

Chemical tests for raw and vulcanized rubber —

**Part 22: Methods for determination of
bromine and chlorine content —**

**Section 22.2 Oxygen flask combustion
technique for determination of bromine
and chlorine**

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Plastics and Rubber Standards Policy Committee (PRM/-) to Technical Committee PRM/23, upon which the following bodies were represented:

- Institute of Water and Environmental Management
- Laboratory of the Government Chemist
- Ministry of Defence
- Royal Society of Chemistry
- Rubber and Plastics Research Association

This British Standard, having been prepared under the direction of the Plastics and Rubber Standards Policy Committee, was published under the authority of the Standards Board and comes into effect on 28 February 1992

© BSI 08-1999

The following BSI references relate to the work on this standard:
Committee reference PRM/23
Draft for comment 89/35456 DC

ISBN 0 580 20647 5

Amendments issued since publication

Amd. No.	Date	Comments

Contents

	Page
Committees responsible	Inside front cover
National foreword	ii
Introduction	1
1 Scope	1
2 Principle	1
3 Interference	1
4 Reagents and materials	1
5 Apparatus	2
6 Selection and preparation of intermediate test portion	3
7 Procedure	3
8 Expression of results	4
9 Test report	6
Annex A (informative) Bibliography	7
Figure 1 — Paper configurations for wrapping test portions for oxygen combustion	3
Figure 2 — Typical titration curve	5
Publication(s) referred to	Inside back cover

National foreword

This Section of BS 7164 has been prepared under the direction of the Plastics and Rubber Standards Policy Committee and is identical with ISO 7725:1991 “*Rubber and rubber products — Determination of bromine and chlorine content — Oxygen flask combustion technique*”, published by the International Organization for Standardization (ISO).

BS 7164 rationalizes all the methods that deal with chemical analysis of raw, compounded and vulcanized rubber, some of which have previously been published in BS 903, BS 1673 and BS 5923. Relevant Parts of those standards will be withdrawn or deleted by amendment, as appropriate, when superseded by Parts and Sections of BS 7164.

This Section of BS 7164 supersedes 3.4 of Part B12 of BS 903-B11 and BS 903-B12:1960 which is deleted by amendment.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 8, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

Introduction

This International Standard describes an oxygen flask combustion technique for the destruction of rubber, followed by either a potentiometric titration for chlorine and bromine if they occur singly or together, or a visual titration for bromine only or chlorine only.

Mutual interference, caused by co-precipitation, can be a problem in the potentiometric titration with silver nitrate. This is largely overcome though not completely eliminated by the addition of aluminium nitrate. The problem is least when bromine is in excess. At its worst, when chlorine is in excess, the bromine concentration may appear high, to an extent of 5 % of the chlorine content. The problem may be minimized by carrying out the titration at 60 °C, and keeping the rate of addition of titrant to a minimum.

During combustion of bromine-containing compounds, a small proportion of the bromine appears as bromate instead of bromide. Bromine results can therefore be low by about 1 % of the bromine content. This can be overcome in the case of the potentiometric finish only by treating the solution obtained on combustion with hydrazine sulfate.

The potentiometric titration (see 7.2.1) is sensitive down to 6 µg of bromine or 3 µg of chlorine when these are present singly.

The visual titration (see 7.2.2) lower limits are down to 0,2 mg of bromine or 0,1 mg of chlorine. When both chlorine and bromine are present, the lower limits are 60 µg of bromine and 30 µg of chlorine. These limits are dependent on the electrical sensitivities of the individual instruments used (see 5.3 to 5.5).

1 Scope

This International Standard specifies methods for the determination of chlorine and/or bromine present in raw rubber, uncured mixes or vulcanizates.

The methods are applicable to natural rubbers and to the following synthetic rubbers: isoprene, styrene-butadiene, butadiene, chlorinated, butyl, halogenated butyl, nitrile, ethylene-propylene, polychloroprene and epichlorohydrin.

2 Principle

The test portion is ignited in an atmosphere of oxygen in an oxygen combustion flask containing potassium hydroxide and hydrogen peroxide. The carbon and hydrogen of any organic matter are oxidized and halogen is converted to the potassium salt. These potassium salts are titrated potentiometrically with silver nitrate solution to determine the amount of chlorine and bromine, when they occur together in a rubber sample, or are titrated visually with mercuric nitrate solution to give either chlorine or bromine, when only one halogen is present.

3 Interference

3.1 Iodine will interfere but Zn^{2+} , S, CN^- and CO_3^{2-} do not.

3.2 Bromine and chlorine contained in additives/contaminants will be determined by this method, unless previously removed by extraction.

4 Reagents and materials

WARNING — All recognized health and safety precautions shall be in effect when carrying out the operations specified in this International Standard. Failure to heed the directions contained in this International Standard or those of the manufacturer of the devices used may result in explosions.

During the analysis, use only reagents of recognized analytical reagent grade and only distilled water or water of equivalent purity.

4.1 *Hydrogen peroxide*, 6 % (m/m) solution.

Dilute 20 cm³ of 30 % (m/m) aqueous hydrogen peroxide solution to 100 cm³ with water.

WARNING — 30 % (m/m) hydrogen peroxide is very corrosive to the skin. Wear rubber or plastic gloves and eye protection when handling.

4.2 *Potassium hydroxide*, solution, $c(\text{KOH}) = 0,5 \text{ mol/dm}^3$

Dissolve 2,8 g of potassium hydroxide in 100 cm³ of water.

4.3 *Nitric acid*, concentrated, $\rho = 1,42 \text{ Mg/m}^3$.

4.3.1 *Nitric acid*, dilute, $c(\text{HNO}_3) = 2 \text{ mol/dm}^3$.

Dilute approximately 120 cm³ of concentrated nitric acid (4.3) to 1 dm³ with water.

4.3.2 *Nitric acid*, dilute, $c(\text{HNO}_3) = 0,5 \text{ mol/dm}^3$.

Dilute 30 cm³ of concentrated nitric acid (4.3) to 1 dm³ with water.

4.3.3 *Nitric acid*, dilute, 0,2 % (V/V).