

Australian Standard[®]

Alumina

**Part 4: Determination of specific
surface area by nitrogen
adsorption**

This Australian Standard was prepared by Committee MN/9, Alumina and Materials used in Aluminium Production. It was approved on behalf of the Council of Standards Australia on 24 January 1991 and published on 15 April 1991.

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Aluminium Development Council
Australian Mining Industry Council
Royal Australian Chemical Institute

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PREFACE

This Standard was prepared by the Standards Australia Committee on Alumina and Materials used in Aluminium Production, under the direction of the Minerals Standards Board, as one of a series of Standards for the testing of alumina,

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FOREWORD

In the development of this method, the temperature for degassing was chosen after testing various smelting-grade alumina. It was found that temperatures above 150°C changed the state of some samples with respect to the surface area, presumably through the partial calcination of the hydrated alumina, which is present to some degree in smelting-grade alumina. It was also found that there were significant, but repeatable, differences between the results obtained from analysers from different manufacturers. Hence, the method utilizes a standard reference material to normalize these differences.

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STANDARDS AUSTRALIA

Australian Standard

Alumina

Part 4: Determination of specific surface area by nitrogen adsorption

1 SCOPE This Standard sets out a single-point instrumental method for the determination of specific surface area (SSA) of smelter-grade alumina by nitrogen adsorption. The method is applicable to alumina having a surface area between 25 m²/g and 100 m²/g.

2 REFERENCED DOCUMENT The following document is referred to in this Standard:

AS

2850 Chemical analysis—Interlaboratory test programs—For determining precision of analytical method(s)—Guide to the planning and conduct

3 PRINCIPLE The method is based on the ability of the alumina to adsorb nitrogen molecules on its surface. The test sample is degassed for 1 h at 150°C, and then cooled by immersion in a bath of liquid nitrogen. Specific surface area is determined using either the static or the dynamic procedure. In the static procedure, nitrogen is adsorbed by the sample, and the volume or pressure difference in the adsorption cell is measured. In the dynamic procedure, the quantity of nitrogen adsorbed is measured by sensing the change in thermal conductivity of a flowing mixture of nitrogen and an inert carrier gas, usually helium. Surface area measurements are based on the Brunauer-Emmett-Teller (BET) principle. Results from the sequential analysis of a carbon certified reference material are used to normalize test results.

4 REAGENTS**4.1 Liquid nitrogen**

4.2 Nitrogen gas—high-purity.

4.3 Helium gas—high-purity. (Required only where a dynamic instrument with gas-mixing ability is chosen for use.)

4.4 Nitrogen-helium gas mixture—high-purity mixture containing approximately 30 mole percent nitrogen in helium (if required for the specific dynamic instrument of choice).

4.5 Reference material—NPL Surface Area Standard, Vulcan 3-G (2700) graphitized carbon. The certified value of this material is 71.3 ± 2.7 m²/g at 300°C using a degassing time of 1 h.

NOTE: The reference material is available from National Physical Laboratories (NPL), United Kingdom.

5 APPARATUS

5.1 Specific surface analyser—a specific surface analyser employing low temperature (−196°C) nitrogen adsorption. The method for adsorption may be dynamic or static. The accuracy of the temperature measurement system on the instrument shall be validated and shown to be within $\pm 10^\circ\text{C}$ of the stated temperature.

5.2 Degassing equipment—suitable for the type of analyser being used.

5.3 Balance—capable of weighing to an accuracy of ± 0.001 g.

6 SAMPLING AND SAMPLE PREPARATION A 50 g test sample is prepared from the laboratory sample using a riffle or a rotary divider, taking particular care to avoid loss of fine particles through dusting. A representative test portion of optimum mass (depending on the requirements of the instrument and anticipated surface area) shall be taken from the test sample.

7 PROCEDURE

7.1 Determination of specific surface area of reference material The surface area of the reference material shall be determined with every batch of test samples, as follows:

- Weigh the required mass of reference material (4.5) to the nearest 0.001 g.
- Insert the reference material into the adsorption cell and degas in accordance with the manufacturer's instructions, at 300°C for 1 h.
- Determine the surface area of the reference material immediately after degassing.
- Using the procedure recommended by the instrument manufacturer, and the mass of sample dried at the degassing temperature, obtain and record the specific surface area, expressed to one decimal place.