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REFERENCE GASES—PREPARATION OF GRAVIMETRIC STANDARDS

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Australian Environment Council

Australian Gas Association

Confederation of Australian Industry

CSIRO, Division of Fossil Fuels

Department of Environment and Planning, S.A.

Department of Industrial Relations and Employment, N.S.W.

Environment Protection Authority of Victoria

Federal Chamber of Automotive Industries

National Association of Testing Authorities, Australia

Royal Australian Chemical Institute

State Pollution Control Commission, N.S.W.

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REFERENCE GASES—PREPARATION OF GRAVIMETRIC STANDARDS

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PREFACE

This Standard was prepared by the Association's Committee on Reference Gases, under the direction of the Chemical Standards Board, to standardize a gravimetric procedure for the preparation of reference gases.

This Standard method is based on ISO 6142, *Gas analysis—Preparation of calibration gas mixtures—Weighing methods.*

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STANDARD ASSOCIATION OF AUSTRALIA

Australian Standard

REFERENCE GASES—PREPARATION OF GRAVIMETRIC STANDARDS

1 SCOPE. This Standard sets out a method for the preparation of gravimetric standards for which the accuracy of each gas component is better than 0.5 percent relative or 0.02 percent absolute on a mole to mole basis, whichever is smaller and whatever the values of the selected concentrations.

NOTE: Appendix C sets out information which should be provided when ordering reference gases to be supplied to this Standard.

2 APPLICATION. The method is applicable only to gaseous components which do not react with each other or with the cylinder walls, and to condensable components which remain totally vaporized in the cylinder at all pressures and temperatures anticipated during the use of the contents.

NOTE: Retrograde condensation may occur with some hydrocarbon gas mixtures.

3 REFERENCED DOCUMENTS. The following Standards are referred to in this Standard:

AS

1777 Aluminium cylinders for compressed gases—Seamless—0.1 kg to 130 kg

2030 SAA Gas Cylinders Code

2473 Valves for compressed gas cylinders (threaded outlet)

B114 Alloy steel cylinders for the storage and transport of permanent gases and high pressure liquefiable gases

4 PRINCIPLE. The compositions of gas mixtures are determined from the defined units of mass and molecular mass. Using an evacuated cylinder of known mass, standard mixtures are prepared by adding and weighing each gas. The mass of each gas is found by difference, and the number of moles of each component is calculated. The mole fraction is calculated from the total number of moles.

To avoid having to weigh very small masses of gas, a lower mass limit is fixed for each of the components in the final gas mixture. Where the mass required to produce the desired concentration is less than this limiting value, a known quantity of gas mixture previously prepared in accordance with this Standard is diluted with a known mass of a given gas.

5 APPARATUS. Materials used for surfaces with which the gases may come into contact shall not promote changes in chemical composition of the gases. All devices (regulators, valves, filling connections) shall be leak tight.

5.1 Balance. With a minimum ratio of capacity to resolution (R) of 10^5 and minimum weighing accuracy of $\pm R$. The balance shall be calibrated with balance weights at least equivalent to Class F_1 (Ref. 1.).

5.2 Balance weights. At least equivalent to Class F_1 (Ref. 1.).

5.3 Barometer. With a reading resolution of 10 Pa (1 mm Hg is satisfactory) and calibrated against current meteorological data.

5.4 Gas cylinders. Complying with either AS 1777, AS 2030 or AS B114, as appropriate.

5.5 Valves. Manufactured in accordance with AS 2473.

5.6 Wet and dry bulb thermometer. Accurate to at least 0.1 °C.

5.7 Vacuum pump. Capable of maintaining a vacuum of less than 100 Pa.

5.8 Manifold. A suitable arrangement to allow interconnection of the gas cylinders, vacuum pump, vents and gauges.

6 GASES. All component gases used in primary preparation (before dilution) shall be subject to strict quality control and the purity shall be accurately known to at least ±0.1 percent absolute.

7 PROCEDURE.

7.1 Preparation of cylinder. The cylinder shall be prepared as follows:

(a) The external surface of the cylinder shall be free from dust, moisture, grease and particles. Cylinders suspected of being internally contaminated shall not be used.

(b) Fill the cylinder to full pressure and check for leaks.

(c) Evacuate the cylinder to at least 100 Pa.

(d) Fill with major component gas to a pressure greater than 200 kPa.

(e) Vent the cylinder.

(f) Repeat Steps (c), (d) and (e).

(g) Evacuate the cylinder to less than 100 Pa.

7.2 Weighing the cylinder. The weighing procedure shall be as follows:

(a) Check the mass of the cylinder on a rough scale.

(b) Zero the balance.

(c) Select the balance weight (5.2) corresponding to the mass of the cylinder obtained in Step (a).

(d) Carefully place the cylinder on the centre of the pan and release the beam. Record the mass of the cylinder.

(e) Remove the cylinder and check the balance zero. This should agree with the zero in Step (b) within the resolution of the balance; if not, re-zero the balance.

(f) Repeat Step (d).