

Methods for

Measurement of air pollution —

**Part 10: Determination of the mass
concentration of sulphur dioxide in
ambient air: tetrachloromercurate
(TCM)/pararosaniline method**

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Environment and Pollution Standards Policy Committee (EPC/-) to Technical Committee EPC/35, upon which the following bodies were represented:

- British Coal
- British Gas
- British Steel Corporation
- Cement Makers' Federation
- Department of Health
- Department of Trade and Industry (Laboratory of the Government Chemist)
- Department of Trade and Industry (Warren Spring Laboratory)
- Electricity Supply Industry in England and Wales
- GAMBICA (BEAMA Ltd.)
- Health and Safety Executive
- Institute of Petroleum
- Institution of Environmental Health Officers
- Institution of Gas Engineers
- Lead Development Association
- National Industrial Fuel Efficiency Service
- National Society for Clean Air
- Society of Chemical Industry

The following bodies were also represented in the drafting of the standard, through subcommittees and panels:

- Asbestosis Research Council
- Chartered Institution of Building Services Engineers
- Council of Environmental Conservation
- Department of the Environment
- Department of the Environment (Building Research Establishment)
- Engineering Equipment and Materials Users' Association
- Fibre Cement Manufacturers' Association Limited
- Institute of Energy
- Institute of Occupational Hygienists
- Institute of Occupational Medicine
- Institution of Electrical Engineers
- London Regional Transport

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National foreword

This Part of BS 1747, which has been prepared under the direction of the Environment and Pollution Standards Policy Committee, is identical with ISO 6767:1990 “*Ambient air — Determination of the mass concentration of sulfur dioxide — Tetrachloromercurate (TCM)/pararosaniline method*” published by the International Organization for Standardization (ISO) with the active participation and approval of the UK.

This Part of BS 1747 is one of a series relating to air quality arising from UK participation in the work of ISO/TC 146. Methods for the determination of particular constituents of ambient air will be published as further Parts of this British Standard. Topics relating to air quality characteristics will be published as Parts of BS 6069 “*Characterization of air quality*”.

Cross-references

International Standards	Corresponding British Standards
ISO 4219:1979	BS 1747 <i>Methods for measurement of air pollution</i> Part 6:1983 <i>Sampling equipment used for the determination of gaseous sulphur compounds in ambient air</i> (Identical)
ISO 6349:1979	BS 4559 <i>Methods for preparation of calibration gas mixtures</i> Part 7:1981 <i>Permeation method</i> (Identical)

Typographical error. In clause 3, paragraph 4, line 1, delete the second “the”.
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 10, 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.

1 Scope

This International Standard specifies a spectrophotometric method, known as the tetrachloromercurate (TCM)/pararosaniline method, for the determination of the mass concentration of sulfur dioxide in ambient air within the range of $20 \mu\text{g}/\text{m}^3$ to about $500 \mu\text{g}/\text{m}^3$.

The sampling period is 30 min to 60 min.

If a longer sampling period than 60 min is used, or higher concentrations of sulfur dioxide (up to about $2\,000 \mu\text{g}/\text{m}^3$) are expected, care is necessary to ensure that the concentrations of sulfur dioxide in the absorption solution given in clause 6, paragraph 2 are not exceeded. This can be achieved by a reduction of the volume flow rate during sampling. Sample solutions obtained by this procedure may be stored for up to 24 h before making measurements, provided that they are kept in a refrigerator at about 5°C .

Substances which are known to interfere and which might be present in the air being sampled are listed in 7.5.

Indications of the precision and accuracy of the method, and of the lower detection limit are given in 8.2.

Detection limit, standard deviations and interferences qualify the TCM-method for orientating field measurements in the higher concentration range. When more accurate measurements are necessary, instruments which are specially tested and calibrated should be used.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 4219:1979, *Air quality — Determination of gaseous sulphur compounds in ambient air — Sampling equipment*.

ISO 6349:1979, *Gas analysis — Preparation of calibration gas mixtures — Permeation method*.

3 Principle

Absorption of sulfur dioxide present in the air sample by passage through a sodium tetrachloromercurate (TCM) solution within a specified period resulting in the formation of a dichlorosulfitomercurate complex.

Destruction of any nitrite ions formed in the sodium tetrachloromercurate solution, by nitrogen oxides present in the air sample, by adding sulfamic acid solution. Conversion of the dichlorosulfitomercurate complex into intensely violet coloured pararosaniline methyl sulfonic acid, by adding a formaldehyde solution and an acidified pararosaniline hydrochloride solution to the resultant solution.

Determination of the absorbance of the sample solution at a wavelength of about 550 nm using an appropriate spectrophotometer (or colorimeter) and calculation of the mass concentration of sulfur dioxide by means of a calibration graph prepared using calibration gas mixtures.

Depending on the the equipment available in the laboratory, it may be convenient to use sodium disulfite solutions for routine checks. However, the solutions are only used after proper calibration using a permeation device.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water, preferably double-distilled water, or water of equivalent purity, free from oxidants.

WARNING — Use the reagents in accordance with the appropriate health and safety regulations.

4.1 Sodium tetrachloromercurate (TCM), absorption solution, $c(\text{Na}_2[\text{HgCl}_4]) = 0,04 \text{ mol/l}$.

Dissolve 10,9 g of mercury(II) chloride (HgCl_2), 4,7 g of sodium chloride (NaCl) and 0,07 g of ethylenedinitrilo tetraacetic acid, disodium salt dihydrate $[(\text{HOCOCH}_2)_2\text{N}(\text{CH}_2)_2\text{N}(\text{CH}_2\text{COONa})_2 \cdot 2\text{H}_2\text{O}]$ EDTA in water in a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix well.

Store the solution in a well stoppered bottle.

The solution is stable for several months, but discard it if a precipitate is formed.

NOTE 1 A procedure for recovering mercury from fresh and used solutions is given in Annex C.