

Australian Standard[®]

Methods for sampling and analysis of ambient air

Method 5.1: Determination of oxides of nitrogen—Direct-reading instrumental method

AS 3580.5.1—2011

PREFACE

This Standard was prepared by the Australian members of Joint Standards Australia/Standards New Zealand Committee EV-007, Methods for Examination of Air, to supersede AS 3580.5.1—1993, *Methods for sampling and analysis of ambient air, Method 5.1: Determination of oxides of nitrogen—Chemiluminescence method*. After consultation with stakeholders in both countries, Standards Australia and Standards New Zealand decided to develop this Standard as an Australian Standard rather than as an Australian/New Zealand Standard.

This Standard has been developed as a performance-based standard to include chemiluminescence and other direct-reading instrumental methods.

The requirements for instruments found to be suitable for using this method are given in the United States Environmental Protection Agency (US EPA) Title 40, Part 53 of the Code of Federal Regulations (40 CFR Part 53)—*Ambient Air Monitoring Reference and Equivalent Methods, Subpart B—Procedures for Testing Performance Characteristics of Automated Methods*.

FOREWORD

Nitrogen oxides, which are produced by combustion and industrial processes, are emitted into the air predominantly as nitric oxide (NO) and, to a lesser extent, as nitrogen dioxide (NO₂). In the atmosphere, chemical reactions oxidize NO to produce NO₂, which may undergo further reaction to produce nitric acid, and various gaseous and particulate organic nitrates.

Oxides of nitrogen (NO_x), as determined by this method, is the sum of the NO and NO₂ concentrations in the air sample. It is usually found that where atmospheric nitrogen dioxide concentrations reach levels of concern to health, NO_x consists predominantly of NO and NO₂.

For chemiluminescent instruments employing catalytic converters, this method provides a procedure for the direct measurement of NO and the conversion of NO₂ to NO for direct measurement separately. The measurement of NO_x is the sum of the NO and converted NO₂ concentrations. In addition to NO₂, other gaseous nitrogenous species (e.g. peroxyacetyl nitrate (PAN), nitric acid, ammonia, organic amines, organic nitrates and nitrites), are also converted to NO by the catalytic converter and therefore are also detected. The efficiency of conversion to NO of the other species can be variable and non-quantitative however these typically comprise a very small proportion of the analyser response.

METHOD

1 SCOPE

This Standard sets out the method for the determination of oxides of nitrogen in ambient air using a direct-reading instrumental method. This method applies to the determination of oxides of nitrogen in ambient air where the concentration typically lies within the range 0 to 5 p.p.m. by volume (equivalent to approximately 0 to 6700 $\mu\text{g}/\text{m}^3$ of nitric oxide).

2 NORMATIVE REFERENCES

The following are the normative documents referenced in this Standard:

NOTE: Documents referenced for informative purposes are listed in the Bibliography.

AS/NZS

- 3580 Methods for sampling and analysis of ambient air
- 3580.1.1 Method 1.1 Guide to siting air monitoring equipment
- 3580.2.2 Method 2.2: Preparation of reference test atmospheres—Compressed gas method

ISO

Guide to the expression of uncertainty in measurement (ISO GUM)

US EPA

40 CFR Part 53 US Code of Federal Regulations, Title 40—Protection of Environment, Part 53—Ambient Air Monitoring Reference and Equivalent Methods

3 DEFINITIONS

For the purpose of this Standard, the definitions below apply.

3.1 Fall time

The time interval, after a step decrease in input concentration, between initial instrument response and 90% of final instrument response.

3.2 Full scale (FS)

The nominated maximum concentration for which an instrument has been calibrated. The full scale is selected to cover the normal range of values expected in the sampling environment.

3.3 Interference equivalent

Positive or negative instrument response caused by a substance other than the one being measured.

3.4 Lag time

The time interval between a step change in input concentration and the first observable corresponding change in instrument response.

3.5 Level 1 calibration

A simplified, two-point analyser calibration used when analyser linearity does not need to be checked or verified.

3.6 Level 2 check

A simple check of an analyser's response. These checks may be performed using non-certified test atmospheres (e.g. permeation tubes, expired gas cylinders).

3.7 Linearity

The deviation of an analyser's output from a linear best fit line when subjected to varying reference test atmospheres.

3.8 Lower detectable limit

The minimum pollutant concentration that produces a signal of exactly twice the noise level.

3.9 Noise

Spontaneous, short duration deviations in instrument output, about the mean output, which are not caused by input concentration changes. Noise level is determined as the standard deviation about the mean and is expressed in concentration units.

3.10 Oxides of nitrogen (NO_x)

The sum of the nitric oxide (NO) and nitrogen dioxide (NO₂) concentrations in the air sample.

3.11 Parameter

One of the characteristics related to an air sample, e.g. concentration of constituent or other quantifiable property (e.g. wind speed, temperature).

3.12 Parts per billion (p.p.b.)

A ratio expressing the volume of gaseous pollutant contained in 1 000 000 000 volumes of atmosphere. It may be expressed in terms of microlitres per cubic metre since the values are identical. Alternatively, it is one thousand million times the ratio of the partial pressure of gaseous pollutant to the pressure of the atmosphere in which it is contained.

3.13 Parts per million (p.p.m.)

A ratio expressing the volume of gaseous pollutant contained in 1 000 000 volumes of atmosphere. It may be expressed in terms of millilitres per cubic metre since the values are identical. Alternatively, it is one million times the ratio of the partial pressure of gaseous pollutant to the pressure of the atmosphere in which it is contained.

3.14 Precision

Variation about the mean of repeated measurements of the same pollutant concentration on the same instrument, expressed as one standard deviation about the mean.

3.15 Proficiency audit

An evaluation processes used to measure the performance or effectiveness of a measurement system including analysis and validation.

3.16 Range

Nominal minimum and maximum concentrations that a method is capable of measuring.

3.17 Reference test atmosphere

A test atmosphere containing a known concentration of pollutant.

3.18 Rise time

The time interval, after a step increase in input concentration, between initial instrument response and 90% of final instrument response.

3.19 Span drift

The percentage change in instrument response to an on-scale pollutant concentration over a period of continuous unadjusted operation.

3.20 U_{95}

A measurement of uncertainty at a confidence interval of 95% according to ISO GUM.

3.21 Zero drift

The change in instrument response to zero pollutant concentration over a period of continuous unadjusted operation.

4 PRINCIPLE

A sample of the ambient air is presented to an instrument, in which the response of the detector is recorded as a concentration.

5 APPARATUS

5.1 Instrument

A continuous direct-reading instrument is required, which shall meet or exceed those specifications given in Table 1. The instrument may be used over any range within the limits of the Standard but shall be calibrated in accordance with Clause 7. The manufacturer's published performance specifications shall be deemed as acceptable evidence of conformance to the given requirements providing that they have been carried out in accordance with the procedures based on the US EPA equivalency testing as stated in the US Code of Federal Regulations, Title 40: *Protection of Environment, Part 53—Ambient Air Monitoring Reference and Equivalent Methods*. Direct-reading instruments and methods are constantly evolving and for this reason, this Standard does not specify measurement by chemiluminescence methods alone. Providing the instrument performance, as determined above, is within the specifications given in Table 1, alternate methods may be used within the context of this Standard.

TABLE 1
INSTRUMENT PERFORMANCE SPECIFICATIONS

Parameter	Requirements
Range	0–5 p.p.m.
Noise	1 p.p.b. or 0.1% FS*
Lower detectable limit	2 p.p.b. or 0.2% FS*
Converter efficiency [#]	>95%
Interference equivalent	
Each interferent	±20 p.p.b.
Total interferent	±40 p.p.b.
Response times	
Rise (90%)	≤ 60 s
Fall (90%)	≤ 60 s
Lag	≤ 30 s
Zero drift	
24 hours	1 p.p.b.
7 days	2 p.p.b.
Span drift	
24 hours	1% FS*
7 days	1% FS*
Precision	1% FS*
Linearity	1% FS*
Operating temperature	20–30°C
Humidity	10–80%

* FS = full-scale deflection

If a NO₂ to NO converter is used.

5.2 Zero air

Zero air shall be free from contaminants likely to cause a detectable response on the test analyser. The concentration of oxygen in the zero air shall be within ±2% v/v of the normal composition of air (20.9% v/v).

5.3 Reference test atmosphere

Reference test atmospheres of nitric oxide shall be produced in accordance with AS 3580.2.2 using zero air as a diluent gas.

5.4 Level 1 span and zero calibrations

Level 1 span and zero calibrations shall be performed using reference test atmospheres produced in accordance with AS 3580.2.2 using zero air as a diluent gas. Adjustments to the analyser gain or zero shall only be made when performing Level 1 calibrations.

5.5 Level 1 span and zero checks

Level 1 span and zero checks shall be performed using reference test atmospheres produced in accordance with AS 3580.2.2 using zero air as a diluent gas. These checks are often performed automatically and are used to check the analyser response to a known test atmosphere. Adjustments to the analyser gain or zero shall not be made when performing checks.

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