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**Gas cylinders — Compatibility of cylinder  
and valve materials with gas contents —**

Part 3:

**Autogenous ignition test for non-metallic  
materials in oxygen atmosphere**

*Bouteilles à gaz — Compatibilité des matériaux de bouteilles et de  
robinets avec les contenus gazeux —*

*Partie 3: Essai d'auto-inflammation des matériaux non métalliques sous  
atmosphère d'oxygène*



Reference number  
ISO 11114-3:2010(E)

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## 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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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

This part of ISO 11114 was prepared by Technical Committee ISO/TC 58, *Gas cylinders*.

This second edition cancels and replaces the first edition (ISO 11114-3:1997). No significant technical changes have been made.

ISO 11114 consists of the following parts, under the general title *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 for non-metallic materials in oxygen atmosphere*
- *Part 4: Test methods for selecting metallic materials resistant to hydrogen embrittlement*

## Introduction

The following test method is referenced in ISO 11114-1 and ISO 11114-2.

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

Other oxygen compatibility test methods include oxygen index (see ISO 4589-3), heat of combustion and adiabatic compression on materials (see ISO 21010).



# Gas cylinders — Compatibility of cylinder and valve materials with gas contents —

## Part 3: Autogenous ignition test for non-metallic materials in oxygen atmosphere

### 1 Scope

This part of ISO 11114 specifies a test method to determine the autogenous ignition temperature of non-metallic materials in pressurized gaseous oxygen.

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

A comprehensive bibliography of the published material on which this part of ISO 11114 is based is included.

It is intended that this part of ISO 11114 be used for the selection of non-metallic 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 cylinder valves for highly oxidizing gases as specified in ISO 10297.

### 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 the autogenous ignition temperature).

### 3 Preparation of test samples

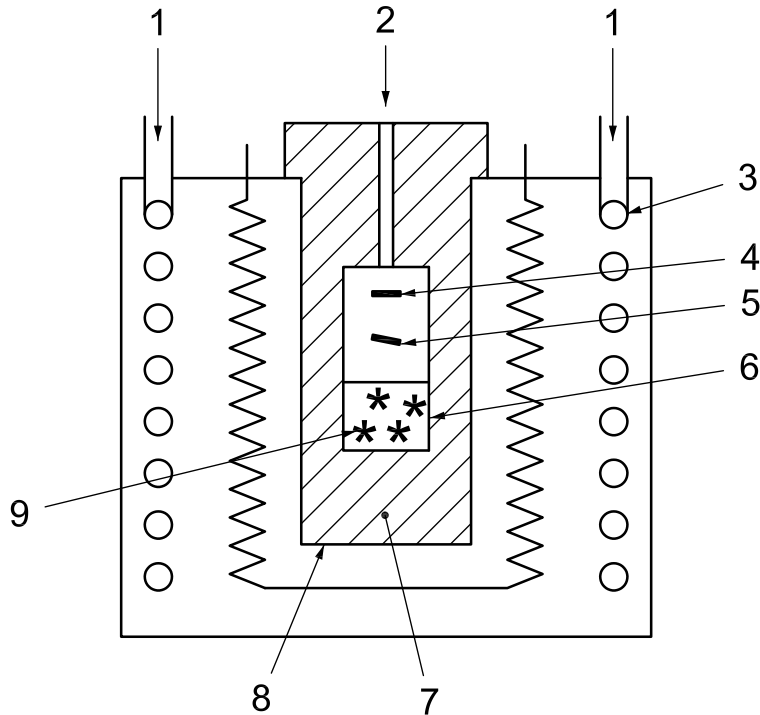
Test samples shall be prepared in such a manner as to prevent contamination.

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.

This mass should be adjusted to take into consideration the volume of the test reaction chamber. A sample of 0,5 g in a test reaction chamber volume of between 30 cm<sup>3</sup> and 250 cm<sup>3</sup> 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 sufficient power to raise temperature at a constant rate as specified in Clause 6.



**Key**

- 1 water
- 2 oxygen
- 3 cooling coil
- 4 pressure transducer
- 5 temperature transducer
- 6 sample holder
- 7 reaction chamber
- 8 electrical heater
- 9 test sample

**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  $\pm 2$  °C between 25 °C and 500 °C.

A pressure transducer shall be provided. The pressure transducer 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.

The autogenous ignition temperature can 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 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 a volume fraction of 99,5 %. The hydrocarbon content shall be limited to  $100 \times 10^{-6}$  by volume (volume fraction  $\leq 10^{-4}$ ).

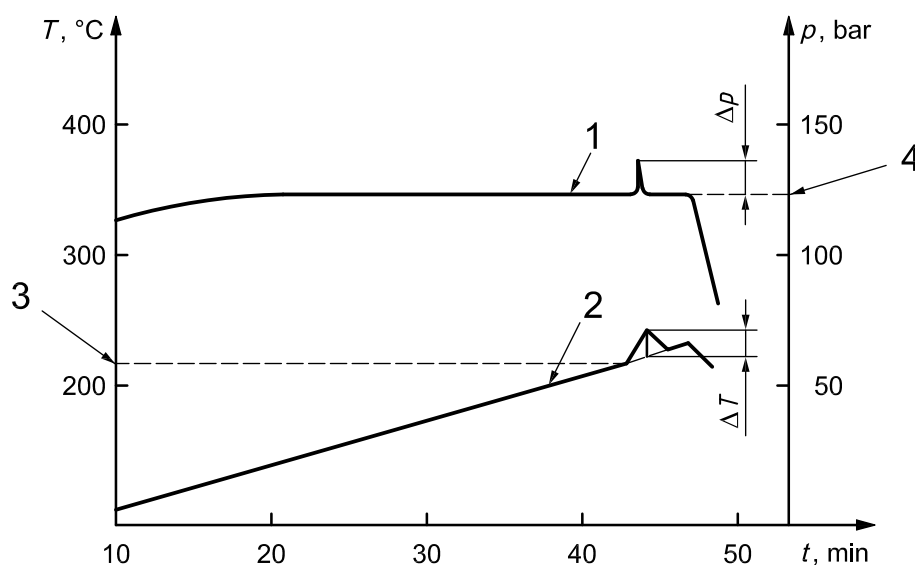
## 6 Test procedure

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 common purging procedure is to pressurize the reaction chamber to 10 bar and vent to atmospheric pressure and to repeat this procedure three 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 the 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 than 20 °C/min), when using an electrical filament furnace, can result in a lower autogenous ignition temperature than when using the normal heating rate (between 5 °C/min and 20 °C/min).



NOTE Figure 2 shows the temperature and pressure versus time graph for a typical autogenous ignition test carried out under nearly constant pressure.

### Key

- 1 pressure
- 2 temperature
- 3 autogenous ignition temperature
- 4  $\Delta p$

Figure 2 — Typical autogenous ignition test graph

## 7 Results

The autogenous ignition temperature is the temperature, expressed in degrees Celsius, at which the sudden increase in temperature and associated increase in pressure commences.

This point is determined from the temperature and pressure versus time recording graph as illustrated in Figure 2. The magnitude of the increase in temperature,  $\Delta T$ , and increase in pressure,  $\Delta p$ , caused by the ignition are determined as illustrated in Figure 2.

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

NOTE 2 The temperature and pressure increases,  $\Delta T$  and  $\Delta p$  (see Figure 2), characterize the violence of the reaction. Special requirements can also apply to these two parameters.

NOTE 3 Because the temperature and pressure increases,  $\Delta T$  and  $\Delta p$ , are affected by the relationship between the size of the sample, the volume of the reaction chamber and the heating capacity of the heater, a comparison between the results from different types of test apparatus 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 ISO 11114-3			Test N° Date		
<b>1 - TEST PERFORMED FOR CUSTOMER</b>					
_____					
<b>2 - TESTED MATERIAL</b>					
_____					
Function: _____					
Conditions of use: _____ Temperature, °C _____ Pressure, bar _____					
Assumed generic type: _____					
Condition, shape, appearance: _____					
Manufacturer: _____					
Supplier: _____					
Trade name: _____					
<b>3 - TESTING CONDITIONS AND RESULTS</b>					
Mass of test sample, g: _____					
Heating rate, °C/min: _____					
Starting pressure, bar (gauge): _____					
Pressure, bar			Temperature, °C		
Autogenous ignition test	Peak	$\Delta p$	Autogenous ignition test	Peak	$\Delta T$
Remarks:					
<b>4 - AUTOGENOUS IGNITION TEMPERATURE, °C</b>					
<b>5 - COMMENTS</b>					
Authorized signature (and test laboratory identification)					
The user of this form is allowed to reproduce this form.					

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