IS 14300 : 1995

भारतीय मानक

अजादिरेक्टिन युक्त नीम आधारित पायसनीय सान्द्र – विशिष्टि

Indian Standard

NEEM BASED EC CONTAINING AZADIRACHTIN — SPECIFICATION

(First Reprint JANUARY 1997)

ICS 65.100

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

December 1995

Price Group 1

AMENDMENT NO. 1 MARCH 2000
TO
IS 14300 : 1995 *NEEM* BASED EC CONTAINING AZADIRACHTIN — SPECIFICATION

(*Page* 1, *clause* 3.3.1) — Substitute the following for the existing text:

'The nominal azadirachtin content shall be declared by the manufacturer, when tested by the method prescribed in Annex A. The Azadirachtin content (m/m) of any of the sample shall not be less than declared value.'

(*Page* 2) — Insert the following Annex at the end of text:

ANNEX A

DETERMINATION OF AZADIRACHTIN CONTENT IN *NEEM* BASED FORMULATION EC

A-1 PRINCIPLE

Azadirachtin in the sample is dissolved in methanol-water (90:10) and analyzed on HPLC at 215 nm.

A-2 APPARATUS

A-2.1 High Performance Liquid Chromatography

HPLC unit equipped with ultraviolet (UV) detector and printer-plotter-cum-integrator and operated under the following suggestive parameters. These parameters may be varied as per available facilities provided standardization is done:

i)	Column	—	C-18, 25 cm × 4.6 mm Stainless Steel 5 μ Particle size or equivalent
ii)	Flow rate	—	1.0 ml/min approx
iii)	Detector	—	Ultraviolet (UV) 215 nm
iv)	Mobile phase	—	Acetonitrile : Water (35:65) (*v/v*)
v)	Retention time	—	Azadirachtin — A 10-12 minutes (approx.) Azadirachtin — B 11-13 minutes (appox.)

A-2.2 Microsyringe — 25 µl capacity.

A-3 REAGENTS

A-3.1 Reference Standard — Working standard of known purity.

A-3.2 Acetonitrile — HPLC Grade.

A-3.3 Water — HPLC Grade.

A-3.4 Methanol — HPLC Grade.

A-3.5 Volumetric Flasks — 10 ml, 50 ml and 100 ml.

A-3.6 Pipettes — Graduated, 2 ml and 5 ml.

A-4 PREPARATION OF STANDARD SOLUTIONS

A-4.1 Preparation of Reference Standard Solution

Weigh accurately the 2.0 mg working standard of known purity into 50 ml volumetric flask and dissolve in methanol: water (90:10). Make up to the mark and shake well. Take 2 ml of this solution with the help of pipette into a syringe prepared as per A-4.3. Elute it into 10 ml volumetric flask with repeated washings with methanol : water (90:10). Make up to the mark, shake well.

A-4.2 Preparation of Sample Solution

Weigh accurately sample quantity so as to contain 2.0 mg of Azadirachtin in 50 ml volumetric flask and dissolve in methanol: water (90:10). Shake well and keep it aside to separate the layers. Take 2 ml of this solution with the help of pipette into a syringe prepared as per A-4.3. Elute it into 10 ml volumetric flask with repeated washings with methanol and water (90:10). Make up to the mark and shake well

A-4.3 Preparation of Thin Column of C8 Material in Syringe

Take a sep pak cartridge containing 0.5 g of C18 packing material and wet it with solvent methanol : water (90:10) before loading the sample solution. Alternatively take a 5 ml glass syringe of 10 mm internal diameter and insert a small wad of silanised glass wool inside the syringe to the bottom. Add C18 powder (30-40 µm) to form about 1 cm height column (approx 0.5 g), put another wad of glass wool and wet with solvent (methanol : water 90:10).

A-5 ESTIMATION

A-5.1 Inject 20 µl of working standard and sample solution respectively to get area reproducibility for two consecutive injections. The area of two consecutive injections should not vary by more than 2 percent. From the HPLC chromatogram calculate percentage of Azadirachtin in the sample as in A-5.2.

A-5.2 Calculation

$$\text{Azadirachtin content, percent by mass} = \frac{A_1 \times M_2}{A_2 \times M_1} \times P$$

where

A_1 = peak area of Azadirachtin in sample solution (Aza *A+B*;)

A_2 = peak area of Azadirachtin in working standard (Aza *A+B*);

M_1 = mass, in g of the sample taken for test;

M_2 = mass, in g of the working standard Azadirachtin; and

P = purity of working standard.

A-5.3 Analytical Tolerance

Adopt analytical tolerance after analyzing the fortified samples of oil formulations, if necessary (normally the tolerance level is 10-15 percent).

(FAD 1)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 MARCH 2002
TO
IS 14300 : 1995 *NEEM* BASED EC CONTAINING AZADIRACHTIN — SPECIFICATION

(*Page* 1, *clause* 3.2.5) — Delete.

(FAD 1)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 3 MAY 2002
TO
IS 14300 : 1995 NEEM BASED EC CONTAINING AZADIRACHTIN — SPECIFICATION

[*Page* 2, *clause* A-2.1, *Item* (v) (*see also Amendment No.* 1)] — Insert the following at the end:.

'The above retention time is required to be confirmed by the respective standard.'

(FAD 1)

Reprography Unit, BIS, New Delhi, India

Pesticides Sectional Committee, FAD 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee, had been approved by the Food and Agriculture Division Council.

NEEM based EC containing azadirachtin is used for the control of pests of agricultural crops.

In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act,* 1968 and Rules framed thereunder and the *Standards of Weights and Measures (Packaged Commodities) Rules,* 1977. However, the standard is subject to the restrictions imposed under these wherever applicable.

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 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

NEEM BASED EC CONTAINING AZADIRACHTIN — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for *NEEM* based EC containing azadirachtin.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard.

IS No.	*Title*
1448 [P : 20] : 1982	Method of test for petroleum and its products P:20 Flash point by Abel apparatus (*first revision*)
2771 (Part 1) : 1990	Fibreboard boxes: Part 1 Corrugated fibreboard boxes (*second revision*)
6940 : 1982	Methods of test for pesticides and their formulations (*first revision*)
8190 (Part 2) : 1988	Requirements for packing of pesticides: Part 2 Liquid pesticides (*second revision*)
10627 : 1983	Methods for sampling of pesticide formulations
14299 : 1995	*NEEM* extract concentrate containing azadirachtin

3 REQUIREMENTS

3.1 The material shall consist of *NEEM* based azadirachtin, it may be dissolved in suitable solvent(s) together with emulsifying agent(s) and with stabilizer(s).

3.2 Physical Characteristic

3.2.1 *Description*

The material shall be homogeneous, stable, light to dark brown viscus liquid with a repulsive odour. It shall readily form an emulsion on dilution with water, suitable for use as spray.

3.2.2 *Cold Test*

When tested by the method prescribed in 13.4 of IS 6940:1982, no turbidity or separation of solid or oily matter shall occur.

3.2.3 *Flash Point*

When tested by the method prescribed in IS 1448 [P : 20]:1982, the flash point of the material shall be above 24.5°C.

3.2.4 *Emulsion Stability*

Any separation including creaming at the top and sedimentation at the bottom of 100 ml of emulsion prepared in standard hard water with 2 ml of concentrate shall not exceed 2 ml when tested by the method prescribed in 13.3 of IS 6940 : 1982.

3.2.5 *Heat Stability*

The material shall also meet the requirements given in 3.2.1 to 3.2.4, 3.3.1 to 3.3.3 of this standard after conducting the test prescribed in 13.4 of IS 6940 : 1982.

NOTE — Heat stability test shall not be conducted after the expiry of half of shelf life of the product as ascertained from the date of manufacture and expiry declared on the containers.

3.3 Chemical

3.3.1 *Azadirachtin Content*

The nominal azadirachtin content shall be declared. When tested by the method prescribed in Annex A of IS 14299 : 1995 the azadirachtin content (*m/m*) of any of the sample shall not be less than declared nominal value.

3.3.2 *Acidity/Alkalinity*

When tested by the method prescribed in 13.5 of IS 6940 : 1982, the acidity or alkalinity of the material shall not be more than 0.5 percent by mass as H_2SO_4 or NaOH respectively.

3.3.3 *Aflatoxin Content*

When tested by the method prescribed in Annex B of IS 14299 : 1995, no aflatoxin shall be present.

4 PACKING

The material shall be packed in Aluminium or HDPE containers. For bulk packing HDPE drums shall be used. It shall also conform to the general requirements given in IS 8190 (Part 2) : 1988.

5 MARKING

5.1 The containers shall be marked legibly and indelibly with the following information:

a) Name of the material;

b) Name of the manufacturer;

c) Batch number;

d) Date of manufacture;

e) Date of expiry;

f) Net mass in litres;

g) Azadirachtin content, percent (*m/m*), *nominal and*;

h) Any other requirement as given under the *Insecticides Act*, 1968 and Rules framed thereunder; and *Standards of Weights and Measures (Packaged Commodities) Rules*, 1977.

5.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

5.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 10627:1983.

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.

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 Handbook' and 'Standards Monthly Additions'.

This Indian Standard has been developed from Doc: No. FAD 1 (360)

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Reprography Unit, BIS, New Delhi, India

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(FAD 1)

Reprography Unit, BIS, New Delhi, India

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(FAD 1)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 3 MAY 2002
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