INTERNATIONAL STANDARD

ISO 11114-3

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Transportable gas cylinders — Compatibility of cylinder and valve materials with gas contents —

Part 3:
Autogenous ignition test in oxygen atmosphere

Bouteilles à gaz transportables — Compatibilité des matériaux des bouteilles et des robinets avec les contenus gazeux —
Partie 3: Essai d'auto-inflammation sous atmosphère d'oxygène

Reference number
ISO 11114-3:1997(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11114-3 was prepared by the European Committee for Standardization (CEN) in collaboration with ISO Technical Committee TC 58, *Gas cylinders*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

ISO 11114 consists of the following parts, under the general title *Transportable gas cylinders — Compatibility of cylinder and valve materials with gas contents*:

— *Part 1: Metallic materials*

— *Part 2: Non-metallic materials*

— *Part 3: Autogenous ignition test in oxygen atmosphere*

Annexes A, B and ZZ of this part of ISO 11114 are for information only.

Annex ZZ provides a list of corresponding International and European Standards for which equivalents are not given in the text.

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Foreword

This text of EN ISO 11114-3:1997 has been prepared by Technical Committee CEN/TC 23 "Transportable gas cylinders", the secretariat of which is held by BSI, in collaboration with Technical Committee ISO/TC 58 “Gas cylinders”.

The text of the draft standard was submitted to the formal vote and was approved by CEN as EN ISO 11114-3 on 97-09-18.

This European Standard has been submitted for reference into the RID and/or in the technical annexes of the ADR.

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 1998, and conflicting national standards shall be withdrawn at the latest by April 1998.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

This Standard is one of a three-part Standard concerning compatibility of gases and gas mixtures with materials :

- Part 1 : Metallic materials ;
- Part 2 : Non metallic materials ;
- Part 3 : Autogenous ignition test in oxygen atmosphere.

The following test method is referenced in EN ISO 11114-1.

Further information about oxygen compatibility is given in EN ISO 11114-1 and prEN ISO 11114-2.

Other oxygen compatibility test methods include oxygen index (see ISO 4589), heat of combustion and adiabatic compression on materials.

1 Scope

This standard specifies a test method to determine the autogenous ignition temperature of non metallic materials in pressurized gaseous oxygen.

Autogenous ignition temperature is a criterion for ranking materials, and can be used to assist with the choice of materials used in the presence of gaseous oxygen.

Annex B of this standard is a comprehensive bibliography of the published material on which this standard is based.

NOTE : This standard can be used for the selection of nonmetallic materials for gas cylinders and accessories ; for example, to select the materials in order to meet the requirement for type-testing for oxygen compatibility of all oxygen gas cylinder valves as specified in EN 849.

2 Principle

A small quantity of the test material is heated in pressurized oxygen. A continuous recording is made of pressure and temperature in order to determine the point of autogenous ignition, at which point a sudden increase in temperature and pressure is observed (this point is known as autogenous ignition temperature).

3 Preparation of test samples

Test samples shall be prepared in such a manner as to prevent contamination.

Test samples may be in liquid or solid form. In the case of solids, the material shall be finely divided. A sample mass between 0,06 g and 0,5 g is used for each test.

NOTE : This mass should be adjusted to take into consideration the volume of the test cell. A sample of 0,5 g in a test cell volume of between 30 cm^3 and 250 cm^3 has been found to be suitable.

4 Test apparatus

Figure 1 gives an example of a suitable test apparatus. The test sample is put into a small, carefully cleaned inert sample holder, placed in a reaction chamber within an electric oven with power sufficient to raise temperature at a constant rate as specified in clause 6.

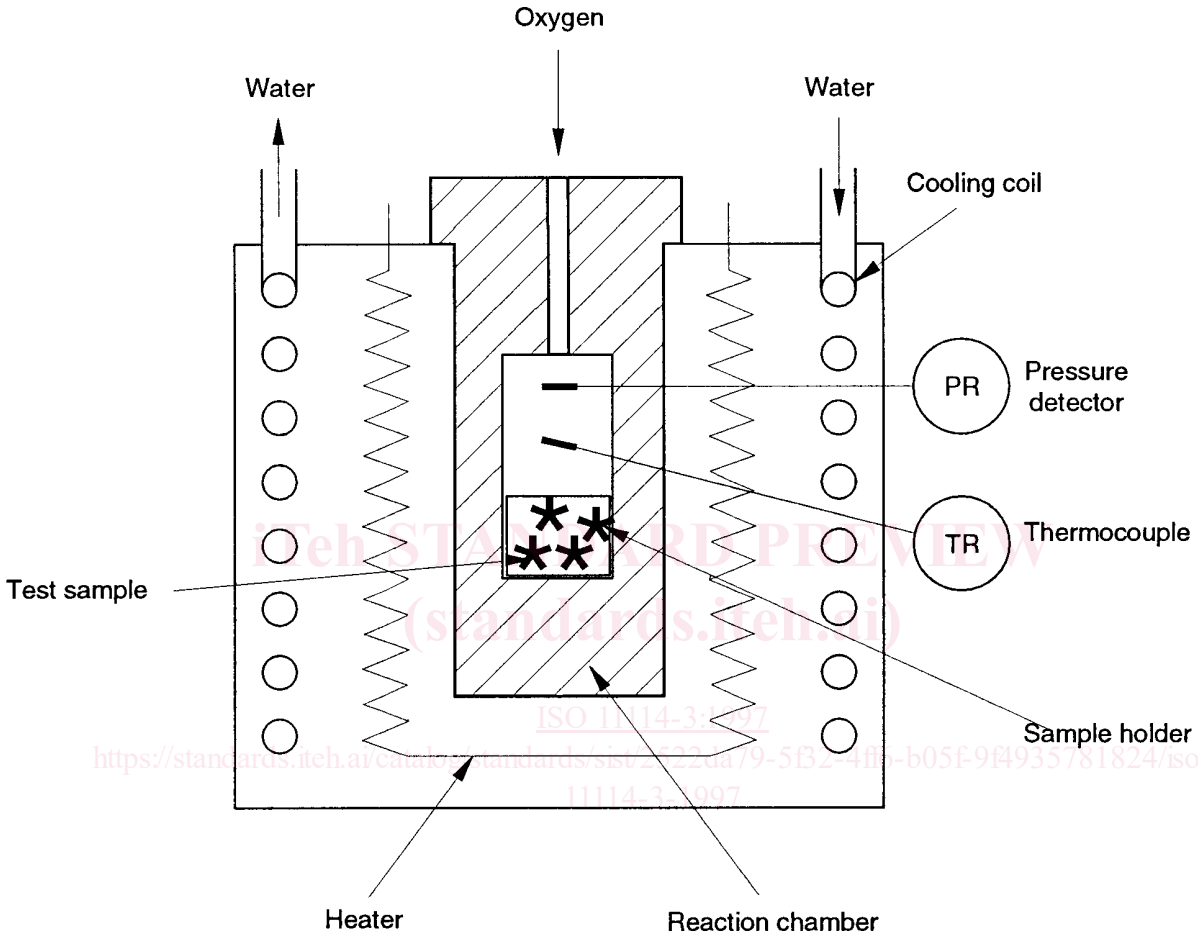


Figure 1 : Example of apparatus for ignition test

A thermocouple shall be positioned as closely as possible to the test sample. The thermocouple shall have an accuracy of ± 2 °C between 25 °C and 500 °C.

A pressure detector shall be provided. The pressure detector shall have an accuracy of 1 % at full scale.

A pressure regulating device may be used if it is intended to keep oxygen pressure constant during the test.

NOTE : Autogenous Ignition Temperature may depend on oxygen pressure. When the oxygen pressure is increased, the autogenous ignition temperature decreases and stabilizes beyond a certain pressure. Therefore, for ranking of materials, when the minimum Autogenous Ignition Temperature has to be determined, a starting pressure of 10 MPa (100 bar) is recommended.

The equipment, and in particular, the reaction chamber, shall be designed to resist violent internal reactions (explosions). In some designs, the temperature and pressure measuring devices could be exposed to the flame from the test sample.

5 Oxygen purity

The oxygen used for the test shall have a purity of at least 99,5 % (V/V). The hydrocarbon content shall be limited to 100 ppm by volume (volume fraction $\leq 10^{-4}$).

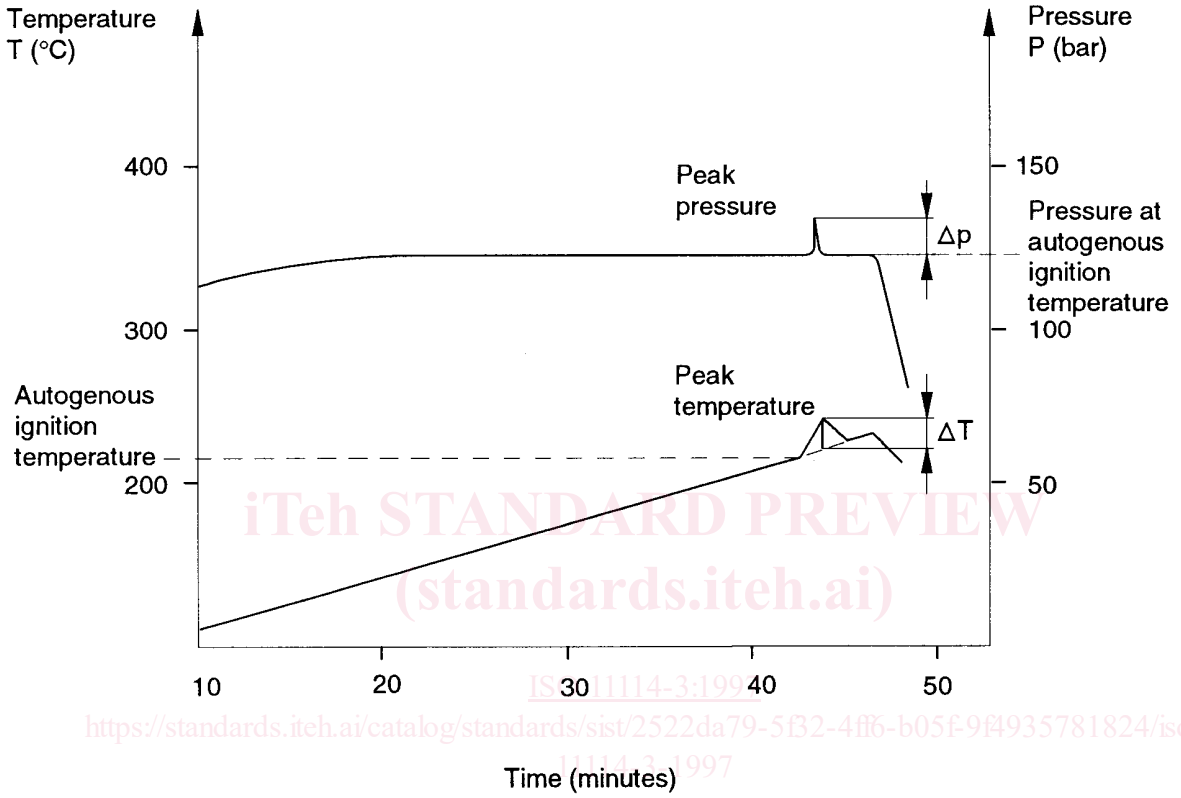
6 Test procedure

The sample holder containing the test sample is placed into the reaction chamber. The reaction chamber is then sealed and purged with oxygen to remove any air and any possible residual combustion products from preceding tests. The recommended purging procedure is to pressurize the reaction chamber to 1 MPa (10 bar) and vent to atmospheric pressure and to repeat this procedure 3 times. The oxygen pressure is then increased to the pressure required for the particular test.

The temperature is then raised at a constant heating rate up to autogenous ignition temperature or up to a maximum temperature of 500 °C. The heating rate shall be recorded. If constant pressure is desired, adequate control shall be performed.

From the continuous recording of the two parameters (temperature and pressure), the autogenous ignition temperature is determined, which corresponds to the sudden increase in temperature and pressure caused by the internal reaction (see figure 2).

NOTE : The use of a high heating rate (greater then 20 °C/min), when using an electrical filament furnace, may result in a lower autogenous ignition temperature than when using the normal heating rate (between 5 °C/min and 20 °C/min).



NOTE : The figure shows the temperature and pressure versus time curves for a typical autogenous ignition test carried out under nearly constant pressure.

Figure 2 : Typical autogenous ignition test curves

7 Results

The autogenous ignition temperature is the temperature, expressed in °C, at which the sudden increase of temperature and associated increase of pressure commences.

This point is determined from the temperature, pressure, time recording graph as illustrated in figure 2. The magnitude of the increase in temperature, ΔT and increase in pressure, Δp caused by the ignition are determined as illustrated in figure 2.

NOTE 1 : In certain cases, a material can be required to have an autogenous ignition temperature higher than a specified critical value.

NOTE 2 : The temperature and pressure increases, Δ T and Δp (see figure 2), may characterize the violence of the reaction. Special requirements can also apply to these two parameters.

NOTE 3 : Because the temperature and pressure increases, Δ T and Δp, are affected by the relationship between the size of the sample, the volume of the reaction chamber and the heating capacity of the oven, comparison between the results from different types of equipment cannot easily be made.

8 Test record

The test results shall be recorded on a test record sheet, an example of which is shown in annex A.

Annex A (informative)

Test Record Sheet

AUTOGENOUS IGNITION TEST EN ISO 11114-3	Test n° Date

1 - TEST PERFORMED FOR _____

2 - TESTED MATERIAL _____

Function _____

Conditions of Use Temperature in °C _____ Pressure in bar ____

Assumed generic type _____
Condition, Shape, appearance _____
Manufacturer _____
Supplier _____
Trade name _____

3 - TESTING CONDITIONS AND RESULTS

. Mass of test sample in grammes : _____
. Heating rate in °C/min : _____
. Starting pressure in bar (gauge) : _____

Pressure in bar			Temperature in °C		
at autogenous ignition test	Peak	Δp	autogenous ignition test	Peak	ΔT

Remarks :

4 - AUTOGENOUS IGNITION TEMPERATURE (°C)

5 - COMMENTS

Authorized signature
(and test laboratory identification)