SLOVENSKI STANDARD
SIST ISO 15337:2013

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Zunanji zrak - Titracija plinske faze - Umerjanje analizatorjev ozona

Ambient air - Gas phase titration - Calibration of analysers for ozone

Air ambiant - Titrage en phase gazeuse - Étalonnage des analyseurs d'ozone

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Ambient air — Gas phase titration — Calibration of analysers for ozone

Air ambiant — Titrage en phase gazeuse — Étalonnage des analyseurs d'ozone

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Foreword

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ISO 15337 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 3, *Ambient atmospheres*.

Introduction

For ambient ozone (O_3) analysers, the primary standard measurement principle for calibration is UV photometry. This International Standard provides an alternative secondary measurement principle and method based on gas phase titration of an O_3 gas mixture with excess nitric oxide (NO). When using this method, the generated O_3 calibration gases are traceable to a certified primary NO measurement standard.

Ambient air — Gas phase titration — Calibration of analysers for ozone

1 Scope

This International Standard specifies the gas phase titration (GPT) method for the calibration of ambient air ozone (O_3) analysers. The method is applicable to the calibration of O_3 concentrations in the range 10 µg m^{-3} (5 nmol mol^{-1} mole fraction) to 2 000 µg m^{-3} (1 000 nmol mol^{-1} mole fraction). This International Standard uses the reference conditions of 25 °C and 101,325 kPa; however, reference temperatures of 0 °C and 20 °C are also acceptable.

2 Normative references

The following referenced documents are indispensable for the application 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 7996, *Ambient air — Determination of the mass concentration of nitrogen oxides — Chemiluminescence method*

3 Principle

Gas phase titration (GPT) is based on the simple gas phase bimolecular reaction:

$$NO + O_3 \rightleftharpoons NO_2 + O_2 \tag{1}$$

with a bimolecular reaction rate constant of $1{,}8 \times 10^{-14}$ cm^3 $molecule^{-1}$ s^{-1} (or 0,44 µmol mol^{-1} s^{-1} when concentrations are expressed as mole fractions) at 298 K.

This reaction is fast, and equilibrium lies far to the right hand side of Expression (1) if the kinetic conditions specified in Annex A are satisfied. This International Standard is based on mixing O_3 with excess nitric oxide (NO) in a dynamic flow system where, first, the NO and O_3 are mixed at relatively high concentrations (to effect essentially complete reaction of the O_3) and, second, the reaction products and any excess NO are then diluted with zero reference gas (e.g. synthetic air) to final calibration concentrations. Since the reaction is stoichiometric, the molar decrease in measured NO is equal to the added O_3; this is also equal to the nitrogen dioxide (NO_2) reaction product.

The NO is obtained from a certified NO measurement standard, a calibrated gas mixture in a compressed gas cylinder. Other calibrated sources could be used (e.g. see VDI 2453-3 [11]), and adapted to the GPT calibration apparatus. The change in NO concentration at the GPT output manifold is measured by a chemiluminescence NO analyser. A stable O_3 generator is used to produce variable concentrations of the gas to cover the calibration range of interest.

4 Reagents and materials

4.1 Sample line and connectors, made of material that is inert to O_3 and NO, such as glass or fluorocarbon polymer [e.g. perfluoro(alkoxy alkane) (PFA), polytetrafluoroethylene (PTFE) or perfluoro (ethylene-propylene) plastic (FEP) are acceptable]; these shall be as short as possible to keep the residence time to a minimum.

NOTE Whenever a sampling line is cleaned or replaced, it can take several hours to equilibrate with ambient conditions.

4.2 Zero reference gas, for calibration of the GPT procedure. If synthetic air is used, the oxygen (O_2) content shall be at the normal atmospheric concentration of 20,9 % ± 2 % volume fraction. No O_3, nitrogen oxides or any other interfering substance that can cause an undesired measurable positive or negative response in the analysis shall be detectable in the zero air.

NOTE Details on a system for making zero air from ambient air can be found in ASTM D5011 [9].

4.3 NO measurement standard, stored in a compressed gas cylinder and containing a known concentration of NO in nitrogen, in the range 10 μmol mol^{-1} to 100 μmol mol^{-1}, for use in the calibration procedure. This NO cylinder shall be traceable to a primary measurement standard (e.g. a certified reference material), and the NO_2 impurity shall be less than 0,5 % mass fraction of the NO concentration.

5 Apparatus

Usual laboratory equipment and, in particular, the following.

5.1 Ozone generator, capable of producing steady O_3 concentrations in the required range throughout the period of the calibration. Conventional UV low-pressure mercury vapour lamps are adequate for this purpose; however, both voltage and temperature regulation shall be provided for a stable O_3 output.

CAUTION — Ozone is a toxic gas and good laboratory practice should limit indoor ozone concentrations to less than 200 µg m^{-3} (100 nmol mol^{-1}). Consult a reference text for more details on hazards of ozone and appropriate safety precautions. Any excess should be vented into an activated charcoal scrubber (with negligible back-pressure) or outdoors well away from any sampling intake. Comply with any local regulations currently in force for handling, use and disposal of ozone.

5.2 Gas flow controllers and meters: there are two options for controlling and measuring the gas flows; see 5.2.1 and 5.2.2. Electronic mass flow controllers (5.2.1) are recommended because of their inherent low measurement uncertainty and greater precision.

5.2.1 Electronic mass flow controllers, calibrated and capable of maintaining constant gas flow rates within ± 0,5 % throughout the calibration period. Components in contact with NO shall be of a non-reactive material.

5.2.2 Manual gas flow control and meters, capable of maintaining constant gas flow rates within ± 2 % throughout the calibration period. The gas flow meters shall be capable of measuring the required gas flows within ± 2 %.

5.3 Reaction chamber, to provide a suitable environment for the quantitative reaction between NO and O_3 at high concentration. This chamber shall be made of materials inert to O_3 and nitrogen oxides, such as borosilicate glass, PFA, FEP or PTFE. Its volume shall be limited so that the residence time of the gas mixture in this volume is less than 60 s (see Annex A for predetermining the volume for given flow conditions).

5.4 Dilution chamber, to provide a suitable environment for the mixing of reaction products and dilution air. It shall be made of materials inert to O_3 and nitrogen oxides, such as borosilicate glass, PFA, FEP or PTFE. Its volume should be sufficiently large to allow complete mixing of the gas components, but small enough to limit the residence time to less than 60 s.

5.5 Output manifold, to serve as a multi-port interface to allow sampling of the output from the GPT calibration system. It shall be made of materials inert to O_3, such as borosilicate glass, PFA, FEP or PTFE. It shall be of sufficient diameter and be vented to ensure an insignificant pressure drop from inside to outside the manifold. The vent outlet shall be located downstream of the other outlet ports so as to prevent intrusion of ambient air.

5.6 Temperature sensor, to measure the temperature of the detection cell of the O_3 analyser, readable to within ± 0,5 °C.

5.7 Pressure meter, to indicate the pressure in the detection cell of the O_3 analyser, readable to within ± 2 hPa.

5.8 Chemiluminescence nitrogen oxides analyser, whose NO channel meets the requirements of ISO 7996. The purpose of this instrument is to determine quantitatively the decrease in NO response equivalent to the O_3 added in the GPT system.

NOTE Additional details on calibrating such an analyser can be found in VDI 2453-2 [10].

5.9 Pressure regulator for source gas cylinder, whose internal components are inert to NO.

6 Calibration procedure

6.1 Overview

A schematic diagram of a calibration system is shown in Figure 1. The following procedure is written for the option where

a) both NO and O_3 analysers are simultaneously sampling the output manifold of the GPT system,

b) concentrations are expressed as mole fractions, in nanomoles per mole.

A suitable and accurately known NO concentration in air is generated and measured with the chemiluminescence NO analyser (5.8). Then, O_3 is generated to titrate some of the NO. The decrease in NO is equal to the added O_3. Finally, with the O_3 generator (5.1) still on, the NO flow is turned off so that the ambient O_3 analyser can measure the known O_3 concentration via the common manifold. By varying the O_3 generator output, other known O_3 concentrations can be generated in a similar manner. A linear least squares analysis of the O_3 analyser responses and corresponding calculated O_3 concentrations will produce a linear calibration function for the O_3 analyser.

The linearity of the chemiluminescence NO analyser shall have been recently verified by means of a linear least squares analysis on its calibration data, and the calculated correlation coefficient shall be better than 0,99 for the NO calibration range of interest. (It should be noted that the calibration function for the NO analyser need not be used in the following calibration calculations.)

6.2 Calibration of the ambient ozone analyser

6.2.1 Install the instruments in a suitable location and provide temperature control of the measurement room to minimize any temperature dependence of the instruments. Follow the manufacturer's operating instructions for the analysers to set the various operating parameters correctly, including the sample flow rate and, if applicable, activation of the electronic temperature and pressure compensation on the O_3 analyser. Also, follow the diagnostic procedure as outlined in the manufacturer's operations manual to verify that instrument functions are within their performance specifications. The measured concentrations should be recorded by means of a suitable recording device (e.g. chart recorder or electronic data acquisition system).