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Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

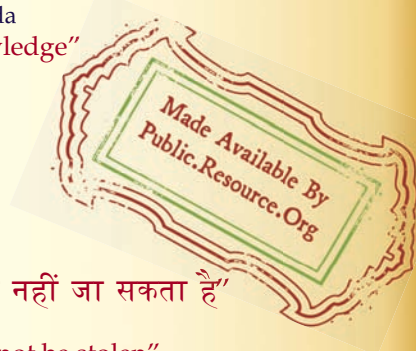
IS 2323 (2011): SPICES AND CONDIMENTS — MUSTARD, WHOLE AND GROUND - Specification [FAD 9: Spices and Condiments]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक
मिर्च एवं मसाले — सरसों, साबुत और पिसी — विशिष्टि
(दूसरा पुनरीक्षण)

Indian Standard
SPICES AND CONDIMENTS — MUSTARD, WHOLE AND
GROUND — SPECIFICATION
(*Second Revision*)

ICS 67.220.10

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

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Spices and Condiments Sectional Committee had been approved by the Food and Agriculture Division Council.

Mustard is one of the important and commonly used spice in the Indian dietary as well as for the production of edible oil. It is marketed as whole dry seeds. It is also used in the production of mustard powder which is obtained by grinding mustard seeds.

Originally two separate standards, IS 2323 and IS 2799 were issued on specification for mustard whole and mustard powder respectively. In the first revision of IS 2323 in 1983, the requirements of mustard powder in IS 2799 were incorporated in IS 2323. Consequently, IS 2799 : 1984 was withdrawn. In this second revision, the requirements have been updated to align with the standards for mustard, whole and ground, laid down under the *Prevention of Food Adulteration Rules*, 1955 and also with the ISO Standard on the subject, ISO 1237 : 1981 'Mustard seed — Specification'. The categorization of mustard, whole, into various grades has been removed and only a single standard has been prescribed for mustard, whole, in this revision.

There are numerous varieties of mustard in the market. Some of the important and well-known types are:

<i>Brassica alba</i> (Linn.) boiss. (<i>Sinapis alba</i> Linn.)	White mustard (<i>SAFED RAI</i>)
<i>Brassica campestris</i> Linn. var. <i>dichotoma</i> (Roxb.) Watt	Brown <i>SARSON</i> , <i>KALI SARSON</i>
<i>Brassica campestris</i> Linn. var. <i>Sarson</i> Prain	Yellow <i>SARSON</i>
<i>Brassica campestris</i> Linn. var. <i>Toria</i> Duthie and Fuller	Indian rape, <i>TORIA</i>
<i>Brassica juncea</i> (Linn.) Coss, et Czern.	Indian mustard, <i>RAI</i>
<i>Brassica nigra</i> (Linn.) W. D. J. Koch	Black mustard, <i>BANARASI RAI</i>

Of the above, the last two types namely, *Brassica juncea* (Linn.) and *Brassica nigra* (Linn.) are mainly used as a condiment. The other types of mustard are used for the production of mustard oil. There is no definite demarcation between the uses of the different varieties of mustard as a spice and for the production of oil. All the varieties marketed as mustard have, therefore, been covered under this standard.

Due consideration has also been given to the *Prevention of Food Adulteration Rules*, 1955 and *Standard of Weights and Measures (Packaged Commodities) Rules*, 1977. However, this standard is subject to restrictions imposed under these rules, 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

SPICES AND CONDIMENTS — MUSTARD, WHOLE AND GROUND — SPECIFICATION

(*Second Revision*)

1 SCOPE

This standard prescribes the requirements and methods of test for mustard, whole and ground for use as spices and condiments.

2 REFERENCES

The following standards 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 indicated below:

<i>IS No.</i>	<i>Title</i>
264 : 2005	Nitric acid — Specification (<i>third revision</i>)
265 : 1993	Hydrochloric acid — Specification (<i>fourth revision</i>)
460 (Part 1) : 1985	Specification for test sieves: Part 1 Wire cloth test sieves (<i>third revision</i>)
915 : 2006	One-mark volumetric flasks — Specification (<i>second revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
1117 : 1975	Specification for one-mark pipettes (<i>first revision</i>)
1797 : 1985	Methods of test for spices and condiments (<i>second revision</i>)
1997 : 1982	Specification for burettes (<i>second revision</i>)
4162 (Part 1) : 1985	Specification for graduated pipettes: Part 1 General requirements
5887 (Part 3) : 1999/ISO	Methods for detection of bacteria responsible for food poisoning: Part 3
6579 : 1993	General guidance on methods for the detection of <i>salmonella</i> (<i>second revision</i>)
7807 : 1975	Methods of test for asafoetida
13145 : 1993	Spices and condiments — Methods of sampling (<i>first revision</i>)
14216 : 1994	Code for hygienic conditions for spices and condiments processing units

3 REQUIREMENTS

3.1 Appearance

The mustard, whole, shall have the shape, size and colour characteristics of the variety of mustard supplied. The seeds shall be mature, hard, sound and reasonably dried. The mustard powder shall be prepared by grinding clean mustard seeds.

3.2 Taste and Flavour

The mustard, whole, shall have the pungency characteristic of the variety of the mustard supplied. The seeds shall be free from musty odour and rancidity.

3.3 Freedom from Moulds, Insects, etc

The seeds shall be free from living insects and shall be practically free from mould growth, dead insects, insect fragments and rodent contamination, visible to the naked eye (corrected, if necessary, for abnormal vision), or using the required magnifying instrument. If the magnification exceeds $\times 10$, this fact shall be mentioned in the test report. The proportion of insect damaged matter shall not exceed 1 percent (*m/m*).

3.4 Freedom from Argemone Seeds

The mustard, whole, shall be free from argemone seeds when tested in accordance with the method prescribed in Annex A.

3.5 Extraneous Matter, Shrivelled, and Slightly Damaged Seeds

The seeds shall be whole and mature and shall not contain more than 0.7 percent (*m/m*) of extraneous matter, which includes dust, dirt, stones, lumps of earth, chaff, stem or straw, food grains including oilseeds of any other kind or any other impurity when determined in accordance with the method given in 4 of IS 1797. The proportion of damaged or shrivelled mustard seeds shall not exceed 2 percent (*m/m*).

3.6 Fineness

The mustard powder shall be ground to such a fineness that all of it passes through 600-micron IS Sieve [see IS 460 (Part 1)] and nothing remains on the sieve.

3.7 The mustard, whole and ground shall also comply with the requirements given in Table 1.

3.8 Hygienic Conditions

The mustard, whole and ground shall be processed and packed under hygienic conditions (*see* IS 14216).

3.9 Pesticide residues and metallic contaminants in the product shall not exceed the limits as prescribed in the *Prevention of Food Adulteration Act, 1954* and the Rules made thereunder.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in clean, sound and dry container made of metal, glass, food grade plastics, wood or jute bags. The wooden boxes or jute bags shall be suitably lined with moisture proof lining which shall not impart any foreign smell to the product. The packing material shall be free from any fungal or insect infestation and should not impart any foreign smell. Each container shall be securely closed and sealed.

4.2 Marking

The following particulars shall be marked directly on the container/package or a label affixed on it:

- a) Name and address of the manufacturer or packer;

- b) Name of the material (whole or ground);
- c) Trade name or brand name, if any;
- d) Batch or Code number;
- e) Net mass when packed;
- f) Best before (month and year); and
- g) Any other markings required under the *Standards of Weights and Measures (Packaged Commodities) Rules, 1977* and the *Prevention of Food Adulteration Rules, 1955*.

5 SAMPLING

Representative samples of the material shall be drawn and tested for conformity to this standard as prescribed in IS 13145.

6 METHODS OF TEST

The samples of mustard, whole and ground shall be tested for ascertaining conformity of the material to the requirements in accordance with the relevant clauses given in col 5 of Table 1.

7 QUALITY OF REAGENTS

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

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

Table 1 Requirements for Mustard, Whole and Ground
(Clause 3.7)

Sl No.	Characteristics	Requirements		Method of Test, Ref to	
		Whole (3)	Ground (4)	Clause of IS (5)	Annex (6)
(1)	(2)				
i)	Moisture, percent by mass, <i>Max</i>	10.0	7.0	9 of IS 1797	—
ii)	Non-volatile ether extract, percent by mass, <i>Min</i>	28.0	28.0	14 of IS 1797	—
iii)	Total ash, percent by mass, <i>Max</i>	6.5	6.5	6 of IS 1797	—
iv)	Acid insoluble ash, percent by mass, <i>Max</i>	1.0	1.0	8 of IS 1797	—
v)	Starch, percent by mass, <i>Max</i>	—	2.5	8 of IS 7807	—
vi)	Crude fibre, percent by mass, <i>Max</i>	—	8.0	13 of IS 1797	—
vii)	Volatile oil content on dry basis, percent by mass, <i>Max</i>	0.3	0.3	15 of IS 1797	—
viii)	Allyl thiocyanate, percent by mass, <i>Min</i>				B
	a) <i>B. nigra</i>	1.0	—	—	
	b) <i>B. juncea</i>	0.70	—	—	
ix)	p-hydroxy benzyl isothiocyanate, percent by mass, on dry basis in <i>Sinapis alba</i> , <i>Min</i>	2.3	—	—	C or D
x)	<i>Salmonella</i> (in 25 g)	Absent	Absent	IS 5887 (Part 3)	—
xi)	Test for argemone oil	—	Negative	—	—

ANNEX A

(Clause 3.4)

TEST FOR THE PRESENCE OF ARGEMONE SEEDS

A-1 REAGENTS

A-1.1 Concentrated Hydrochloric Acid, sp gr 1.19 (see IS 265).

A-1.2 Concentrated Nitric Acid, see IS 264.

A-1.3 Dragen Dorff's Reagents, shall be prepared as follows:

Solution A = Bismuth subnitrate: 800 mg,
glacial acetic acid: 10 ml, and
water: 40 ml

Solution B = Potassium iodine: 8 g, and water:
20 ml

Stock solution = Solutions A + B (equal volume)

Dilute solution = Stock solution + acetic acid +
water (1 + 2 + 10)

A-2 PROCEDURE

A-2.1 Place the sample in between the two halves of

a half folded Whatman No. 1 filter paper and press the seed by means of a paper weight so that it bursts with a sound, leaving two stains of oil on the two sides of the filter paper. Soak one oil spot with concentrated hydrochloric acid and test with Dragendorff's reagent and test the second spot with concentrated nitric acid.

A-2.2 Test Result

If the spot, tested with Dragendorff's reagent shows an orange red colour and the spot tested with nitric acid shows an orange yellow to crimson colour argemone seeds are present. The positive results in both the tests performed simultaneously is conformity for the presence of argemone seeds.

A-2.2.1 If there is no positive reaction, the mustard seeds shall be deemed to be free from argemone seeds.

ANNEX B

[Table 1, Sl No. (viii)]

DETERMINATION OF ALLYL ISOTHIOCYANATE

B-1 PRINCIPLE

After two successive soakings of the sample, the first in water at a temperature of 70°C and the second in alcoholic medium, distillation of the allyl isothiocyanate liberated into an alcoholic ammonium hydroxide solution, addition to the distillate of a standard volumetric silver nitrate solution, and titration of the excess silver nitrate with standard volumetric potassium, or ammonium, thiocyanate solution in the presence of ammonium iron (III) sulphate.

B-2 REAGENTS

All reagents shall be of recognized analytical quality. The water used shall be distilled water or water of at

least equivalent purity.

B-2.1 Ethanol, 95 percent (v/v).

B-2.2 Ammonium Hydroxide Solution, $\rho_{20} = 0.925$ g/ml.

B-2.3 Nitric Acid, $\rho_{20} = 1.40$ g/ml.

B-2.4 Silver Nitrate, standard volumetric solution, $c(\text{AgNO}_3) = 0.1$ mol/l.

B-2.5 Potassium Thiocyanate or Ammonium Thiocyanate, standard volumetric solution, $c(\text{KCNS})$ or $c(\text{NH}_4\text{CNS}) = 0.1$ mol/l.

B-2.6 Ammonium Iron (III) Sulphate Solution, saturated when cold.

B-3 APPARATUS

Usual laboratory apparatus, and in particular the following.

B-3.1 Grinding Mill

B-3.2 Entrainment Distillation Apparatus (*see* Fig. 1 for a suitable example).

B-3.3 Burette, graduated at 0.05 ml intervals, complying with the requirements of IS 1997, Class A.

B-3.4 Analytical Balance

B-4 PROCEDURE

B-4.1 Preparation of the Sample

After very careful mixing of the sample, take 15 to 20 g and grind it.

B-4.2 Test Portion

Take about 2 g of the ground sample and weigh it to the nearest 0.001 g.

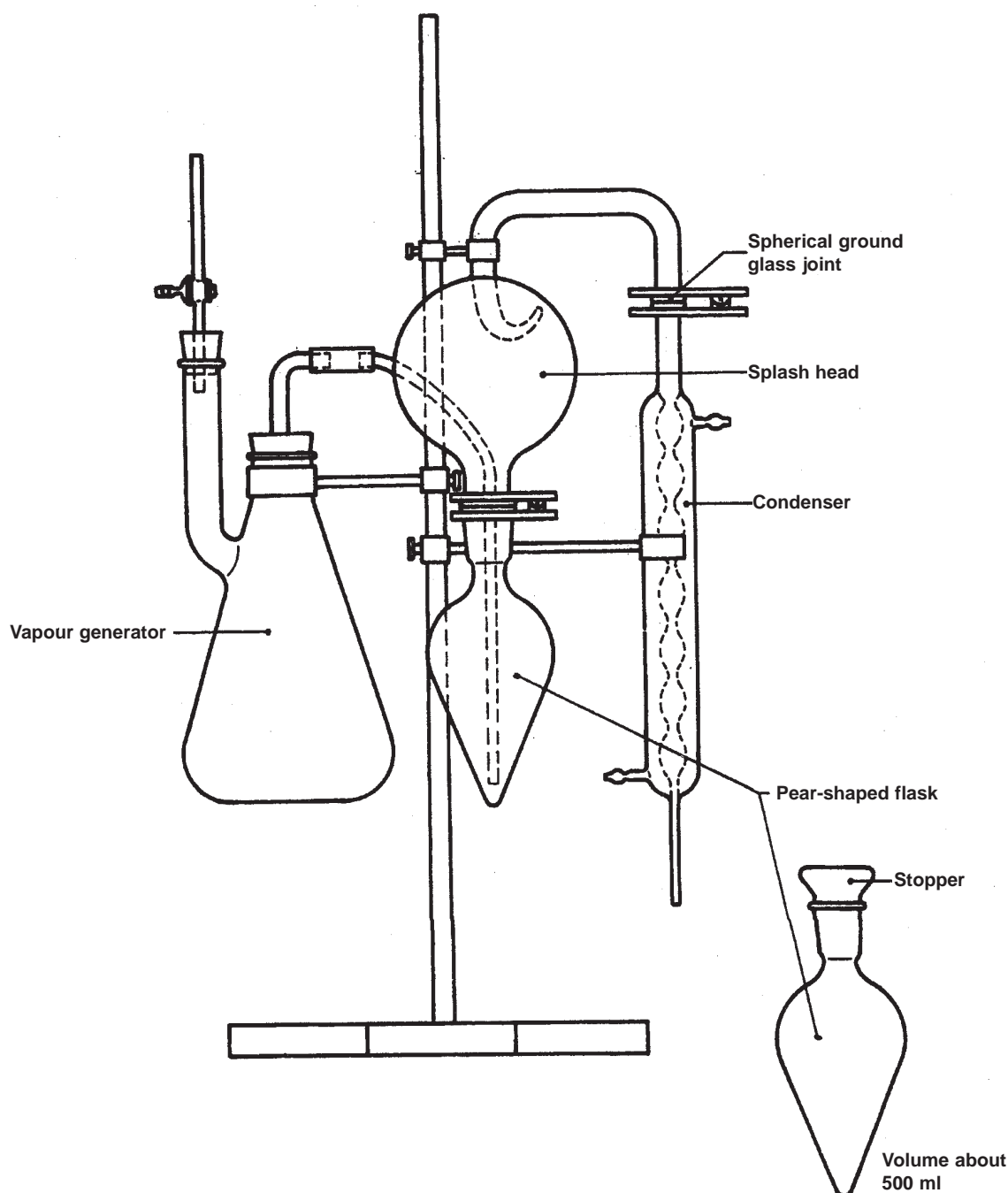


FIG. 1 ENTRAINMENT DISTILLATION APPARATUS

B-4.3 Determination

Transfer the test portion to the pear-shaped flask of the distillation apparatus, add 80 ml of water previously heated to $70 \pm 20^\circ\text{C}$, close the flask with its ground glass stopper and leave to stand for 15 min. Then add 20 ml of the ethanol (*see B-2.1*) and allow to soak for 45 min.

After the soaking, connect the flask quickly to the distillation apparatus. Distil, and collect the distillate in a conical flask containing a mixture of 5 ml of the ammonium hydroxide solution (*see B-2.2*) and 10 ml of the ethanol (*see B-2.1*). (Entrainment distillation lasts, on average, for 5 min) The quantity of the distillate should be at least 100 ml.

Add to the distillate 10 ml of the standard volumetric silver nitrate solution (*see B-2.4*) and leave for 12 h at ambient temperature (the operation will be faster, if the conical flask is placed for 1 h in a water bath heated to 70 to 80°C).

Filter through a fine filter paper, rinse the flask and residue several times with hot water (approximately 90°C).

To the bulked filtrate and washings add 10 ml of the nitric acid (*see B-2.3*) and then titrate with the standard volumetric potassium, or ammonium, thiocyanate solution (*see B-2.5*) using the ammonium iron(III) sulphate solution (*see B-2.6*) as indicator, until a persistent pink colour is obtained.

B-4.4 Number of Determinations

Carry out two determinations on the same prepared sample.

B-5 EXPRESSION OF RESULTS

B-5.1 Method of Calculation and Formula

Allyl isothiocyanate
content, expressed
as a percentage by
mass on dry basis $= \frac{4.95(10 - A)}{10^3} \times \frac{100}{m} \times \frac{100}{100 - H}$

where

m = mass of the test portion, in g;

V = volume of the standard volumetric

potassium, or ammonium, thiocyanate solution used in the titration, in ml; and

H = moisture content of the sample, expressed as a percentage by mass, determined by the method specified in Annex A.

Take as the result the arithmetic mean of the two determinations (*see B-4.4*), provided that the requirement for repeatability (*see B-5.2*) is satisfied.

NOTE — If the standard volumetric solutions used are not of the exact concentrations indicated in B-2, a suitable correction factor should be used in calculating the result.

B-5.2 Repeatability

The difference between the results of the two determinations (*see B-4.4*), carried out simultaneously or in rapid succession by the same analyst, shall not exceed 1 percent of the mean value.

B-6 NOTES ON PROCEDURE

B-6.1 During the analysis, all contact with copper or rubber shall be avoided, especially in the distillation apparatus. Use cork or, preferably, ground glass stoppers.

B-6.2 The enzymic activity of the mustard seed diminishes with age; thus, it may be necessary to modify the analytical method in the case of old seed.

After a preliminary determination giving particularly low figures for allyl isothiocyanate, add to the distillation residue 5 g of *Sinapis alba* (take care to check that sulphur-containing volatile substances are not present in it) and then proceed to a second determination.

The addition of the two results will give the real figure of allyl isothiocyanate that may be formed in the sample. However, the actual quality of the sample should be considered as very reduced and it is recommended that the two figures, found before and after the addition of *Sinapis alba*, should be given in the test report.

B-6.3 The enzymic activity of the seeds increases during certain periods of the year (particularly in spring); thus, identical results are not always found with a given lot of seed, according to the season in which the analysis is carried out.

ANNEX C

[Table 1, Sl No. (ix)]

**DETERMINATION OF p-HYDROXYBENZYL ISOTHIOCYANATE
(COLORIMETRIC METHOD)**

C-1 PRINCIPLE

Decomposition, by enzymatic hydrolysis, of the sinalbin (glucoside of *Sinapis alba*) into glucose, the hydrogen sulphate of sinapin and p-hydroxybenzyl isothiocyanate, the last mentioned giving p-hydroxybenzyl and thiocyanate. Colorimetric determination of the thiocyanate so formed.

C-2 REAGENTS

All reagents shall be of recognized analytical quality. The water used shall be distilled water or water of at least equivalent purity.

C-2.1 Calcium Carbonate, pulverized.

C-2.2 Mercury (II) Chloride, 50 g/l solution.

C-2.3 Potassium Hexacyanoferrate (II), 106 g/l solution.

C-2.4 Zinc Acetate Solution — Dissolve 21.9 g of zinc acetate $[(CH_3COO)_2Zn]$ in water, add 3 ml of glacial acetic acid (CH_3COOH) and dilute to 100 ml with water.

C-2.5 Nitric Acid, approximately 1 mol/l solution.

C-2.6 Sodium Hydroxide, approximately 1 mol/l solution.

C-2.7 Ammonium Iron (III) Sulphate, 200 g/l solution in approximately 0.5 mol/l sulphuric acid solution.

C-2.8 Potassium Thiocyanate or Ammonium Thiocyanate, standard volumetric solution, $c(KCNS)$ or $c(NH_4CNS) = 0.1$ mol/l, that is containing 5.808 g of CNS^- — per litre.

C-3 APPARATUS

Usual laboratory apparatus, and in particular the following:

C-3.1 Grinding Mill

C-3.2 One-Mark Volumetric Flasks, of capacities 50, 250 and 1 000 ml, complying with the requirements of IS 915, class A.

C-3.3 Pipettes, delivering 2 ml and 5 ml, complying with the requirements of IS 1117, class A or IS 4162 (Part 1).

C-3.4 Colorimeter, suitable for measurements at a wavelength of 450 nm.

C-3.5 Analytical Balance**C-4 PROCEDURE****C-4.1 Preparation of Test Sample**

Carefully render the sample homogeneous, then take a portion of 20 to 25 g of the mustard seed and grind it.

C-4.2 Test Portion

Take approximately 5 g of the ground sample and weigh it to the nearest 0.001 g.

C-4.3 Hydrolysis

Transfer the test portion to a 250 ml beaker. Add 100 ml of water at $70 \pm 2^\circ C$ and at least 100 mg of the calcium carbonate (*see C-2.1*). Cover the beaker with a watch glass. Leave to soak for 15 min at $70^\circ C$, cool, add 20 ml of the sodium hydroxide solution (*see C-2.6*), and leave in contact for 15 min.

C-4.4 Clarification

Add a sufficient quantity of the nitric acid solution (*see C-2.5*), to bring the contents of the beaker to a pH of about 6.0 to 6.5. Pour the contents of the beaker into a 250 ml volumetric flask and, shaking the flask, add 2 ml of the potassium hexacyanoferrate(II) solution (*see C-2.3*) and then 2 ml of the zinc acetate solution (*see C-2.4*).

Dilute to 250 ml with water and add 2 ml of water by pipette (*see C-3.3*) (to take into account the volume of the precipitate). Shake, and filter through a rapid filter shaded from bright light. The filtrate (*F*) should be clear and colourless.

C-4.5 Determination

Add to a 50 ml volumetric flask,

- a) 5 ml of the filtrate (*F*); and
- b) 5 ml of the ammonium iron (III) sulphate solution (*see C-2.7*).

Dilute to 50 ml with water, shake, and measure the absorbance at a wavelength of 450 nm by means of the colorimeter (*see C-3.4*).

C-4.6 Calibration Curve

Transfer, by means of a pipette (*see C-3.3*) 5 ml of the standard volumetric potassium, or ammonium,

thiocyanate solution (*see* C-2.8) to a 1 000 ml volumetric flask and dilute to the mark with water.

Into a series of five 50 ml volumetric flasks, transfer the volumes of this diluted potassium, or ammonium, thiocyanate solution indicated in the following table:

<i>Volume of Diluted Potassium, or Ammonium, Thiocyanate Solution</i> ml	<i>Corresponding Mass of Thiocyanate Ion</i> µg
5	145.2
10	290.4
15	435.6
20	580.8
25	726

Add to each flask, 5 ml of the ammonium iron (III) sulphate solution (*see* C-2.7), dilute to the mark with water, shake, and measure the absorbance as indicated in C-4.5.

Plot a calibration curve, giving the absorbance as a function of the number of micrograms of thiocyanate.

C-4.7 Matching Test

Carry out a matching test in the same conditions as the actual test, but adding 2 drops of the mercury (II) chloride solution (*see* C-2.2) to correct for errors due to the reaction of phenols with the iron (III) salts.

Note on the calibration curve the difference in absorbance between the test solution, containing the thiocyanate and phenols, and the matching test solution.

C-4.8 Number of Determinations

Carry out two determinations on the same prepared sample.

C-5 EXPRESSION OF RESULTS

C-5.1 Method of Calculation and Formula

p-hydroxybenzyl
isothiocyanate

content, expressed

as a percentage by mass on dry basis $= 2.85 \frac{m_1}{10^6} \times \frac{250}{5} \times \frac{100}{m_0} \times \frac{100}{100-H}$

where

m_0 = mass of the test portion, in g;

m_1 = mass of thiocyanate read from the calibration curve, in µg; and

H = moisture content of the sample, expressed as a percentage by mass, determined by the method specified in Annex A.

2.84 is the conversion factor from thiocyanate ion (CNS⁻) to p-hydroxybenzyl isothiocyanate.

Take as the result the arithmetic mean of the two determinations (*see* C-4.8), provided that the requirement for repeatability (*see* C-5.2) is satisfied.

C-5.2 Repeatability

The difference between the results of the two determinations (*see* C-4.8), carried out simultaneously or in rapid succession by the same analyst, shall not exceed 2 percent of the mean value.

C-6 NOTE ON PROCEDURE

The enzymic activity of the seeds increases during certain periods of the year (particularly in spring); thus, identical results are not always found with a given lot of seed, according to the season in which the analysis is carried out.

ANNEX D

[Table 1, Sl No. (ix)]

DETERMINATION OF p-HYDROXYBENZYL ISOTHIOCYANATE (ARGENTIMETRIC METHOD)

D-0 INTRODUCTION

Laboratories not in possession of a colorimeter may determine thiocyanate by argentimetry. In this case it is necessary either:

- to carry out a preliminary check for the absence of Cl⁻ ions in the seed (no reaction

with silver nitrate on the ash of the mustard seed); or

- to provide for correction by carrying out the determination of Cl⁻ ions on an aliquot portion of the filtrate.

This method may be substituted for the colorimetric method by agreement between the parties concerned.

D-1 PRINCIPLE

Decomposition, by enzymic hydrolysis, of sinalbin (glucoside of *Sinapis alba*) into glucose, the hydrogen sulphate of sinapin and p-hydroxybenzyl isothiocyanate, the last-mentioned giving p-hydroxybenzyl alcohol and thiocyanate. Determination of the thiocyanate thus formed, by argentimetry in nitric acid medium; back titration of the excess of silver nitrate using standard volumetric potassium thiocyanate solution in the presence of ammonium iron (III) sulphate.

D-2 REAGENTS

All reagents shall be of recognized analytical quality. The water used shall be distilled water or water of at least equivalent purity.

The reagents necessary for hydrolysis and clarification (*see* Annex C), together with the following:

D-2.1 Nitric Acid, $\rho_{20} = 1.40$ g/ml.

D-2.2 Silver Nitrate, standard volumetric solution, $c(\text{AgNO}_3) = 0.1$ mol/l.

D-2.3 Potassium Thiocyanate, standard volumetric solution, $c(\text{KCNS}) = 0.1$ mol/l.

D-2.4 Ammonium Iron (III) Sulphate Solution, saturated when cold.

D-3 APPARATUS

The apparatus necessary for the preparation of the sample, hydrolysis and clarification (*see* Annex C), together with the following:

D.3.1 One-Mark Pipettes, of capacities 5 ml and 100 ml, complying with the requirements of IS 1117, class A.

D.3.2 Burette, graduated at every 0.05 ml, complying with the requirements of IS 1997, class A.

D-4 PROCEDURE**D.4.1 Preparation of Test Sample, Test Portion, Hydrolysis and Clarification**

Proceed as specified in C-4.1 to C-4.4 of Annex C.

D-4.2 Titration

Add to the beaker, shaking after each addition of following:

- 100 ml, by means of a pipette (*see* D-3.1), of the filtrate (F) (*see* C-4.4), and approximately equivalent to 2 g of mustard seed;
- 1 ml of the nitric acid (*see* D-2.1);
- 5 ml, by means of a pipette (*see* D-3.1), of the standard volumetric silver nitrate solution (*see* D-2.2); and
- 2 ml of the ammonium iron (III) sulphate solution (*see* D-2.4).

Shake the flask to coagulate the precipitate, and titrate with the potassium thiocyanate solution (*see* D-2.3) until a persistent faint red colour is obtained.

D-4.3 Number of Determinations

Carry out two determinations on the same prepared sample.

D-5 EXPRESSION OF RESULTS**D-5.1 Method of Calculation and Formula**

p-hydroxybenzyl isothiocyanate content, expressed as a percentage by mass on the dry basis =

$$0.0165(5-V) \frac{250}{100} \times \frac{100}{m} \times \frac{100}{100-H}$$

where

m = mass of the test portion (*see* C-4.2), in g;

V = volume of the standard volumetric potassium thiocyanate solution (*see* D-2.3) used in the titration, in ml; and

H = moisture content of the sample, expressed as a percentage by mass, determined by the method specified in Annex A.

NOTE — If the standard volumetric solutions used are not of the exact concentrations specified in D-2, a suitable correction factor should be used in calculating the result.

Take as the result the arithmetic mean of the two determinations (*see* D-4.3), provided that the requirement for repeatability (*see* D-5.2) is satisfied.

D-5.2 Repeatability

The difference between the results of the two determinations (*see* D-4.3) carried out simultaneously or in rapid succession by the same analyst, shall not exceed 0.1 g of p-hydroxybenzyl isothiocyanate per 100 g of dry matter in the sample.

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