
**Rubber, vulcanized or
thermoplastic — Resistance to ozone
cracking —**

**Part 3:
Reference and alternative methods for
determining the ozone concentration
in laboratory test chambers**

*Caoutchouc vulcanisé ou thermoplastique — Résistance au
craquelage par l'ozone —*

*Partie 3: Méthode de référence et autres méthodes pour la
détermination de la concentration d'ozone dans les enceintes d'essai
de laboratoire*



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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html

This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 1431-3:2000), which has been technically revised.

The main changes compared to the previous edition are as follows:

- a calculation error has been corrected in the formula in [B.1.4](#);
- the value of gas constant has been corrected in the formulae in [C.2.4](#) and [C.3.4](#).
- [Annex A](#) has been changed from normative to informative, and a citation has been added to [Clause 8](#).

A list of all parts in the ISO 1431 series can be found on the ISO website.

Introduction

A number of techniques exist for the analysis of gaseous ozone/air mixtures used for ozone crack testing of rubbers. These include wet-chemical procedures, electrochemical cells, UV absorption and chemiluminescence with ethylene.

In principle, the wet-chemical, electrochemical and UV absorption methods are all absolute, but in practice they do not in general yield the same results.

Wet-chemical methods, which usually consist of the absorption of ozone in a potassium iodide solution and titration of the iodine released with sodium thiosulfate, were traditionally used in the rubber industry and were specified in national standards. They are not suitable for continuous operation or control and hence are less desirable in practice than instrumental methods. The results obtained have been shown to be sensitive to small variations in test procedures and the concentration and purity of reagents, and there has been much controversy over the stoichiometry of the reaction.

Electrochemical methods are widely used in the rubber industry and found to be convenient in continuously monitoring and controlling ozone. Chemiluminescence methods have also been used.

More recently, UV absorption analysers, which have the same monitoring and controlling ability, have been increasingly used. Most important, this technique has been adopted by all major environmental agencies as the standard and is regarded by them to be absolute.

Consequently, this standard UV absorption method is adopted as the reference technique against which all others are intended to be calibrated. Like any measurement instrument, the accuracy of any particular UV instrument is dependent on the calibration and maintenance of its components, and hence even UV analysers should be checked against acknowledged standard instruments. Studies are being undertaken in several countries to propose a primary-standard apparatus.

Although this document is concerned with ozone analysis, it also draws attention to the influence of atmospheric pressure on the rate of cracking of rubber at constant ozone concentrations as normally expressed in terms of parts by volume. As established by interlaboratory tests conducted in North America^[3], the variation in ozone resistance that can result between laboratories operating at significantly different atmospheric pressures can be corrected by specifying ozone concentration in terms of the partial pressure of ozone (see [Annex C](#)).

Attention is drawn to the highly toxic nature of ozone. Efforts should be made to minimize the exposure of workers at all times. In the absence of more stringent or contrary national safety regulations, it is recommended that 10 parts of ozone per hundred million parts of air of the surrounding atmosphere by volume be regarded as an absolute maximum concentration, while the maximum average concentration should be appreciably lower.

Unless a totally enclosed system is being used, an exhaust vent to remove ozone-laden air is recommended.

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Rubber, vulcanized or thermoplastic — Resistance to ozone cracking —

Part 3: Reference and alternative methods for determining the ozone concentration in laboratory test chambers

1 Scope

This document describes three types of method for the determination of ozone concentration in laboratory test chambers.

Method A — UV absorption: this is the reference method, and is used as the means of calibration for the alternative methods B and C.

Method B — Instrumental techniques:

B1: electrochemical

B2: chemiluminescence

Method C — Wet-chemical techniques:

Procedure I

Procedure II

Procedure III

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1431-1, *Rubber, vulcanized or thermoplastic — Resistance to ozone cracking — Part 1: Static and dynamic strain testing*

ISO 13964, *Air quality — Determination of ozone in ambient air — Ultraviolet photometric method*

3 Terms and definitions

No terms and definitions are listed in this document.

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

— IEC Electropedia: available at <http://www.electropedia.org/>

— ISO Online browsing platform: available at <http://www.iso.org/obp>

4 Principle

An ozone/air mixture is sampled from an ozone exposure chamber and the ozone concentration is determined by the UV absorption reference method or by alternative instrumental or chemical-analysis methods calibrated against the UV absorption method.

5 Apparatus

Apparatus used for the determination of the ozone concentration shall be one of the following types:

- UV absorption
- Electrochemical
- Chemiluminescence
- Wet-chemical

The reference method is UV absorption, and all equipment shall be calibrated against the UV absorption method as specified in [Clause 6](#).

The apparatus used for the UV absorption method shall be in accordance with ISO 13964, except that it shall be capable of measuring ozone concentrations specified in ISO 1431-1.

Descriptions of alternative methods are given in [Annex B](#) (instrumental methods) and [Annex C](#) (wet-chemical methods).

6 Calibration

Calibration of the apparatus for determining the ozone concentration shall be in accordance with the procedures given in ISO 13964.

7 Procedure

The UV method shall be carried out in accordance with ISO 13964.

Other instrumental methods shall be used in accordance with the manufacturer's instructions, attention being paid in particular to initial setting up, zero adjustment and maintaining and checking the instrument as mentioned in [Annex B](#).

Wet-chemical methods shall be carried out in accordance with [Annex C](#).

8 Expression of results

Generally, the ozone concentration φ_{O_3} is expressed in parts of ozone by volume per hundred million parts of air by volume (pphm).

However, the ozone concentration may also be expressed in mg/m³ or in mPa. The expression mg/m³ indicates the number of ozone molecules in the volume which is available for ozone cracking and depends on both pressure and temperature (see [Annex A](#)).

For conversion purposes, the following formula is valid:

$$\varphi_{O_3} \left[\text{mg/m}^3 \right] = 5,78 \times 10^{-3} \times \frac{p}{T} \times \varphi_{O_3} \left[\text{pphm} \right]$$

where

p is the atmospheric pressure, in hPa;

T is the temperature, in K.

In terms of the partial pressure of ozone:

$$p_{O_3} [\text{mPa}] = 10^{-3} p \phi_{O_3} [\text{pphm}]$$

where p , is the atmospheric pressure, in hPa.

At 1 013 hPa and 273 K, 1 pphm = 1,01 mPa.

9 Test report

The test report shall contain the following information:

- a) a reference to this document, i.e. ISO 1431-3;
- b) the method used, i.e. type of instrument or wet-chemical;
- c) the measurement interval if measurement was not continuous;
- d) the ozone concentration or range of concentrations measured, expressed in pphm or mg/m³ or mPa partial pressure of O₃, corrected if necessary by a calibration factor;
- e) the date of the test.

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