(S)}{M(A)} \tag{B.9}$$

— the compressibility factor ratio Q of the compressibility factors of the sample Z(S) and of the air Z(A):

$$Q = \frac{Z(S)}{Z(A)} \tag{B.10}$$

Furthermore,
$$\frac{D \cdot Q}{F} = 1$$
 (B.11)

If the densities are not known but the real molar volumes for the gaseous sample $V_{\text{mol}}(S)$ and for the air $V_{\text{mol}}(A)$ are known, the following is valid:

$$D = F \cdot \frac{V_{\text{mol}}(A)}{V_{\text{mol}}(S)}$$
(B.12)

For the conversion, substance characteristics have to be selected for the pressure and temperature conditions under which the flows of the components were brought together during the determination. If the sample is a mixture or if an air / inert gas mixture is used as oxidizer in the place of air, the substance characteristics are to be calculated according to the well-known formulae of physical chemistry or to be determined by measurements on the mixtures. If the substance characteristics are unknown or cannot be calculated, a conversion of the values will not be possible.

Table B.1 — Formulas for the conversion

	Test mixture prepared as			
To be calculated:	Volume fraction of flammable substance in the test mixture $\varphi(S)$	Mass fraction of flammable substance in the test mixture $w(S)$	Mole fraction of flammable substance in the test mixture $x(S)$	
Volume fraction of flammable substance in the test mixture $\varphi(S)$ in %	-	$\phi(S) = \frac{w(S)/D \cdot 100}{w(S)/D + \left[100 - w(S)\right]}$	$\phi(S) = \frac{Q \cdot x(S) \cdot 100}{Q \cdot x(S) + \left[100 - x(S)\right]}$	
Mass fraction of flammable substance in the test mixture w(S) in %	$w(S) = \frac{\phi(S)/D \cdot 100}{\phi(S)/D + \left[100 - \phi(S)\right]}$	-	$w(S) = \frac{x(S) \cdot F \cdot 100}{x(S) \cdot F + \left[100 - x(S)\right]}$	
Mole fraction of flammable substance in the test mixture $x(S)$ in %	$x(S) = \frac{\phi(S) \cdot 100}{\phi(S) + \left[100 - \phi(S)\right] \cdot Q}$	$x(S) = \frac{w(S)/Q \cdot 100}{w(S)/Q + \left[100 - w(S)\right]}$	-	
Ideal mass concentration of flammable substance in the test mixture at atmospheric conditions β_{at}^{id} (S) in g / m ³	$\beta_{at}^{id}(S) \approx \rho_{at}(A) \cdot D \cdot \frac{\phi(S)}{100}$	$\beta_{at}^{id}(S) \approx \frac{\rho_{at}(A) \cdot F \cdot w(S)}{w(S) + [100 - w(S)]F}$	$\beta_{at}^{id}(S) \approx \rho_{at}(A) \cdot \frac{D \cdot Q \cdot x(S)}{Q \cdot x(S) + [100 - x(S)]}$	

Annex C (informative)

Examples to describe flame detachment

Dimensions in millimetres



Figure C.1 — Flame detachment

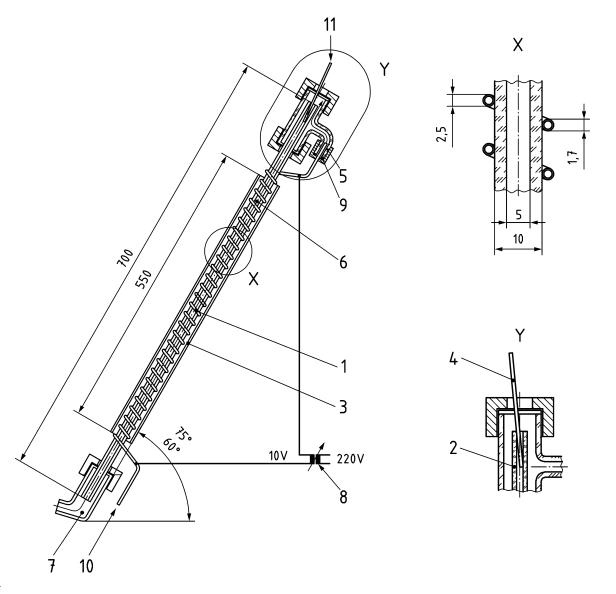


Figure C.2 — Halo (undetached flame)

Annex D (informative)

Example of recommended evaporator equipment

Dimensions in millimetres



Key

- 1 glass evaporator tube
- 2 helical steel tube (tight fit when heated)
- 3 heat shield (glass tube)
- 4 needle of the metering device for the liquid flammable substance
- 5 glass sealing cap with septum
- 11 liquid

- 6 thermocouple
- 7 test mixture outlet
- 8 low-voltage supply
- 9 gasket
- 10 air

Figure D.1 — Evaporator equipment for producing test mixtures from liquid flammable substances

The glass evaporator tube (1) equipped with tightly fitted helical steel tube (2) is located in a glass tube heat shield (3) of sufficient length. The evaporator tube is heated by applying a low-voltage to the helical tube. The temperature in the evaporator tube is controlled and measured by a thermocouple positioned inside the tube (6). The air is preheated as it flows up through the hot helical steel tube. Thus, the preheated air enters the top of the evaporator tube, together with the liquid from the needle of the metering device (4). The needle is arranged in such a way that the liquid flowing from the tip of the needle immediately comes into contact with the wall of the evaporator tube. Formation of liquid drops as the sample flows from the needle tip has to be avoided. To achieve the most efficient evaporation, the evaporator is inclined at an angle of 60° to 75° to the horizontal and the temperature is maintained so that all the liquid evaporates in the upper half of the tube.

Annex E (normative)

Safety measures

E.1 General

The following measures shall be followed as appropriate for the method used.

E.2 General safety measures

- a) Check the apparatus for gas tightness.
- b) Ensure that the test mixtures and waste gases are vented without risk.
- c) Ensure that the heating device, if used, is adequately ventilated so that no explosive atmosphere can form in the vicinity of the heater (e.g. from leakage of the test apparatus).
- d) When mixtures are produced, check to ensure that they do not react together prior to ignition, and that they can be stored without any change if necessary.
- e) If explosive mixtures are prepared in a separate vessel at pressures higher than 1 bar, it has to be ensured that the vessel and the lines can withstand the maximum explosion pressure in case of accidental ignition.
- f) Assess the potential toxic hazard of the samples, test mixtures and waste products and ensure appropriate protective measures are in place.
- g) Guard the high-voltage connections on the transformer, the supply lines and the electrodes in the test vessel so that protection against electric shock hazard is ensured.

E.3 Additional safety measures concerning the tube method

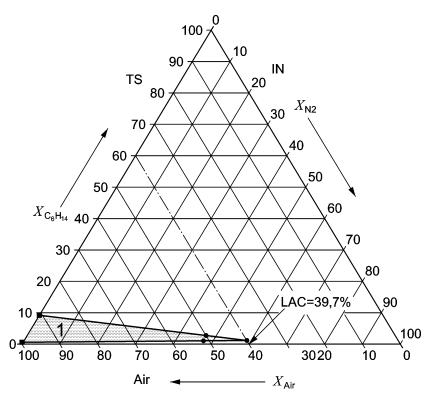
- a) Ideally, the initial ignition tests should be carried out using a test mixture whose test substance content lies outside the estimated explosion range.
- b) Ensure adequate protection is provided in the event that the test vessel should burst; carry out the work behind a splitter protection device (e.g. unbreakable pane of the heating cabinet).
- c) In the event that the test vessel bursts, the flow of test substance should be stopped at once.
- d) Ensure that the pressure venting device of the test vessel is adequate and operational. Ensure that adequate containment is provided should pressure venting occur.
- e) Ensure that the exhaust system will not generate any underpressure inside the test vessel, to avoid excess air being sucked in.

Annex F (informative)

Examples of the determination of the LOC

F.1Example 1: determination of the LOC - short procedure

Figure F.1 shows the results of the determination of the LAC for a ternary n-hexane, air and nitrogen system, using the tube method (T). This result is typical of that obtained for a vaporized liquid.



17	
к	Δ1/

1	explosion region	N_2	inert gas (nitrogen)
LAC	limiting air concentration	C_6H_{14}	test substance (n-hexane)
Y	molar fraction in %	Air	air

Figure F.1 — Determination of the LAC of a ternary system of n-hexane, air and nitrogen at $100\,^{\circ}\text{C}$ and ambient pressure

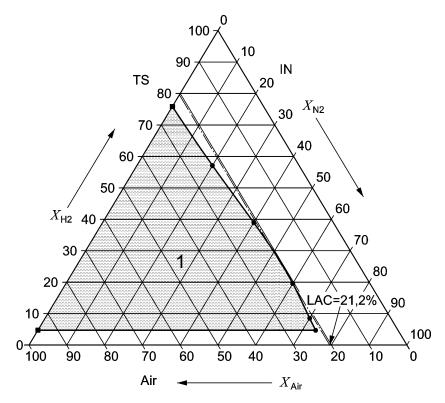
From the molar fraction of 39,7 % for LAC the molar fraction of LOC is calculated as follows.

$$LOC = 0,209 \times 39,7\% = 8,3\%$$
 (F.1)

F.2Example 2: determination of the *LOC* – extended procedure

For some ternary systems the *LAC* does not occur at the apex of explosion region. Examples are flammable substances with high intramolecular energies and test substances with molecular heat capacities similar to nitrogen, e.g. hydrogen, carbon monoxide and some other substances.

As an example Figure F.2 shows the *LAC* determination for a ternary system of hydrogen, nitrogen and air according to the bomb method (B).



Key

 $\begin{array}{lll} 1 & \text{ explosion region } & N_2 & \text{ inert gas (nitrogen)} \\ \text{LAC} & \text{ limiting air concentration } & H_2 & \text{ test substance (hydrogen)} \\ \end{array}$

X molar fraction in % Air air

Figure F.2 — Determination of the *LAC* of a ternary system of hydrogen, air and nitrogen at 20 °C and ambient pressure

From the molar fraction of 21,2 % for the *LAC* the molar fraction for the *LOC* is calculated as follows.

$$LOC = 0,209 \times 21,2\% = 4,4\%$$
 (F.2)

Annex G (normative)

Verification

This verification procedure shall be used for new apparatus as well as for checking the performance of existing apparatus. Existing apparatus shall be checked every 12 months or when any part of the apparatus has been changed or renewed. The verification shall be carried out according to the procedures given in 4.2.5 and 4.3.5 respectively with at least one of the substances listed in Table G.1 and G.2. It is recommended to use ethene or methane for verification if the apparatus is mainly used to determine explosion limits of gases. In the case the apparatus is mainly used to determine the explosion limits of flammable liquids, it is recommended to use n-hexane or 1,3,5-trimethylbenzene. Verification will be confirmed if the *LEL* value and the *UEL* value obtained from these experiments are within the ranges given in Table G.1 and G.2 respectively. The data given in Table G.1 and G.2 is not corrected according to 4.5.

The purity of ethene and methane respectively used as a reference substance has to be at least 99,8 %. The purity of n-hexane has to be 99,0 % or better and that of 1,3,5-trimethylbenzene 98,0 % or better.

If the apparatus is to be used at elevated temperatures, it has to be verified that the temperature difference inside the test vessel is not more than 10 K.

Table G.1 — Data for verification of the apparatus with respect to the lower explosion limit

Substance	T °C	LEL Method T mole percent %	LEL Method B mole percent %
Ethene	RT ¹	2,5 ± 0,10	2,5 ± 0,10
Methane	RT¹	4,4 ± 0,20	4,6 ± 0,20
n-Hexane	RT¹	1,0 ± 0,10	1,0 ± 0,10
1,3,5-Trimethylbenzene 70 0,8		0,8 ± 0,08	0.8 ± 0.08
¹ RT = Room temperature			
NOTE The data have been obtained at 1 bar and with air as an oxidizer.			

Table G.2 — Data for verification of the apparatus with respect to the upper explosion limit

Substance	T °C	UEL Method T mole percent %	UEL Method B mole percent %
Ethene	RT¹	32,3 ± 0,40	30,8 ± 0,70
Methane	RT^1	16,6 ± 0,20	16,8 ± 0,20
n-Hexane	40	8,3 ± 0,10	7,7 ± 0,25
1,3,5-Trimethylbenzene 130		7,1 ± 0,20	6,6 ± 0,10
¹ RT = Room temperature			
NOTE The data have been obtained at 1 bar and with air as an oxidizer.			

- NOTE 1 The values given in Tables G.1 and G.2 show the bandwidth of determinations by several laboratories, which participated in the development of the method. For reasons stated in 4.5, the results of these determinations cannot however be evaluated according to the rules for interlaboratory tests.
- NOTE 2 The values given in Table G.1 and G.2 can be converted to other units by using the formula given in Annex B.
- NOTE 3 Additional special sets of data obtained with an apparatus whose verification has been confirmed, can be useful to check the performance of the apparatus in special explosion ranges or special types of flammable substance.

Annex H

(informative)

Example of a form expressing the results¹

Test Report

Name of the testing house:			
Determination of LEL [], UEL	Method T []B[]		
Sample:			Purity:
			Source:
Oxidizer: air / mixture of:		Inert gas(in case of LOC de	etermination):
Test conditions			
Temperature in the test vessel:		°C	
Humidity of air (in case of Anne	x G substances) mol%	
Test apparatus			
Method T		Method B	
Cylinder: l:	mm	Shape: cylindrical [] length to	o diameter:
		spherical [] volume:	
		Ignition source:	
Production of the test mixtur			
= -		tric metering gas pumps)	= =
	= =	ss flow controllers	==
Deviation of the molar fraction	of test substan	ce in test mixture %	relative / absolute
LEL, UEL: Results of the att	empts to igni	ite LOC	
	Molar		Molar fraction %
	fraction %		
last ignition		LAC	
5 tests without ignition		Inert gas content at LAC	
Absolute deviation of the test mixture production		Test substance amount	
Result of the determination		Absolute deviation of t test mixture production	he
	<u>I</u>	Result of the determination	

 $^{^{1}}$ Only the user of this European Standard may copy this form for reporting the results of the determinations.

At the temperature stated, the sample has no explosion range in this oxidizer		
Comments:		
Date	Operator:	
Signature		

Annex I

(informative)

Significant Changes between this European Standard and EN 1839:2012 and EN 14756:2006

The significant changes with respect to EN 1839:2012 and EN 14756:2006 are listed in Table I.1.

Table I.1 — The significant changes with respect to EN 1839:2012 and EN 14756:2006

		Туре		
Significant changes	Annex / Clause	Minor and editorial changes	Extension	Major technical changes
Merged with EN 14756. Minor technical changes but significant editorial	Whole standard	X		

NOTE The technical changes referred include the significant technical changes from the EN revised but is not an exhaustive list of all modifications from the previous version.

Explanations:

Minor and editorial changes: clarification

decrease of technical requirements

minor technical change editorial corrections

Changes in a standard classified as 'minor and editorial changes' refer to changes regarding the previous standard, which modify requirements in an editorial or a minor technical way. In addition, changes of the wording to clarify technical requirements without any technical change are classified as 'minor and editorial changes'.

A reduction in the level of an existing requirement is also classified as a 'minor and editorial change'.

Extension: addition of technical options

Changes in a standard classified as 'extension' refers to changes regarding the previous standard, which add new or modify existing technical requirements, in a way that new options are given, but without increasing requirements for equipment that was fully compliant with the previous standard. These 'extensions' will therefore not have to be considered for products in conformity with the preceding edition.

Major technical changes: addition of technical requirements

increase of technical requirements

Changes in a standard classified as 'major technical change' refer to changes regarding the previous standard, which add new or increase the level of existing technical requirements, in a way that a product in conformity with the previous standard will not always be able to fulfil the requirements given in the standard. 'Major technical changes' have to be considered for products in conformity with the previous edition.

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 to provide one voluntary means of conforming to essential requirements of 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.

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 Annex II of Directive 2014/34/EU

Essential Requirements of Directive 2014/34/EU	Clause(s)/sub-clause(s) of this EN	Remarks/Notes
Annex II, Clause 1.01; 1.0.4; 1.0.6a; 1.0.6c; 1.2.1, 1.2.2; 1.2.3 and 1.5.7		

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.

Bibliography

[1] ISO 10156:2010, Gases and gas mixtures — Determination of fire potential and oxidizing ability for the selection of cylinder valve outlets



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