IS 15522 : 2004
ISO 12644 : 1996

भारतीय मानक

ग्राफिक प्रौद्योगिकी — फॉलिंग रॉड विस्कोमीटर द्वारा पेस्ट स्याही और माध्यम के चासनी सोखने के गुण ज्ञात करना

Indian Standard

GRAPHIC TECHNOLOGY — DETERMINATION OF RHEOLOGICAL PROPERTIES OF PASTE INKS AND VEHICLES BY THE FALLING ROD VISCOMETER

ICS 87.080

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

November 2004

Price Group 6

NATIONAL FOREWORD

This Indian Standard which is identical with ISO 12644 : 1996 'Graphic technology — Determination of rheological properties of paste inks and vehicles by the falling rod viscometer' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendations of the Printing Inks, Stationery and Allied Products Sectional Committee and approval of the Chemical Division Council.

The text of this ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Some terminology and certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

For tropical countries like India, the standard temperature and the relative humidity shall be taken as 27 ± 2°C and 65 ± 5 percent respectively.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounding off value should be the same as that of the specified value in this standard.

Indian Standard

GRAPHIC TECHNOLOGY — DETERMINATION OF RHEOLOGICAL PROPERTIES OF PASTE INKS AND VEHICLES BY THE FALLING ROD VISCOMETER

1 Scope

This International Standard specifies the procedure for determining the viscosity and yield value of paste inks and vehicles which are unreactive under normal room conditions.

It is applicable to inks in the apparent viscosity range of 2 Pa · s to 200 Pa · s.

2 Definitions

For the purposes of this International Standard, the following definitions apply.

2.1 viscosity: Measure of the internal friction of a liquid in motion. The viscosity is generally defined as the ratio of the shear stress (2.2) to the shear rate (2.3):

$$\eta = \frac{\sigma}{\gamma} \qquad \ldots (1)$$

2.2 shear stress, σ: Force per area in a direction parallel to the applied force. Unit: Pa.

NOTES

1 For the falling rod viscometer, the shear stress is proportional to the total weight of the rod and the weight loads in accordance with the equation

$$\sigma = \frac{W}{A} = \frac{mg}{2\pi rl} \qquad \ldots (2)$$

where (see figures 1 and 2)

- σ is the shear stress;
- W is the total weight of the rod and the weight loads;
- A is the apparent shearing area;
- g is the gravitational acceleration;
- m is the total mass;
- r is the radius of the rod;
- l is the length of the aperture.

2 The shearing length of the aperture of a falling rod viscometer usually contains both a tapered and a parallel section; therefore, it is understood that A is not the true shearing area but an apparent shearing area.

2.3 shear rate, γ: Velocity gradient through a stressed liquid in a direction perpendicular to the shearing area. Unit: s^{-1}.

NOTE — For the falling rod viscometer, γ is inversely proportional to all fall time according to the equation

$$\gamma = \frac{L}{r \cdot \ln(R/r) \cdot t} \quad \ldots (3)$$

where

γ is the shear rate;

L is the falling distance of the rod;

r is the radius of the rod;

R is the radius of the aperture;

t is the fall time.

If the ratio of the radii of the rod and aperture is close to unity, the term may be simplified to

$$\gamma = \frac{L}{st} \quad \ldots (4)$$

where s is the thickness of the ink in the nip determined by the difference between radii of the aperture and of the rod.

2.4 apparent viscosity, η_a: Ratio of the shear stress σ to the shear rate γ for a given shear stress or shear rate. Unit: Pa · s.

2.5 Newtonian liquid: Liquid whose shear stress is proportional to shear rate.

2.6 non-Newtonian liquid: Liquid whose shear stress is not proportional to shear rate.

NOTES

1 There are two types of non-Newtonian liquids: With shear thickening liquids, the viscosity increases with shear rate; with shear thinning liquids, the viscosity decreases with shear rate.

2 If the viscosity of a liquid decreases with application of steady mechanical stress from a value at the state of rest to a final value and increases again if the stress ceases, the liquid is called thixotropic.

2.7 flow curve: Graph of the shear stress σ as a function of the shear rate γ or vice versa.

2.8 Casson model (see A.1)**:** Flow model which assumes a non-linear increase of shear stress σ with increasing shear rate γ. A minimum stress σ_0 is required to initiate flow.

2.9 Bingham model (see A.2)**:** Flow model which assumes a linear increase of the shear stress σ with increasing shear rate γ. A minimum stress σ_0 is required to initiate flow.

2.10 Power Law model (see A.3)**:** Flow model which assumes an increase of the shear stress σ of a liquid proportional to the Nth power of the shear rate γ.

2.11 yield stress, σ_0: Minimum stress required to initiate flow of a liquid. Unit: 1 Pa.

2.12 pseudo yield stress, σ_y: Shear stress at a defined low shear rate when applying the Power Law model, typically to 2,5 s^{-1}.

2.13 reference temperature: Temperature (25 °C) for which all results are reported. Unit: °C.

NOTE — Measurements made at temperatures different from this temperature are corrected (see 6.2.2).

2.14 test temperature: Actual temperature of the aperture ring during measurements. Unit: °C.

2.15 shortness ratio: Ratio of yield stress or pseudo yield stress to the apparent viscosity. Unit: 1 s^{-1}.

3 Test method

3.1 Principle

The principle of this test is the measurement of the relative velocity between a vertical rod and an aperture ring. The bottom of the rod is inserted into the aperture. The gap is filled with the test fluid, which is sheared when the rod falls.

By loading the rod with different load weights, different shear rates are obtained. By applying linear regression methods to the measured fall times as a function of load weight, the viscosity and the yield stress can be calculated.

3.2 Apparatus

3.2.1 Falling rod viscometer

The viscometer consists of

— a cylindrical rod (figure 1) made from metal or any other hard material. In order to obtain comparable values for shear stress and resulting shear rate the mass of the steel rod should be (132 ± 1) g.

— a metal ring (figure 2) with a defined cylindrical or conical aperture. The ring is fixed on a support and should be temperature controlled. Since the diameter of rod and aperture are critical they are manufactured within low tolerances. These dimensions shall be supplied by the manufacturer. To minimize possible gap differences, only matching sets of rod and aperture ring shall be used.

— load weights to be loaded on top of the rod. Series of load weights are combined to sets. Sets of load weights with the following masses should be used:

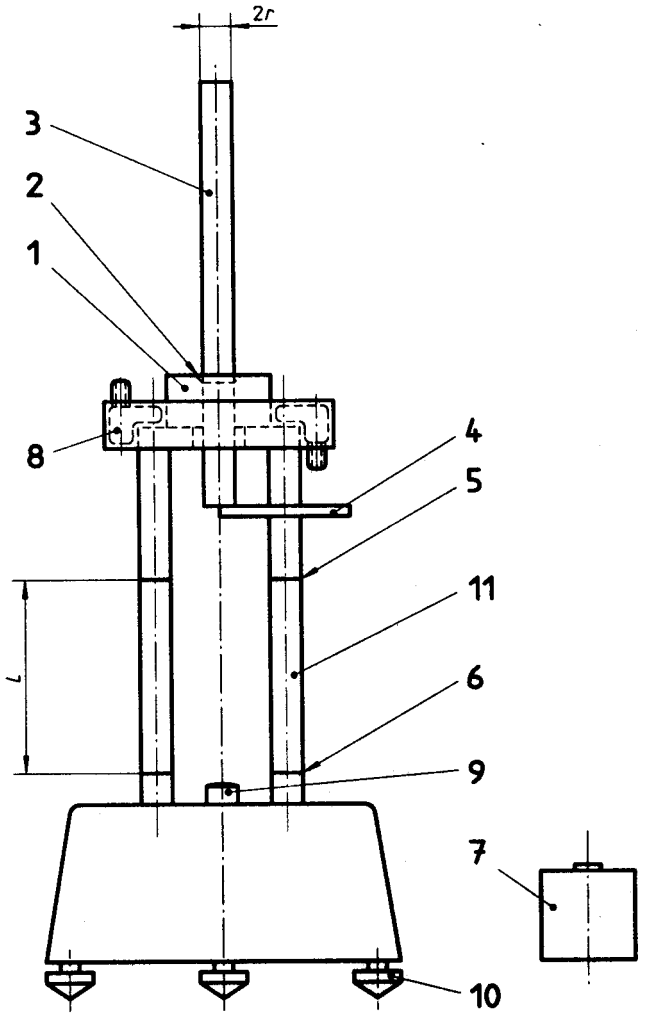
A:	5 000,	4 000,	3 000,	2 000,	1 000
B:	3 000,	2 000,	1 500,	500	
C:	1 500,	1 000,	800,	500	
D:	800,	600,	400,	200	
E:	400,	300,	200,	100	
F:	200,	100,	50,	0	

The tolerance for the masses of load weights shall be ± 0,2 g;

— a designated measuring distance marked on the strut. The tolerance shall be ± 0,2 mm. Sensors may be placed at the marks.

— a levelling device.

— a timing device. The tolerance shall be ± 0,1 s (should be ± 0,01 s).



Key

1	Ring	9	Level
2	Aperture	10	Horizontally adjusting screw
3	Rod	11	Strut
4	Support	r	Radius of the rod
5	Measuring distance, upper mark	R	Radius of the aperture
6	Measuring distance, lower mark	l	Length of the aperture
7	Weight	L	Measuring distance
8	Water jacket		

Figure 1 — Falling rod viscometer

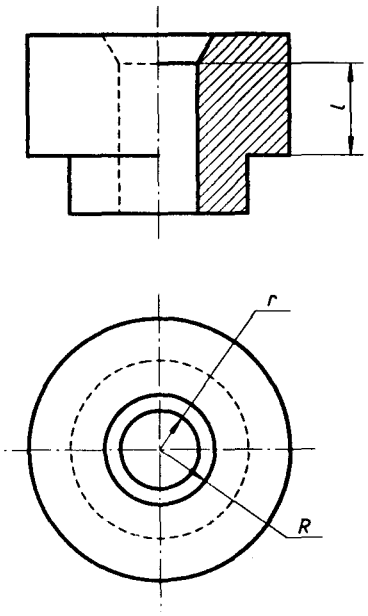


Figure 2 — Aperture ring

3.2.2 Temperature control

Means shall be provided for measurement and control of the test temperature.

3.2.3 Others

Non-scratching spatulas.

Standard viscosity oils (at least 2) for calibration.

NOTE — The viscosity of the standard viscosity oils shall be in the same range as that of the test samples. The viscosity of these oils shall be traceable to a standards institution. An internal standard may be used for comparative studies only.

3.3 Ambient temperature control

The test shall be carried out under controlled ambient temperature. This can be achieved either by placing the viscometer in a thermostatically controlled cabinet or by working under constant room temperature.

If working in a cabinet, the inner temperature should not vary from the test temperature by more than ± 0,5 °C. For room conditioning, a difference of ± 2 °C to the test temperature is allowed. The standard reference temperature shall be (25 ± 0,2) °C.

3.4 Preparation for testing

Prior to use, the test sample (about 5 g) shall be kneaded by a spatula and equilibrated to test temperature. The sample shall be homogeneous and not contain any coarse particles.

The proper set of load weights is selected according to the expected results.

NOTE — The fall time with the highest load weight should normally be in the range 4 s to 10 s. For heat-set printing inks it may be desirable to use a shorter fall time for the highest weight.

An amount of the test sample sufficient to coat the rod and aperture is applied to the lower part of the rod. By turning the rod, the sample is distributed uniformly. Running a single pass with the highest weight load, the rod and the aperture ring are wetted by the liquid. The rod is inserted into the aperture and rests on the support before the test run is started.

3.5 Test procedure

The sample is tested with the selected series of load weights in descending order. The fall time shall not exceed 60 s. After each run, the rod is scraped with the spatula and the liquid which was scraped off is reapplied on the lower part of the rod. During the test, additional liquid shall not be added.

At the beginning and at the end of the test, the temperature of the sample is checked.

For highly thixotropic samples, it may be necessary to make a dummy run first.

3.6 Cleaning

After the test, the instrument shall be cleaned immediately with a lint-free wiper and a suitable solvent.

4 Calibration

When installing the viscometer, locate the instrument on a sturdy bench in a draught-free environment. Use the levelling device to obtain proper vertical alignment. The timing device and the distance between the two sensors are calibrated during initial installation. The timing device should be recalibrated regularly.

The calibration shall be performed by testing the standard viscosity oils according to the procedure described in 3.5.

4.1 Calibration for the Casson and Bingham models (see A.1 and A.2)

Assuming that the standard viscosity oils are strictly Newtonian, the calculation is as follows:

From (1), (2) and (4) follows:

$$\eta = \frac{\sigma}{\gamma} = \frac{mg}{2\pi rl} \times \frac{st}{L} \qquad \ldots (5)$$

where

η is the viscosity;

σ is the shear stress;

γ is the shear rate;

m is the mass;

g is the gravitational acceleration;

r is the radius of the rod;

l is the length of the aperture;

s is the thickness of the ink in the nip;

t is the fall time;

L is the measuring distance.

Fixed parameters and constants are combined as device factors α and β

$$\alpha = \frac{L}{s} \quad \ldots (6)$$

and

$$\beta = \frac{g}{2\pi r l} \quad \ldots (7)$$

The unit of α is 1 and the unit of β is Pa/kg.

Since the shear rate

$$\gamma = \frac{\alpha}{t} \quad \ldots (8)$$

and shear stress

$$\sigma = \beta m \quad \ldots (9)$$

the viscosity of a Newtonian fluid can be calculated from the slope of a plot of γ versus σ for different masses. The measured viscosity is the reciprocal slope of the linear regression line.

If a certain set of rod and aperture ring shows viscosity variations of > 20 % from the specifications of the standard viscosity oil it shall be discarded. Smaller differences are compensated by using a correction factor Φ:

$$\Phi = \eta_{\text{true}} / \eta_{\text{measured}} \quad \ldots (10)$$

The correction factor Φ is specific for a single set of rod and aperture. It is recommended that a calibrated set of aperture and rod be kept as an internal standard.

4.2 Calibration for the Power Law model

For determination of the device factor β (stress constant) defined in equation (7) it is necessary to measure the radius r of the rod and the length l of the aperture. Both dimensions shall be measured to 0,01 mm. Given these data the device factor β is calculated according to equation (7).

The value of the device factor α defined in equation (6) shall be computed from fall time runs of the standard viscosity oils. For that purpose, measurements shall be taken with at least two such oils, covering the viscosity range of interest, and at least four fall times. The known viscosity of the oils is divided by the fall time t for each weight load with the mass m and this quotient is plotted versus the mass for each standard viscosity oil. According to equation (11), the slope of the regression line is the quotient β/α of the two device factors.

$$\frac{\eta_{\text{std}}}{t} = \frac{\beta}{\alpha} m \quad \ldots (11)$$

where

η_{std} is the viscosity of the standard viscosity oil;

t is the fall time;

m is the mass of the weight load.

Having calculated β from the geometric dimensions of rod and aperture according to the above method, α can easily be calculated. With the device factors α and β determined, values for the shear rate γ and shear stress σ can be calculated from the fall time t and the mass m used as shown in equations (8) and (9).

5 Calculation

5.1 Calculation for Casson and Bingham models (see A.1 and A.2)

Calculation for Casson and Bingham models requires:

— at least 4 fall times as a function of different mass of the weight loads;

— the device factors α, β;

— the correction factor Φ;

— the test temperature at the beginning and the end of the test.

Equations (8) and (9) together with the device factors α and β are used to calculate γ and σ values. For the Casson model, the linear regression of $\sqrt{\gamma}$ as a function of $\sqrt{\sigma}$ leads to η as described in A.1. For the Bingham model the linear regression of γ as a function of σ leads to η as described in A.2.

The correlation coefficient should be calculated as a measure of the repeatability of the test. For correlation coefficient values of 0,999 or better the results are reliable. In case of lower correlations the test shall be repeated.

5.2 Calculation for Power Law model (see A.3)

Calculation for Power Law model requires:

— at least four fall times as a function of different mass of the weight loads;

— the device factors α and β;

— the test temperature at the beginning and end of the test.

Equations (8) and (9) are used together with the device factors α and β to calculate γ and σ values. A linear regression of these values plotted double logarithmically according to equation (18) in A.3 is used to determine k and N. These values are used with equation (18) in A.3 to calculate apparent viscosity (η_{2500}), the pseudo yield stress ($\sigma_{2,5}$), and optionally, the shortness ratio. Correlation coefficients for the regression line shall be 0,999 or better for reliable results. Temperature corrections may be applied as described in 6.2.

6 Corrections

6.1 Corrections for Casson and Bingham models

6.1.1 Viscosity corrections

According to the calibration procedure, the viscosity is corrected by

$$\eta = \eta_{\text{measured}} \Phi \qquad \ldots (12)$$

(see 4.1).

6.1.2 Temperature corrections

Viscosity is strongly temperature dependent. Therefore the temperature shall be checked before and after the test. The test temperature is defined to be the arithmetical average of these values.

Principally, if the test temperature varies from reference temperature (25 °C) by more than 0,2 °C before the runs the thermostatic equipment shall be reset. If the temperature during the test varies more than 1 °C, the test shall be repeated. If the variation is less, the following equation is used to correct the fall time to the reference temperature of 25 °C:

$$t = t_{\text{measured}} \left[1 + \delta \left(\vartheta - 25\right)\right] \qquad \ldots (13)$$

where

t is the fall time;

ϑ is the test temperature, in degrees Celsius;

δ is proportional to the temperature gradient of viscosity.

A value of $\delta = 0{,}1\ °C^{-1}$ may be assumed for printing inks unless more precise data are available.

6.2 Corrections for Power Law model

6.2.1 Viscosity corrections

As in 6.1.1.

6.2.2 Temperature corrections

Viscosity is strongly temperature dependent. Therefore the temperature shall be checked before and after the test. The test temperature is defined to be the arithmetical average of these values.

Principally, if the test temperature varies from reference temperature (25 °C) by more than 0,2 °C before the runs the thermostatic equipment shall be reset. If the temperature during the test varies more than 1 °C, the test shall be repeated. If the variation is less, the following equation is used to correct the fall time to the reference temperature of 25 °C:

$$\eta = \eta_{\text{calculated}} \left[1 + \delta \left(\vartheta - 25\right)\right] \quad \ldots (14)$$

where

η is the viscosity;

ϑ is the test temperature, in degrees Celsius;

δ is proportional to the temperature gradient of viscosity.

A value of $\delta = 0{,}1\ °C^{-1}$ may be assumed for printing inks unless more precise data are available. The same equation may be used to correct pseudo yield stress, but with $\delta = 0{,}05\ °C^{-1}$.

7 Report

The following information shall be reported:

— Viscosity data referring to the reference temperature of 25 °C;
— Sample designation;
— Test temperature (if not 25 °C);
— Flow model used for calculation;
— Viscometer model;
— Rod material (if not steel);
— Any deviations from this International Standard;
— Date of testing;
— Operator.

Annex A
(normative)

Flow models

All models mentioned in this annex are suitable for describing the rheological behaviour of inks. The selection of a specific model should be determined by practical experience.

The values of each of the rheological quantities strongly depend on the selection of the flow model. There are no equations to transform the results from one model to any other.

Three main flow models are used to describe the rheological behaviour of paste inks and vehicles (A.1 to A.3).

A.1 Casson model

This model assumes

— a non-linear increase of shear rate γ with increasing shear stress σ;

— a minimum stress required to initiate flow.

This is called non-linear plastic flow and follows the function

$$\eta = \frac{\left(\sqrt{\sigma} - \sqrt{\sigma_0}\right)^2}{\gamma} \qquad \ldots (15)$$

where

η is the viscosity;

γ is the shear rate;

σ is the shear stress;

σ_0 is the minimum stress (yield stress).

In practice, σ_0 is obtained from the abscissa intercept $\sqrt{\sigma_0}$ of the linear regression line when $\sqrt{\gamma}$ is plotted versus $\sqrt{\sigma}$ (see figure A.1). The slope of this line is $\sqrt{1/\eta}$.

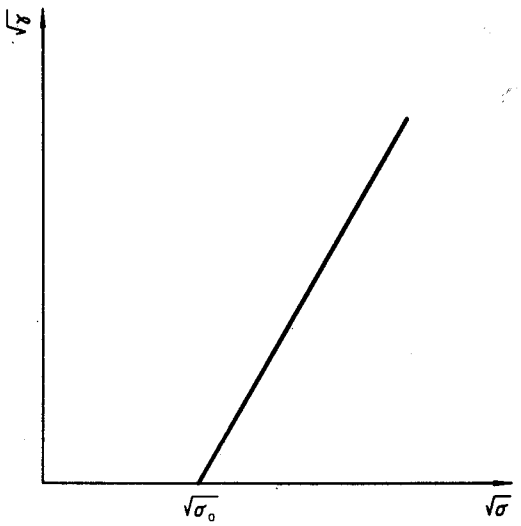


Figure A.1 — Casson model

A.2 Bingham model

The Bingham model assumes

— a linear increase of the shear rate γ with increasing shear stress σ;

— a minimum stress required to initiate flow.

This is called ideal plastic flow and follows the function

$$\eta = \frac{\sigma - \sigma_0}{\gamma} \quad \ldots (16)$$

where

γ is the shear rate;

σ_0 is the minimum stress (yield stress);

η is the viscosity.

In practice, γ_0 is obtained as the abscissa intercept σ_0 of the linear regression line when γ is plotted as a function of σ (see figure A.2). The slope in this diagram is $1/\eta$.

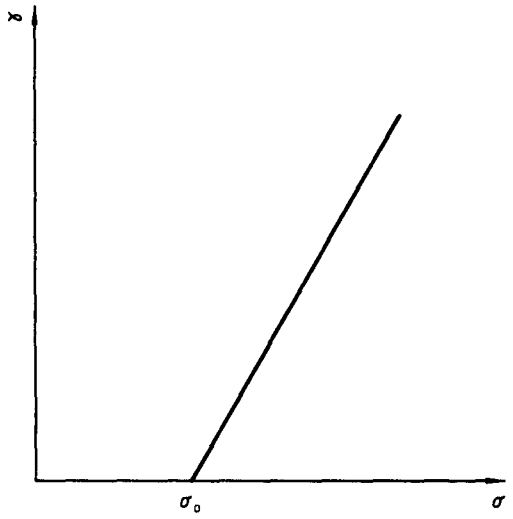


Figure A.2 — Bingham model

A.3 Power Law model

The Power Law model assumes that the shear stress σ of a liquid varies with shear rate γ in accordance with the function

$$\sigma = k\gamma^N \quad \ldots (17)$$

where

k is a constant related to the viscosity of the liquid;

N is a constant describing the rate at which shear stress varies with shear rate.

Three possible ranges of N have the following meaning:

$N < 1$ shear thinning liquid;

$N = 1$ Newtonian liquid;

$N > 1$ shear thickening liquid.

In practice, a double logarithmic plot of shear stress versus shear rate is used to obtain a linear relation.

$$\log \sigma = \log k + N \log \dot{\gamma} \qquad \ldots (18)$$

Values of N and k are obtained from a linear regression of a plot of the data where the ordinate intercept yields log k and the slope of the line yields N (see figure A.3). With equation (17) and/or (18) values of σ at any desired shear rate may be obtained.

Reporting should include the following information:

— apparent viscosity at 2 500 s^{-1};

— the value of N as the degree of non-Newtonianism;

— the pseudo yield stress at 2,5 s^{-1} (optionally);

— and the shortness ratio (optionally);

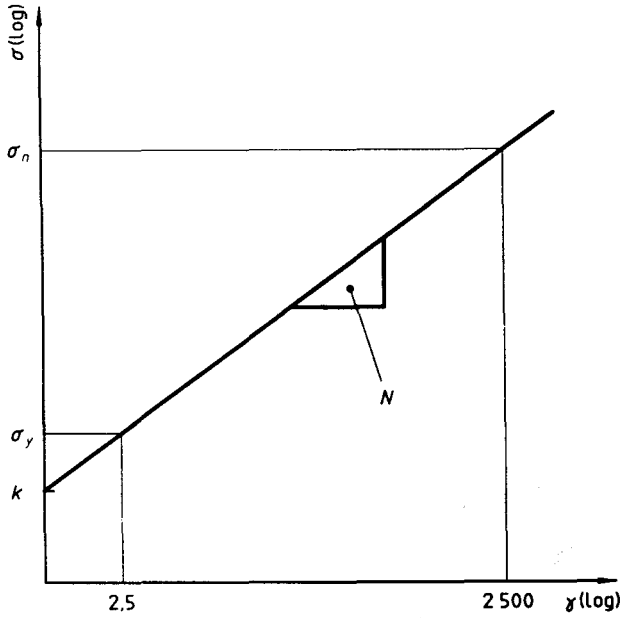


Figure A.3 — Power Law Model

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act,* 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

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