

BUREAU OF INDIAN STANDARDS

DRAFT FOR COMMENTS ONLY

(Not to be reproduced without the permission of BIS or used as an Indian Standard)

भारतीय मानक मसौदा

खाद के लिए नीम केक - विशिष्टि

(आई एस 8558 का पहला पुनरीक्षण)

Draft Indian Standard

NEEM CAKE FOR MANURING – SPECIFICATION

(*First Revision of IS 8558*)

ICS No. 65.080

Soil Quality and Fertilizers Sectional
Committee, FAD 07

Last Date of Comments: **25 July 2024**

FOREWORD

(*Formal clauses will be added later*)

Large quantities of *Neem* Cake are available in the country and are used for manuring. It has an additional advantage of possessing pesticidal properties and finds favour with agriculturists. *Neem* Cake acts as nitrification inhibitor.

The standard was first published in 1977 and in the preparation of this standard, assistance was derived from the data furnished by the manufacturers of the oilcakes and different research institutes and particularly the All India Non-edible Oil Industry Association, Pune. Reference was also made to MITRA (C R) *NEEM*, 1963, Indian Central Oilseeds Committee, Hyderabad.

In this revision, the standard has been brought out in the latest style and format of the Indian Standards, and references to Indian Standards wherever applicable have been updated. It also incorporates one amendment issued to this standard.

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 or analysis, shall be rounded off in accordance with IS 2: 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for *Neem* Cake for manuring. The oilcake obtained by all processes, including solvent extraction, is covered in this standard.

2 REFERENCES

The standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

<i>IS No.</i>	<i>Title</i>
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
IS 266 : 1993	Sulphuric acid — Specification (<i>third revision</i>)
IS 323 : 2009	Rectified spirit for industrial use — Specification (<i>second revision</i>)
IS 460 (Part 1) : 2020	Test Sieves — Specification: Part 1 Wire cloth test sieves (<i>fourth revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)

3 REQUIREMENTS

3.1 Description

The material shall be of uniform texture, clean and free from adulterants, such as sand, dust and metallic pieces.

3.2 The material shall pass wholly through 2.36 mm IS Sieve of which not less than 70 percent shall pass through 850 microns IS Sieve [*see IS 460(Part 1)*].

3.3 The material shall comply with the requirements given in Table 1.

Table 1 Requirements for *Neem* Cake for Manuring
(*Clause 3.3*)

SI No.	Characteristic	Requirement	Method of Test (Ref to)
(1)	(2)	(3)	(4)
i)	Moisture, percent by mass, <i>Max</i>	10	Annex A
ii)	Water insoluble organic nitrogen, percent by mass, on moisture-free basis, <i>Min</i>	2.5	Annex B
iii)	Total ash, percent by mass, <i>Max</i>	13	Annex C
iv)	Acid insoluble ash, percent by mass, on moisture-free basis, <i>Max</i>	5	Annex D

4 PACKING

The material shall be packed in HDPE bags or jute bags with loose polyethylene film or in such other containers as agreed to between the purchaser and the supplier.

5 MARKING

5.1 Each bag shall be suitably marked indicating clearly that the material is meant 'for manuring' and shall also be marked with the following information:

- a) Name of the material;
- b) Name and address of the manufacturer;
- c) Batch number;
- d) Net quantity;
- e) Date of packing; and
- f) Any other information required under the *Legal Metrology (Packaged Commodities) Rules, 2011*.

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

Representative test samples of the material shall be drawn as prescribed in Annex E.

7 TEST METHODS

Tests shall be carried out by the appropriate methods referred to in col (4) of Table 1.

8 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see IS 1070*) shall be used in tests.

NOTE - 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A
[Table 1, Sl No. (i)]
DETERMINATION OF MOISTURE

A-1 PREPARATION OF THE SAMPLE

Grind about 100 g of the sample so that it passes through 1.00 mm IS Sieve [see IS 460 (Part 1)]. Transfer this prepared sample to a well-stoppered glass bottle.

A-2 PROCEDURE

Weigh accurately about 5 g of the prepared sample (see A-1) in a tared aluminium dish with a cover, having a diameter of at least 50 mm and a depth of about 40 mm. Shake the dish until the contents are evenly distributed. With cover removed, place the dish and cover in an air-oven maintained at $(135 \pm 2)^\circ\text{C}$ and dry for at least 2 h. Place cover on dish, cool in a desiccator and weigh. Repeat the process of heating, cooling and weighing until the difference in mass between two successive weighings is less than one milligram.

A-3 CALCULATION

$$\text{Moisture, percent by weight} = \frac{(M_1 - M_2)}{(M_1 - M)} \times 100$$

where

M_1 = mass, in g, of the dish with the material before drying;

M_2 = mass, in g, of the dish with the material after drying; and

M = mass, in g, of the empty dish.

ANNEX B
[Table 1, Sl No. (ii)]
DETERMINATION OF WATER INSOLUBLE ORGANIC NITROGEN

B-1 APPARATUS

B-1.1 Kjeldahl Flask – 500 ml capacity.

B-1.2 Distillation Assembly

The assembly consists of a round-bottom flask of a 1 000 ml capacity fitted with a rubber stopper through which passes one end of the connecting bulb tube. The other end of the bulb tube is connected to the condenser which is attached by means of a rubber tube to a dip tube which dips into a known quantity of standard sulphuric acid or boric acid solution contained in a conical flask of 500 ml capacity, to which 3 to 4 drops of methyl red indicator solution have been added.

B-2 REAGENTS

B-2.1 Potassium Sulphate or Anhydrous Sodium Sulphate

B-2.2 Copper Sulphate

B-2.3 Concentrated Sulphuric Acid – r.d. 1.84 (*see* IS 266).

B-2.4 Sodium Hydroxide Solution – Dissolve about 450 g of sodium hydroxide in 1000 ml of water.

B-2.5 Standard Sulphuric Acid – 0.5 N.

B-2.6 Standard Sodium Hydroxide Solution – 0.25 N.

B-2.7 Methyl Red Indicator Solution – Dissolve 1 g of methyl red in 200 ml of rectified spirit (*see* IS 323), 95 percent by volume.

B-2.8 Boric Acid Solution – Saturated

Dissolve 60 g of boric acid in 1 litre of hot water, cool and allow to mature for 3 days before decanting the clear liquid.

B-2.9 Magnesium Oxide – Carbonate free, freshly ignited.

B-3 PROCEDURE

Transfer carefully about 2 g of the prepared sample (*see* A-1), accurately weighed, to the Kjeldahl flask. Add about 10 g of potassium sulphate or anhydrous sodium sulphate, about 0.5 g of copper sulphate and 25 ml or more, if necessary, of concentrated sulphuric acid. Place the flask in an inclined position, and heat below the boiling point of the acid until frothing ceases. Increase heat until the acid boils vigorously and digest for a time after the mixture is clear or until oxidation is complete (about 2 h). Cool the contents of the flask. Transfer quantitatively to the round-bottom flask with water, the total quantity of water used being about 200 ml. Add a few pieces of pumice stone to prevent bumping. Add carefully the sodium hydroxide solution in quantity which is sufficient to make the solution alkaline by the side of the flask so that it does not mix at once with the acid solution but forms a layer below the acid layer. Assemble the apparatus taking care that the tip of the dip tube extends below the surface of the standard sulphuric acid solution in the receiver. Mix the contents of the flask by shaking and distil until all ammonia has passed over into the standard sulphuric acid solution. Titrate with standard sodium hydroxide solution.

Carry out a blank determination using all reagents in the same quantities but without the material to be tested.

B-4 CALCULATION

$$\text{Water insoluble organic nitrogen, percent by mass (on moisture free-basis)} = \frac{140 (B-A) N}{m (100-M)}$$

where

B = volume in ml of the standard sodium hydroxide solution used to neutralize the acid in blank determination;

- A* = volume in ml of the standard sodium hydroxide solution used to neutralize the excess acid in the test with the material;
N = normality of the standard sodium hydroxide solution;
m = mass in g of the material taken for the test; and
M = percentage moisture in the sample (*see A-3*).

ANNEX C

[Table 1, Sl No. (iii)]

DETERMINATION OF TOTAL ASH

C-1 PROCEDURE

Weigh accurately about 2 g of the dried material (*see A-2*) in a tared porcelain, silica or platinum dish. Ignite with the flame of a Meker burner for about one hour. Complete the ignition by keeping in a muffle furnace at (550 ± 20) °C until grey ash results, Cool in a desiccator and weigh. Ignite the dish again in the muffle furnace for 30 min, cool and weigh. Repeat this process until the difference in mass between two successive weighings is less than 1 mg. Note the lowest mass.

NOTE – Preserve the dish containing this ash for the determination of acid insoluble ash (*see Annex D*).

C-2 CALCULATION

$$\text{Total ash (on moisture free basis), percent by mass} = \frac{(M_2 - M)}{(M_1 - M)} \times 100$$

where,

M_2 = the lowest mass in g of the dish with the ash;

M_1 = mass in g of the dish with the dried material taken for the test; and

M = mass in g of the empty dish.

ANNEX D

[Table 1, Sl No. (iv)]

DETERMINATION OF ACID INSOLUBLE ASH

D-1 REAGENTS

D-1.1 Dilute Hydrochloric Acid - approximately 5 N, prepared from concentrated hydrochloric acid (*see IS 265*)

D-1.2 Procedure

Weigh accurately 2 g of the dried material (*see A-2*) in a tared porcelain, silica or platinum dish. Ignite with a Meker burner for about 1 h. Complete the ignition by keeping in a muffle furnace at (550 ± 20) °C until grey ash results. Moisten with concentrated hydrochloric acid and evaporate to dryness. Keep in an electric air-oven maintained at (135 ± 2) °C for about 3 h. Cool and add 25 ml of dilute hydrochloric acid, cover with a watch-glass and heat on a water-bath for 10 min. Cool and filter through Whatman filter paper No. 42 or its equivalent. Wash the residue with hot water until tile washings are free from chlorides as tested with silver nitrate solution and return the filter paper and residue to the dish. Ignite it in a muffle furnace at (550

± 20) °C for 1 h. Cool in a desiccator and weigh. Ignite the dish again for 30 min, cool and weigh. Repeat this process till the difference between two successive weighings is less than one milligram. Note the lowest mass.

D-2 CALCULATION

$$\text{Acid insoluble ash (on moisture free basis), percent by mass} = \frac{(M_2 - M)}{(M_1 - M)} \times 100$$

where,

M_2 = lowest mass in g of the dish with the acid insoluble ash;

M_1 = mass in g of the dish with dried material (*see* M_1 in C-2); and

M = mass in g of the empty dish.

ANNEX E

(Clause 6)

SAMPLING OF NEEM CAKE FOR MANURING

E-1 GENERAL REQUIREMENTS

In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

E-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

E-1.2 The sampling device shall be clean and dry when used.

E-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling device and the containers for samples from adventitious contamination.

E-1.4 The samples shall be placed in clean and dry containers. The sample containers shall be of such a size that they are almost completely filled by the sample.

E-1.5 Each container shall be sealed airtight after filling and marked with full details of sampling, date of sampling, batch or code number, name of the manufacturer and other important particulars of the consignment.

E-1.6 All the samples shall be stored in such a manner that there is no deterioration of the material.

E-1.7 Sampling shall be done by a person agreed to between the purchaser and the vendor and in the presence of the purchaser (or his representative) and the vendor (or his representative).

E-2 SCALE OF SAMPLING

E-2.1 Lot

All the bags in a single consignment of the material, drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be grouped separately and the batches in each group shall constitute a separate lot.

E-2.2 For ascertaining the conformity of the material in the lot to the requirements of the specification, samples shall be tested from each lot separately.

E-2.3 The number of bags to be selected from the lot shall depend on the size of the lot and shall be in accordance with Table 2.

Table 2 Scale of Sampling

No. of Bags in the Lot	Sample Size
(1)	(2)
Upto 50	3
51 to 100	4
101 to 200	5
201 to 300	7
301 to 800	8
801 to 1300	9
1301 and above	10

E-2.3.1 The bags shall be chosen at random from the lot and for this purpose a random number table shall be used. For guidance and use of random number tables, IS 4905 may be referred. In the absence of a random number table, the following procedure may be adopted:

Starting from any bag in the lot count them as 1, 2, 3, up to r and so on where r is the integral part of N/n . N being the number of bags in the lot and n is the number of bags to be chosen. Every r^{th} bag so counted shall be withdrawn to constitute the required sample size.

E-3 TEST SAMPLE AND REFEREE SAMPLE

E-3.1 Draw by an appropriate sampling device equal quantities of material from the top, bottom and the sides of each bag select according to Table 2. The total quantity of material drawn from each bag shall be sufficient to make triplicate determinations for all the characteristics given in the specification. Mix all the portions of material drawn from the same bag thoroughly.

E-3.1.1 From the mixed material from each selected bag a small but approximately equal quantity of material shall be taken from each selected bag so as to form a composite sample. The quantity of material in the composite sample shall be sufficient to make triplicate determinations for all the characteristics tested on the composite sample. The composite sample shall be divided into 3 equal parts and transferred to clean and dry containers and labelled with the particulars of sampling given in **E-1.5** and sealed airtight. One of these samples shall be for the purchaser, another for the supplier and the third for the referee.

E-3.1.2 The portions of material from each selected bag (after the material for composite sample has been taken) shall constitute a test sample representing that particular bag. These shall be transferred immediately to clean and dry sample containers sealed air-tight. These shall be levelled with the particulars of sampling given in **E-1.5**. The individual samples obtained as above shall be formed into 3 sets in such a way that each set has a test sample representing each bag selected. One of the sets shall be for the purchaser, another for the supplier and the third for the referee.

E-3.1.3 *Referee Sample*

Referee sample shall consist of a set of test samples (*see* E-3.1.2) and one composite sample (*see* E-3.1.1) and shall bear the seals of purchaser and the supplier. It shall be kept at a place agreed to between the two and used in case of a dispute.

E-4 NUMBER OF TESTS

E-4.1 Tests for water insoluble organic nitrogen shall be conducted on each of the individual samples.

E-4.2 Tests for remaining characteristics given in Table 1 shall be conducted on the composite sample.

E-5 CRITERIA FOR CONFORMITY

The lot shall be declared as conforming to the requirements of the specification if each of the test results on the individual sample satisfies the specified requirements and all test results on the composite sample meet the relevant requirements given in the specification.