

IS : 7874 (Part I) - 1975
(Reaffirmed 1990)

Indian Standard

**METHODS OF TESTS FOR
ANIMAL FEEDS AND FEEDING STUFFS**

PART I GENERAL METHODS

(Third Reprint MARCH 1992)

UDC 636.085:543

© Copyright 1976

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

March 1976

BE
S
E ONLY

DE

*Indian Standard***METHODS OF TESTS FOR
ANIMAL FEEDS AND FEEDING STUFFS****PART I GENERAL METHODS****Animal Feeds Sectional Committee, AFDC 15***Chairman***DR B. K. SONI***Representing*

Indian Council of Agricultural Research, New Delhi

Members

| | |
|--|---|
| AGRICULTURAL MARKETING ADVISER TO THE GOVERNMENT OF INDIA | Directorate of Marketing and Inspection (Ministry of Agriculture & Irrigation), Faridabad |
| SHRI S. JAYARAMAN (Alternate) | |
| DR S. L. ANAOKAR | Godrej Soaps Private Limited, Bombay |
| DR S. P. ARORA | National Dairy Research Institute (ICAR), Karnal |
| DR B. N. GUPTA (Alternate) | |
| DR C. S. BARSAUL | U. P. College of Veterinary Science & Animal Husbandry, Mathura |
| DR D. C. JOSHI (Alternate) | |
| BRIG R. C. DATTA | Directorate of Military Farms, Army Headquarters |
| SHRI R. K. TRIPATHI (Alternate) | |
| DIRECTOR (VANASPATI) | Directorate of Sugar and Vanaspati (Ministry of Agriculture & Irrigation), New Delhi |
| DEPUTY DIRECTOR (VANASPATI) (Alternate) | |
| DR N. S. DRONAWAT | The Compound Livestock Feeds Manufacturers' Association of India, Bombay |
| SHRI V. VIRMANI (Alternate) | |
| SHRI R. L. GAJWANI | Voltas Limited, Bombay |
| SHRI K. R. FRANCIS (Alternate) | |
| DR A. N. GHOSH | Animal Husbandry Commissioner, Ministry of Agriculture & Irrigation, New Delhi |
| SHRI S. S. CHHIBBAR (Alternate) | |
| HEAD, ANIMAL NUTRITION DIVISION | Indian Veterinary Research Institute (ICAR), Izatnagar |
| DR J. S. ICHHPONANI | Punjab Agricultural University, Ludhiana |
| DR L. S. HUNDAL (Alternate) | |

(Continued on page 2)

● Copyright 1976

BUREAU OF INDIAN STANDARDS

This publication is protected under the *Indian Copyright Act* (XIV of 1957) and reproduction in whole or in part by any means except with written permission of the publisher shall be deemed to be an infringement of copyright under the said Act.

IS : 7874 (Part I) - 1975

(Continued from page 1)

| <i>Members</i> | <i>Representing</i> |
|---------------------------------------|---|
| DR M. G. JACKSON | Govind Ballabh Pant University of Agriculture & Technology, Pantnagar (Dist Nainital) |
| SHRI N. S. MAINI | Department of Agriculture, Ministry of Agriculture & Irrigation, New Delhi |
| SHRI S. S. CHAUHAN (Alternate) | |
| DR V. M. PATIL | Dairy Development Commissioner, Government of Maharashtra, Bombay |
| DR B. SAHAI (Alternate) | |
| DR D. V. RANONEKAR | Bhartiya Agro-Industries Foundation, Uruli-Kanchan (Dist Pune) |
| DR D. V. R. PRAKASH RAO | Shaw Wallace & Company Limited, Madras |
| SHRI B. LAKSHMANAN (Alternate) | |
| DR A. P. SACHDEV | All India Association of Poultry Industry, Bombay |
| DR G. F. MITHUJI (Alternate) | |
| DR A. SAIKIA | College of Veterinary Science, Assam Agricultural University, Gauhati |
| DR P. K. SHARMA (Alternate) | |
| DR S. K. SARKAR | Roller Flour Millers' Federation of India, New Delhi |
| SHRI S. M. MAZUMDAR (Alternate) | |
| DR (SMT) SAROJINI CHANDRA-SHEKHARAN | Kaira District Co-operative Milk Producers' Union Limited, Anand |
| DR A. S. DAVE (Alternate) | |
| DR N. SATPATHY | The Tata Oil Mills Company Limited, Bombay |
| DR K. C. DANDONA (Alternate) | |
| DR H. C. SAXENA | Hindustan Lever Limited, Bombay |
| DR S. YAMDAGNI (Alternate) | |
| DR K. C. SEN | In personal capacity (403, Jodhpur Park, Calcutta) |
| SHRI K. N. R. SHARMA | Directorate General of Technical Development (Ministry of Industries & Civil Supplies), New Delhi |
| DR P. C. SHUKLA | Institute of Agriculture, Anand |
| SHRI G. V. SIRUR | All India Cottonseed Crushers' Association, Bombay |
| SHRI R. V. BIJUR (Alternate) | |
| SHRI SURESH CHANDRA | General Molasses Co (P) Ltd, Sahibabad (Ghaziabad) |
| SHRI T. S. TAMBOLIA | The Maharashtra Agro-Industries Development Corporation Limited, Bombay |
| DR D. N. RANADE (Alternate) | |
| SHRI L. J. TANNA | Solvent Extractors' Association of India, Bombay |
| SHRI M. K. RAHMAN (Alternate) | |
| SHRI VINAY VIRMANI | Jawala Flour Mills, New Delhi |
| SHRI ANAND VIRMANI (Alternate) | |
| SHRI T. PURNANANDAM, | Director General, ISI (Ex-officio Member) |
| Deputy Director (Agri & Food) | |

Secretary

SHRI G. S. VILKHU

Deputy Director (Agri & Food), ISI

(Continued on page 15)

(Continued from page 2)

Animal Feeds and Mineral Mixtures Subcommittee, AFDC 15 : 2

| <i>Convener</i> | <i>Representing</i> |
|---|--|
| DR M. L. MATHUR | National Dairy Research Institute (ICAR), Karnal |
| <i>Members</i> | |
| DR V. D. MUDGAL (<i>Alternate to</i> Dr M. L. Mathur) | |
| AGRICULTURAL MARKETING AD- VISER TO THE GOVERNMENT OF INDIA | Directorate of Marketing and Inspection (Ministry of Agriculture & Irrigation), Faridabad |
| SHRI S. JAYARAMAN (<i>Alternate</i>) | |
| SHRI M. K. DATTARAJ | Mysore Feeds Private Limited, Bangalore |
| SHRI M. K. PANDURANGA SETTY (<i>Alternate</i>) | |
| HEAD, ANIMAL NUTRITION DIVISION | Indian Veterinary Research Institute (ICAR), Izatnagar |
| DR P. V. RAO (<i>Alternate</i>) | |
| DR J. S. ICHHPONANI | Punjab Agricultural University, Ludhiana |
| DR M. G. JACKSON | Govind Ballabh Pant University of Agriculture & Technology, Pantnagar (Dist Nainital) |
| DR T. B. MIRCHANDANI | Aries Agro-Vet Industries Private Ltd, Bombay |
| DR R. S. ADAVI (<i>Alternate</i>) | |
| DR V. M. PATIL | Dairy Development Commissioner, Government of Maharashtra, Bombay |
| DR B. SAHAI (<i>Alternate</i>) | |
| DR B. C. PATNAYAK | Central Sheep & Wool Research Institute (ICAR), Avikanagar (Rajasthan) |
| DR D. V. R. PRAKASH RAO | Shaw Wallace & Company Limited, Madras |
| SHRI B. LAKSHMANAN (<i>Alternate</i>) | |
| SHRI K. V. R. RAO | The Boots Company (India) Ltd, Bombay |
| DR T. R. NATARAJAN (<i>Alternate</i>) | |
| DR (SMT) SAROJINI CHANDRA- SHEKHARAN | Kaira District Co-operative Milk Producers' Union Limited, Anand |
| DR A. S. DAVE (<i>Alternate</i>) | |
| DR H. C. SAXENA | Hindustan Lever Limited, Bombay |
| DR S. YAMDAONI (<i>Alternate</i>) | |
| DR P. C. SHUKLA | Institute of Agriculture, Anand |
| SHRI VINEET VIRMANI | Nandi Provender Mills, New Delhi |
| SHRI ANAND VIRMANI (<i>Alternate</i>) | |

AMENDMENT NO. 1 FEBRUARY 1977

TO

IS:7874(Part 1)-1975 METHODS OF TESTS
FOR ANIMAL FEEDS AND FEEDING STUFFS

PART I GENERAL METHODS

Addenda

(Cover Page and Page 1 - Designation) -

Add the following under the designation:

'(Superseding IS:1714-1960)'.

(Page 3, clause 0.3) - Add the following

at the end:

"This part thus supersedes IS:1714-1960

'Methods of sampling and test for oilcakes
as livestock feed'."

(AFDC 15)

Reprography Unit, BIS, New Delhi, India

**AMENDMENT NO. 2 SEPTEMBER 1991
TO
IS 7874 (Part 1) : 1975 METHODS OF TESTS FOR
ANIMAL FEEDS AND FEEDING STUFFS
PART 1. GENERAL METHODS**

(Page 4, clause 4.1, line 5) — Substitute '100°C' for '135±2°C'.

(FADC 5)

Reprography Unit, BIS, New Delhi, India

DEV

BE
S
E ONLY

Indian Standard

METHODS OF TESTS FOR ANIMAL FEEDS AND FEEDING STUFFS

PART I GENERAL METHODS

0. FOREWORD

0.1 This Indian Standard (Part I) was adopted by the Indian Standards Institution on 28 November 1975, after the draft finalized by the Animal Feeds Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 The importance of adoption of standard and uniform methods of analysis for quality control purposes needs no emphasis. Such methods not only help reducing divergence in the analytical results but also ensure and enable a proper comparison and correct interpretation of the test results.

0.3 During the past decade a large number of Indian Standards covering a wide range of animal feeds, feeding stuffs and feed supplements have been issued and many more are envisaged to be formulated. All these standards include methods of tests that are common. It was, therefore, considered desirable to have all test methods in a consolidated form as applicable to the whole range of animal feeds, feeding stuffs and feed supplements. It was felt that such a step would not only facilitate easy reference but also prevent undue repetition. Accordingly, this standard is being issued.

0.4 It is intended to issue this standard in three parts. This part will cover general methods such as determination of moisture, crude protein, crude fat, crude fibre, total ash, acid insoluble ash, castor husk and *MAHUA* cake. Part II will cover minerals and trace elements and Part III the microbiological methods.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part I) prescribes the general methods of testing animal feeds and feeding stuffs.

*Rules for rounding off numerical values (revised).

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the test results.

3. PREPARATION OF THE SAMPLE

3.1 Grind about 100 g of the sample so that it passes through 1.00-mm IS Sieve (*see* IS : 460-1962†). Alternatively, ASTM Sieve 18 or BS Sieve 16 or Tyler Sieve 16 may be used. Transfer this prepared sample to a well-stoppered glass bottle.

4. DETERMINATION OF MOISTURE

4.1 Procedure — Weigh accurately about 5 g of the prepared sample (*see* 3.1) 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 hours. 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.

4.2 Calculation

$$4.2.1 \text{ Moisture, percent by mass} = \frac{100 (M_1 - M_2)}{M_1 - M}$$

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.

5. DETERMINATION OF CRUDE PROTEIN

5.1 Principle — The percentage of crude protein is ascertained by multiplying the percentage of nitrogen other than ammoniacal nitrogen, by a factor. The quantity of ammoniacal nitrogen is separately determined and deducted from total nitrogen.

*Specification for water, distilled quality (*revised*).

†Specification for test sieves (*revised*).

5.1.1 For animal feeds and feeding stuffs the factor to be used will be 6.25, except in case of wheat and its products for which it will be 5.70.

5.2 Apparatus

5.2.1 *Kjeldahl Flask* — 500 ml capacity.

5.2.2 *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.

5.3 Reagents

5.3.1 *Potassium Sulphate or Anhydrous Sodium Sulphate*

5.3.2 *Copper Sulphate*

5.3.3 *Concentrated Sulphuric Acid* — r.d. 1.84 (see IS : 266-1961*).

5.3.4 *Sodium Hydroxide Solution* — Dissolve about 450 g of sodium hydroxide in 1 000 ml of water.

5.3.5 *Standard Sulphuric Acid* — 0.5 N.

5.3.6 *Standard Sodium Hydroxide Solution* — 0.25 N.

5.3.7 *Methyl Red Indicator Solution* — Dissolve 1 g of methyl red in 200 ml of rectified spirit (see IS : 323-1959†), 95 percent by volume.

5.3.8 *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.

5.3.9 *Magnesium Oxide* — carbonate free, freshly ignited.

5.4 Procedure

5.4.1 *Total Nitrogen* — Transfer carefully about 2 g of the prepared sample (see 3.1), 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 hours). Cool the contents of the flask. Transfer

*Specification for sulphuric acid (revised).

†Specification for rectified spirit (revised).

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.

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

5.4.2 Alternatively, the ammonia evolved by distillation shall be absorbed in boric acid. Carry out digestion as prescribed in 5.4.1. Transfer completely the contents of the digestion flask into the round-bottom flask through the separating funnel. Rinse the separating funnel with water. The total volume of liquid in the distillation flask should not exceed half the capacity of the flask otherwise frothing may occur. Add excess of sodium hydroxide solution to make the solution alkaline. Connect immediately the round-bottom flask to steam trap and condenser. The condenser should be arranged to dip the dip-tube in 50 ml of boric acid which is kept cool in the conical flask. Add 2 or 3 drops of the mixed indicator. Distil about one-third of total volume of the solution in the flask. Cool and dismantle the distillation assembly. Rinse the tip of the condenser and the dip-tube with water, collecting the washings in the receiver. Titrate the ammonia present in the distillate with sulphuric acid until the grass-green colour changes to steel-grey, an additional drop then giving the purple colour.

5.4.3 Ammoniacal Nitrogen — Weigh accurately 2 to 4 g of the prepared sample (see 3.1). Shake it with water and filter. Wash the residue thoroughly with water. Transfer the filtrate to the distillation flask and dilute to about 200 ml with water. Add about 5 g of magnesium oxide. Connect the flask to the condenser by means of the connecting bulb tube and distil about 100 ml of liquid into the receiver containing standard sulphuric acid and methyl red indicator solution. Titrate the contents of the receiver with standard sodium hydroxide solution.

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

5.5 Calculation

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

where

B = volume in ml of the standard sodium hydroxide solution used to neutralize the acid in blank determination (see 5.4.1.1),

A = volume in ml of the standard sodium hydroxide solution used to neutralize the excess acid in the test with the material (see 5.4.1),

N = normality of the standard sodium hydroxide solution,

m = mass in g of the material taken for the test (see 5.4.1), and

M = moisture percentage (see 4.2.1).

5.5.2 When boric acid solution has been used for absorption, calculation of total nitrogen shall be as given below:

$$\text{Total nitrogen (on moisture-free basis), percent by mass} = \frac{140 V N}{m (100 - M)}$$

where

V = volume in ml of standard sulphuric acid used in titration (see 5.4.2),

N = normality of the standard sulphuric acid,

m = mass in g of the material taken for the test (see 5.4.1), and

M = moisture percentage (see 4.2.1).

5.5.3 Ammoniacal nitrogen (on moisture-free basis), percent by mass = $\frac{140 (b - a) N}{m (100 - M)}$

where

b = volume in ml of the standard sodium hydroxide solution used to neutralize the acid in blank determination (see 5.4.3.1),

a = volume in ml of the standard sodium hydroxide solution used to neutralize the excess acid in the test with the material (see 5.4.3),

N = normality of the standard sodium hydroxide solution,

m = mass in g of the material taken for the test (see 5.4.3), and

M = moisture percentage (see 4.2.1).

IS: 7874 (Part I) - 1975

5.5.4 Crude protein (on moisture-free basis),
percent by mass $= 6.25 (X - Y)$

where

X = percent by mass of total nitrogen (see 5.5.1 or 5.5.2),
and

Y = percent by mass of ammoniacal nitrogen (see 5.5.3).

6. DETERMINATION OF UREA NITROGEN

6.1 Principle — The percentage of urea nitrogen is ascertained by deducting the percentage of ammoniacal nitrogen from percent total ammoniacal nitrogen (see 5.5.3) which includes both ammoniacal and the urea nitrogen. However, a suitable qualitative test for detecting the presence of urea nitrogen should precede the quantitative determination as outlined in this method.

6.2 Apparatus

6.2.1 Kjeldahl Flask — 500 ml capacity.

6.2.2 Distillation Assembly — The assembly consists of a round-bottom flask of 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 contained in a conical flask of 500 ml capacity, to which 3 to 4 drops of methyl red indicator solution have been added.

6.3 Reagents

6.3.1 Defoaming Solution — Dissolve 50 g diglycol stearate in 375 ml of benzene, 75 ml of alcohol, and 250 ml of dibutyl phthalate. Warm, if necessary.

6.3.2 Urease Solution — Dissolve standardized urease in water so that each 10 ml neutralized solution will convert the nitrogen of at least 0.1 g pure urea. Prepare fresh urease solution for every determination.

6.3.2.1 Standardization of urease solution — To determine alkalinity of commercial urease preparation dissolve 0.1 g in 50 ml of water and titrate with 0.1 N hydrochloric acid using methyl red indicator. Add the same quantity of 0.1 N hydrochloric acid to each 0.1 g of urease in preparing the urease solution. To determine the enzyme activity, prepare about 50 ml of a neutralized one percent solution of urease. Add different quantities of solution to 0.1 g samples of pure urea and follow with the enzyme digestion and distillation as directed in the determination. Calculate activity of the urease preparation from quantity of the urease solution that converted the urea, thereby permitting complete recovery of the nitrogen by distillation.

6.3.3 Calcium Chloride Solution — Dissolve 25 g calcium chloride in 100 ml of water.

6.3.4 Standard Sulphuric Acid — 0.5 N or 0.1 N when amount of nitrogen is small.

6.3.5 Standard Sodium Hydroxide Solution — 0.25 N or 0.1 N.

6.3.6 Methyl Red Indicator Solution — Dissolve 1 g of methyl red in 200 ml of rectified spirit (see IS : 323-1959*), 95 percent by volume.

6.3.7 Magnesium Oxide — (carbonate-free), freshly ignited, heavy type.

6.4 Procedure

6.4.1 Weigh accurately about 2 g of the prepared sample (see 3.1) in Kjeldahl flask. Shake it with about 250 ml of water. Add 10 ml urease solution, put the stopper tightly and let it stand for 1 hour at room temperature, or 20 minutes at 40°C. Cool to room temperature, if necessary. Use more urease solution if sample contains greater than 5 percent urea (approximately equal to 12 percent protein equivalent). Rinse stopper and neck with few millilitres of water. Add 2 g of magnesium oxide (heavy type), 1 ml of calcium chloride solution, and 5 ml of defoaming solution. Connect the flask to the condenser by means of the connecting bulb tube and distil about 100 ml of liquid into the receiver containing standard sulphuric acid and methyl red indicator solution. Titrate the contents with the standard sodium hydroxide solution.

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

6.5 Calculation

6.5.1 Total ammoniacal nitrogen (urea and ammoniacal nitrogen) (on moisture-free basis), percent by mass

$$= \frac{140 (B - A) N}{m (100 - M)}$$

where

B = volume in ml of the standard sodium hydroxide solution used to neutralize the acid in blank determination (see 6.4.2),

A = volume in ml of the standard sodium hydroxide solution used to neutralize the excess acid in the test with the material (see 6.4.1),

N = normality of the standard sodium hydroxide solution,

m = mass in g of the material taken for the test (see 6.4.1), and

M = moisture percentage (see 4.2.1).

*Specification of rectified spirit (revised).

IS : 7874 (Part I) - 1975

6.5.2 Urea nitrogen = $a - b$

where

a = percent by mass of total ammoniacal nitrogen (see 6.5.1),
and

b = percent by mass of ammoniacal nitrogen (see 5.5.3).

7. DETERMINATION OF CRUDE FAT

7.1 Reagents

7.1.1 *Petroleum Ether* — of boiling range 40°C to 60°C.

7.1.2 *Hexane, Food Grade* — conforming to IS : 3470-1966*.

7.2 **Procedure** — Weigh accurately about 2.5 g of the prepared sample (see 3.1), dried as described under 4.1 and extract with petroleum ether or hexane, food grade, in a Soxhlet or other suitable extractor. The extraction period may vary from 4 hours at a condensation rate of 5 to 6 drops per second to 16 hours at 2 to 3 drops per second. Dry the extract on a steam-bath for 30 minutes, cool in a desiccator and weigh. Continue at 30-minute intervals this alternate drying and weighing until the difference between two successive weighings is less than one mg. Note the lowest mass.

NOTE — If necessary preserve the fat-free material in a desiccator for the determination of crude-fibre content (see 8).

7.3 Calculation

7.3.1 Crude fat (on moisture-free basis),
percent by mass $= \frac{100 (M_1 - M_2)}{m}$

where

M_1 = mass in g of the extraction flask with dried extract,

M_2 = mass in g of extraction flask, and

m = mass in g of the dried sample (see 7.2) taken for the test.

8. DETERMINATION OF CRUDE FIBRE

8.1 Reagents

8.1.1 *Sulphuric Acid* — 0.255 N [1.25 percent (m/v)], accurately prepared.

*Specification for hexane, food grade.

8.1.2 Sodium Hydroxide Solution — 0.313 N [1.25 percent (*m/v*)], accurately prepared.

8.2 Procedure — Weigh accurately about 2 g of the dried material (*see* 4.1) and extract the fat for about 8 hours with petroleum ether or hexane, food grade, using a Soxhlet or other suitable extractor or use the residue from the crude fat determination (*see* Note under 7.2). Transfer the fat-free dry residue to a one-litre conical flask. Take 200 ml of dilute sulphuric acid in a beaker and bring to the boil. Transfer the whole of the boiling acid to the flask containing the fat-free material and immediately connect the flask with a reflux water condenser and heat, so that the contents of the flask begin to boil within one minute. Rotate the flask frequently, taking care to keep the material from remaining on the sides of the flask out of contact with the acid. Continue boiling for exactly 30 minutes. Remove the flask and filter through fine linen (about 18 threads to the centimetre) held in a funnel, and wash with boiling water until the washings are no longer acid to litmus. Bring to the boil some quantity of sodium hydroxide solution under a reflux condenser. Wash the residue on the linen into the flask with 200 ml of the boiling sodium hydroxide solution. Immediately connect the flask with the reflux condenser and boil for exactly 30 minutes. Remove the flask and immediately filter through the filtering cloth. Thoroughly wash the residue with boiling water and transfer to a Gooch crucible prepared with a thin but compact layer of ignited asbestos. Wash the residue thoroughly first with hot water and then with about 15 ml of 95 percent (by volume) ethyl alcohol. Dry the Gooch crucible and contents at $105 \pm 1^\circ\text{C}$ in the air-oven to constant mass. Cool and weigh. Incinerate the contents of the Gooch crucible at $600 \pm 20^\circ\text{C}$ in a muffle furnace until all the carbonaceous matter is burnt. Cool the Gooch crucible containing the ash in a desiccator and weigh.

NOTE — Alternatively, instead of the conical flask and the reflux condenser, use a tall-form spoutless beaker of 600 to 800 ml capacity and cover it with a round-bottomed flask filled with cold water, which acts as a condenser. If the water in the flask becomes hot, it may be replaced by another flask containing cold water.

8.3 Calculation

$$\text{8.3.1 Crude fibre (on moisture-free basis), percent by mass} = \frac{100 (M_1 - M_2)}{m}$$

where

M_1 = mass in g of Gooch crucible and contents before ashing,

M_2 = mass in g of Gooch crucible containing asbestos and ash, and

m = mass in g of the dried material taken for the test.

IS : 7874 (Part I) - 1975

8.3.2 When the residue from fat determination is used:

$$\text{Crude fibre (on moisture-free basis),} \\ \text{percent by mass} = \frac{(M_1 - M_2) (100 - f)}{m_1}$$

where

M_1 = mass in g of Gooch crucible and contents before ashing,

M_2 = mass in g of Gooch crucible containing asbestos and ash,

f = crude fat (on moisture-free basis), percent by mass
(see 7.3.1), and

m_1 = mass in g of the fat-free material taken for the test.

9. DETERMINATION OF TOTAL ASH

9.1 Procedure — Weigh accurately about 2 g of the dried material (see 4.1) 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 $550 \pm 20^\circ\text{C}$ until grey ash results. Cool in a desiccator and weigh. Ignite the dish again in the muffle furnace for 30 minutes, 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 10).

9.2 Calculation

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

where

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

M = mass in g of the empty dish, and

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

10. DETERMINATION OF ACID INSOLUBLE ASH

10.1 Reagent

10.1.1 Dilute Hydrochloric Acid — approximately 5 N, prepared from concentrated hydrochloric acid (see IS : 265-1962*).

*Specification for hydrochloric acid (revised).

10.2 Procedure — Weigh accurately 2 g of the dried material (see 4.1) in a tared porcelain, silica or platinum dish. Ignite with a Meker burner for about one hour. Complete the ignition by keeping in a muffle furnace at $550^{\circ} \pm 20^{\circ}\text{C}$ until grey ash results. Moisten with concentrated hydrochloric acid and evaporate to dryness. Keep in an electric air-oven maintained at $135 \pm 2^{\circ}\text{C}$ for about 3 hours. Cool and add 25 ml of dilute hydrochloric acid, cover with a watch-glass and heat on a water-bath for 10 minutes. Cool and filter through Whatman filter paper No. 42 or its equivalent. Wash the residue with hot water until the 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 $550^{\circ} \pm 20^{\circ}\text{C}$ for one hour. Cool in a desiccator and weigh. Ignite the dish again for 30 minutes, cool and weigh. Repeat this process till the difference between two successive weighings is less than one milligram. Note the lowest mass.

10.3 Calculation

$$\text{10.3.1 Acid insoluble ash (on moisture-free basis), percent by mass} = \frac{100 (M_2 - M)}{M_1 - M}$$

where

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

M = mass in g of the empty dish, and

M_1 = mass in g of the dish with dried material (see M_1 in 9.2.1).

11. DETERMINATION OF CASTOR HUSK

11.1 Principle — The method is based on the fact that castor husk is not bleached under the conditions which cause the bleaching of almost all other materials of vegetable origin likely to be present in animal feeds or feeding stuffs. The method consists of the treatment of the material with dilute alkali and acid solutions followed by treatment with bleaching powder solution and the isolation of the unbleached castor husk.

11.2 Apparatus

11.2.1 White Photographic Dish

11.3 Reagents

11.3.1 Sodium Hydroxide Solution — 5 percent (*m/v*).

11.3.2 Dilute Hydrochloric Acid — 5 percent (*m/v*)

IS : 7874 (Part I) - 1975

11.3.3 Bleaching Powder Solution — 5 percent (*m/v*), freshly prepared (*see* IS : 295-1951*).

11.4 Procedure — Take three separate 100 g portions of the material and boil for 30 minutes with one litre of the sodium hydroxide solution. Filter through muslin, boil again for 30 minutes with one litre of the dilute hydrochloric acid and filter. Digest the residue for a period depending upon the type of animal feed or feeding stuff with the solution of bleaching powder. When bleaching is complete, filter off the solution. Spread the bleached residue in a thin layer under water in a white photographic dish. Any black pieces are removed and examined microscopically. After identification, the pieces are compared with portions of castor husk which have undergone the above treatment. Castor husk has a characteristic structure, the sharp-angled black pieces of husk showing a distinctive pitted surface, when examined by reflected light under a microscope.

12. DETECTION OF MAHUA CAKE

12.1 Principle — The method is based on the fact that toxic saponins (mowrin) give a typical colour test when extracted.

12.2 Apparatus

12.2.1 Extraction Tube — 150 × 13 mm with a taper tip having an internal diameter 1.5 mm.

12.3 Reagents

12.3.1 Antimony Trichloride Solution — prepared by dissolving 125 g of antimony trichloride in 300 to 400 ml of chloroform. Add 5 g of calcium chloride and filter while hot. Dilute the filtrate to 500 ml with chloroform.

12.3.2 Rectified Spirit — 95 percent (*see* IS : 323-1959†).

12.4 Procedure — Take 10 g of finely ground material in the extraction tube. Tap it to pack it well. Pour rectified spirit into the tube so as to soak the sample. Collect the first drop of the extract on Whatman No. 1 filter paper, about 10 cm in diameter. Dry and then wash by placing 2 to 3 drops of distilled water in the centre of the dried spot. Dry the filter paper completely. Dip the paper in a beaker containing antimony trichloride solution, and then let the paper dry. Heat the paper gently by holding it over a spirit lamp or burner. Care shall be taken as not to over-heat the paper, which shall be evident by its charring. Appearance of a pink colour after heating for five minutes indicates the presence of MAHUA oilcake.

*Specification for bleaching powder, unstabilized.

†Specification for rectified spirit (*revised*).

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones: 331 01 31, 331 13 75

Telegrams: Manaksanstha
(Common to all Offices)

Regional Offices:

| | Telephone |
|---|----------------------------------|
| Central Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002 | { 331 01 31 331 13 75 |
| *Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola, CALCUTTA 700054 | { 36 24 99 |
| Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036 | { 2 18 43 3 16 41 41 24 42 |
| Southern : C. I. T. Campus, MADRAS 600113 | { 41 25 19 41 29 16 |
| †Western : Manakalaya, E9 MIDC, Marol, Andheri (East), BOMBAY 400093 | { 6 32 92 95 |

Branch Offices:

| | |
|--|------------------------|
| 'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMADABAD 380001 | { 2 63 48 2 63 49 |
| ‡Peenya Industrial Area 1st Stage, Bangalore Tumkur Road BANGALORE 560058 | { 38 49 55 38 49 56 |
| Gangotri Complex, 5th Floor, Bhadbhada Road, T. T. Nagar, BHOPAL 462003 | { 6 67 16 |
| Plot No. 82/83, Lewis Road, BHUBANESHWAR 751002 | { 5 36 27 |
| 53/5, Ward No 29, R.G. Barua Road, 5th Byelane, GUWAHATI 781003 | { 3 31 77 |
| 5-8-56C L. N. Gupta Marg (Nampally Station Road), HYDERABAD 500001 | { 23 10 83 |
| R14 Yudhister Marg. C Scheme, JAIPUR 302005 | { 6 34 71 6 98 32 |
| 117/418 B Sarvodaya Nagar, KANPUR 208005 | { 21 68 76 21 82 92 |
| Patliputra Industrial Estate, PATNA 800013 | { 6 23 05 |
| T.C. No. 14/1421, University P.O., Palayam TRIVANDRUM 695035 | { 6 21 04 6 21 17 |

Inspection Offices (With Sale Point):

| | |
|--|-----------|
| Pushpanjali, First Floor, 205-A West High Court Road, Shankar Nagar Square, NAGPUR 440010 | { 2 51 71 |
| Institution of Engineers (India) Building, 1332 Shivaji Nagar, PUNE 411005 | { 5 24 35 |

*Sales Office in Calcutta is at 5 Chowringhee Approach, P. O. Princep 27 68 00
Street, Calcutta 700072

†Sales Office in Bombay is at Novelty Chambers, Grant Road, 89 65 28
Bombay 400007

‡Sales Office in Bangalore is at Unity Building, Narasimharaja Square, 22 36 71
Bangalore 560002

Reprography Unit, BIS, New Delhi, India