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BUREAU OF INDIAN STANDARDS

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भारतीय मानक **मसौदा**

गैस्केट तथा पैकिंग — विस्तारित ग्रेफाइट का ऑक्सीकरण — परीक्षण पद्धतियाँ

DRAFT *Indian Standard*

Gaskets and Packings — Oxidation of Expanded Graphite — Methods of Test

ICS 21.140

Gasket and Packing Sectional Committee, MED 30	**Last date for receipt of comments: 20 Feb 2025**

FOREWORD
(*Formal clause will be added later*)

Flexible graphite is often chosen as the facing material in several different styles of metal reinforced gasket materials for its high temperature stability. It has been used in the sealing industry as an excellent alternative to asbestos fiber especially in steam or high temperature applications. The maximum temperature in which a flexible graphite faced gasket can be used is limited by the oxidation rate of the flexible graphite. The flexible graphite weight loss due to oxidation over time will result in total gasket mass loss, flange load loss and eventually to leakage.

The oxidation rate of the flexible graphite is highly dependent on the temperature, and the amount of oxygen in contact with the graphite. Other factors include the purity of the graphite, presence of oxidation accelerating contaminants, presence of oxidation inhibiting elements, whether the oxygen is flowing or stagnant around the graphite, and the surface area exposed.

Determining the effect of all these factors and therefore the life expectancy of a gasket can be a difficult challenge especially when the flexible graphite is in different gasket configurations, in a flange, and subject to the varying temperatures and chemicals of the process.

As difficult as it is to predetermine a flexible graphite gasket's life in service, it is relatively easy to rank the performance of different flexible graphite by a short-term oxidation test. This test cannot be used to determine a specific gasket's life in a specific application but will give a relative oxidation resistance ranking of different flexible graphite. A good quality oxidation inhibited grade of flexible graphite should have an oxidation rate of less than 4 percent when tested by Method A of this standard.

Typical oxidation rates should be less than 4 percent/h. A good quality non-inhibited grade of flexible graphite will have an oxidation rate of less than 15 percent/h when tested by Method A of this standard. Typical results will be in the (10 to 12) percent range.

Any flexible graphite with an oxidation rate of over 15 percent/h as tested by Method A, has probably been contaminated with impurities that increase the normal rate of graphite oxidation and are not recommended for high temperature gasket applications.

Assistance has been drawn from the following International Standards:

EN 14772:2021	Flanges and their joints — Quality assurance inspection and testing of gaskets in accordance with the series of standards EN 1514 and EN 12560
DIN 28090-2: 2014	Static gaskets for flange connections — Gaskets made from sheets Part 2: Special test procedures for quality assurance

In reporting the result of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'.

DRAFT *Indian Standard*

GASKETS AND PACKINGS — OXIDATION OF EXPANDED GRAPHITE — METHODS OF TEST

1 SCOPE

1.1 This standard covers the method of test for determination of oxidation percent of expanded graphite.

1.2 The standard covers two methods for evaluating expanded graphite materials in high-temperature environments: Method A and Method B, as described below. Either method can be used for evaluation.

1.2.1 *Method A* — It is a general screening method and a preliminary step towards Method B. Small degree of inaccuracies are acceptable.

1.2.2 *Method B* — It is a more accurate and repeatable method compared to Method A. The test requires sophisticated equipment for testing.

2 TERMS AND DEFINITIONS

For the purpose of this standard the following definitions shall apply.

2.1 Graphite — A grey crystalline allotropic form of carbon which occurs as a mineral in some rocks and can be made from coke. It used for nuclear, sealing, lubricant and various applications.

2.2 Oxidation — Oxidation is the loss of electrons during a reaction by a molecule, atom or ion. Oxidation occurs when the oxidation state of a molecule, atom or ion is increased.

2.3 Thermal Gravimetric Analysis (**TGA**) — Thermogravimetric analysis or thermal gravimetric analysis (TGA) is a method of thermal analysis in which the mass of a sample is measured over time as the temperature changes.

3 METHOD A — GENERAL SCREENING

3.1 General

Method A utilizes a muffle oven. Due to contaminants and residuals that can contaminate the oven from sample to sample and to temperature and gas flow variation within the oven, the user must understand that the method is intended for the determination of gross differences in material comparisons and must accept a small degree of inaccuracy.

The usage of simpler equipment allows this test to be easily carried out without requirement of any sophisticated machine or process. At elevated temperatures, the flexible graphite that has the least weight loss (lowest percent oxidation) in this test would be expected to have the longest gasket life.

3.2 Purpose

This test is a short-term general screening method utilizing a ventilated muffle oven to determine flexible graphite oxidation weight loss.

3.3 Equipment

3.3.1 Muffle furnace or oven capable of handling temperature up to 670 °C and having following requirements:

a) Minimum furnace internal dimension : 250 mm × 300 mm × 230 mm;
b) Furnace temperature stability : ± 5 °C; and
c) Ventilated, without forced air supply.

3.3.2 Stainless steel screen with ½ inch mesh or suitable plate/holder for the test. The plate holder must have minimum ½ inch openings.

3.3.3 2 inch × 6 inch steel rule die or any suitable cutting method.

3.3.4 Analytical balance, precision 0.000 1 g.

3.3.5 Timer.

NOTE — All equipment mentioned above must be calibrate where applicable.

4 TEST PROCEDURE

The test procedure for Method A shall be as follows:

a) Cut and identify two samples of flexible graphite sheet 2 inch × 6 inch;
b) Weigh and record the weight of each sample to the nearest 0.000 1 g. Identify this weight as the original weight;
c) Place each sample on the screen in a muffle furnace at 670 °C (+ 5 °C or - 5 °C) for 240 min (+ 1 min or - 1 min). The graphite foil sample must be placed at least 25 mm from the inner surface of the furnace and from each other in case of measuring several samples at the same time. Remove the samples from the muffle furnace and cool in a desiccator until they reach room temperature;
d) Weigh each sample again and record the weight. Identify this weight as final weight;
e) Calculate the percent oxidation rate = $\frac{\{(original\ weight)-(final\ weight)\}}{original\ weight \times 4} \times 100$;
f) Average the results of the two samples and report as the percent per hour oxidation rate; and

g) Report any variation form the recommended test procedure.

5 METHOD B — THERMAL GRAVIMETRIC ANALYSIS

5.1 General

Method B requires the use of a thermal gravimetric analysis (TGA) analyzer, which more accurately monitors the test temperature and environment and electronically graphs the effects of extreme temperature on graphitic materials. Some styles of TGA analyzers can test up to 19 samples at the same time so accurate side-by-side comparisons can be conducted.

5.2 Purpose

5.2.1 This TGA test method measures the weight changes of flexible graphite samples during heating under controlled atmospheres (capable of air, oxygen, and nitrogen). This TGA test method is used to compare the oxidation rate of different flexible graphite grades.

5.2.2 Under the controlled environment, the TGA tester determines the weight change of samples of flexible graphite materials at the controlled temperature according to the selected analysis method. The flexible graphite samples should have the same density and thickness (where possible).

5.3 Equipment

5.3.1 A thermal gravimetric analysis (TGA) tester.

5.3.2 Die cutting tools.

5.3.3 Crucibles.

6 OPERATION PROCEDURE

6.1 Sample Preparation — Cut a circular sample by die cutting tools, then place the sample into a crucible according to Table 1. Take care that gloves and a tweezers are used when taking the sample. Furthermore, where there is any printing on the sample, remove it or use areas where there is not any printing.

Table 1 Sample Preparation
(*Clauses* 6.1 and 6.1.3)

Sl No.	**Parameter**	**TGA One sample**	**TGA Multiple Sample**
(1)	(2)	(3)	(4)
i)	Sample dimensions and weight	Ex. Ø – 5 mm Weight – according foil thickness	Ex. Ø – 5 mm

			Weight – multiple sample up to 1 g total weight
ii)	Balance precision	0.001 mg	0.1 mg
iii)	Heating rate	Up to 40 °C/min	Up to 40 °C/min
iv)	Test atmosphere	Air or mixture of 80 % nitrogen and 20 % oxygen	Air
v)	Air purging rate	20 ml/min	15 l/min

6.1.1 Follow TGA tester operating procedures as per lab or manual.

6.1.2 Before testing, carry out a buoyancy correction.

6.1.3 Select gas supply — Air at a flow rate according Table 1.

6.1.4 Select temperature rate of rise — 40 °C per minute.

6.1.5 Select testing hold temperature — 670 °C

6.1.6 Select hold time — 4 h.

6.1.7 Load samples into TGA tester.

6.1.8 Run tester program and record total weight loss, then convert the value into percent/h (percent total loss/h).

6.1.9 Average the results from at least the two replicate samples and report percent per hour oxidation rate (*see* Fig. 1).

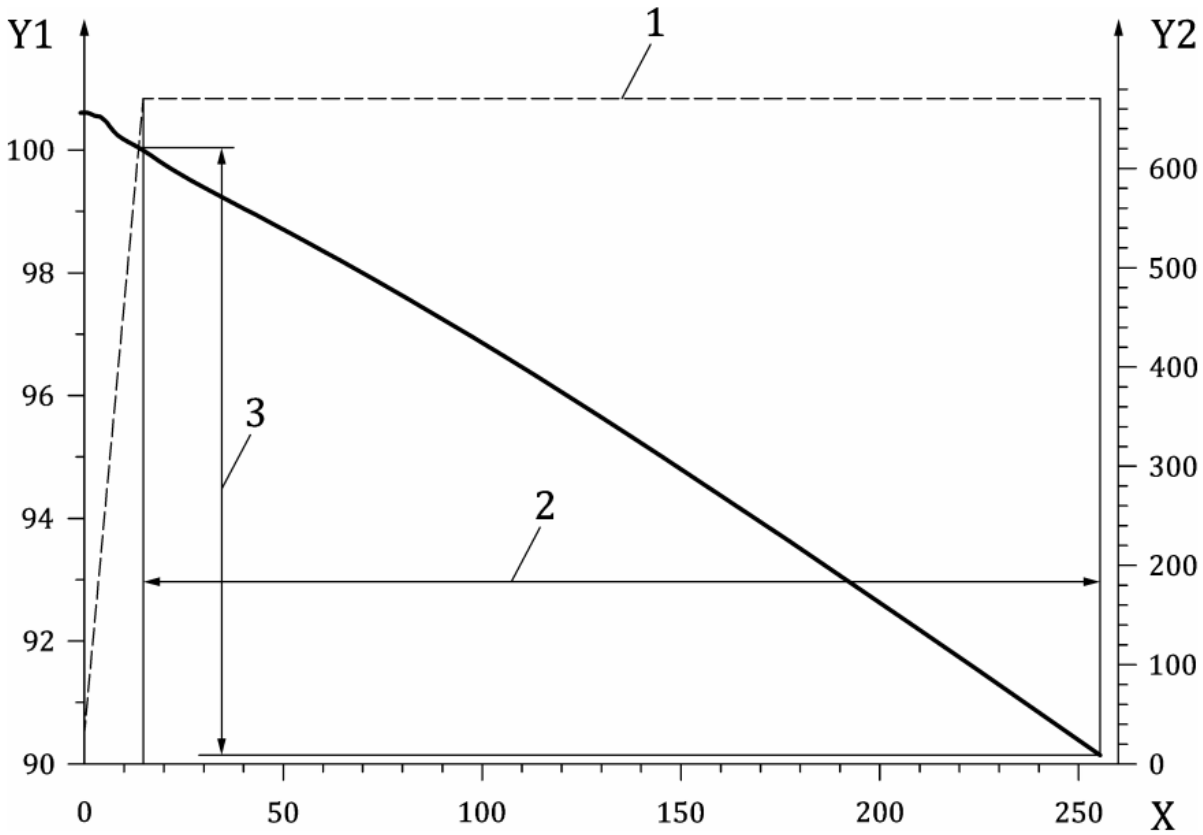

FIG. 1 EXAMPLE FOR EVALUATION. (X – TIME [MIN]; Y1 – RESIDUAL MASS [%]; Y2 – TEMPERATURE [°C]; 1 – COURSE OF TEMPERATURE [°C]; 2 – MEASURING TIME 240 MIN; 3 – TOTAL MASS LOSS: 9.88 %, MASS LOSS/H: 2.47 %.

7 REPORT

Report any variation from the recommended test procedure.