Indian Standard

METHOD OF SAMPLING FOR THE DETERMINATION OF PESTICIDE RESIDUES

PART 1 AGRICULTURAL AND FOOD COMMODITIES

(First Reprint OCTOBER 1997)

UDC 664 : 543 (632.951.028)

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

Gr 3

July 1986

Indian Standard

METHOD OF SAMPLING FOR THE DETERMINATION OF PESTICIDE RESIDUES

PART I AGRICULTURAL AND FOOD COMMODITIES

Sampling Methods for Food Products and Agricultural Inputs Sectional Committee, AFDC 57

Chairman	*Representing*
PROF PREM NARAIN	Indian Agricultural Statistics Research Institute (ICAR), New Delhi
Members	
SHRI S. C. RAI (*Alternate* to Prof Prem Narain)	
COL K. N. ACHARYA	Quartermaster General's Branch, Army Headquarters, New Delhi
LT-COL S. K. BHATTACHARYA (*Alternate*)	
AGRICULTURAL MARKETING ADVISER TO THE GOVERNMENT OF INDIA	Directorate of Marketing and Inspection (Ministry of Rural Reconstruction), Faridabad
SHRI S. K. SHARMA (*Alternate*)	
SHRI R. C. ARORA	Directorate of Economics and Statistics (Ministry of Agriculture), New Delhi
SHRI G. S. AURORA	Federation of Biscuit Manufacturers of India,, Delhi
SHRI D. S. CHADHA	Central Committee for Food Standards (Ministry of Health and Family Welfare), New Delhi
SMT DEBI MUKHERJEE (*Alternate*)	
SHRI P. R. DONGRE	Central Statistical Organization, New Delhi
DR D. R. GHODEKAR	National Dairy Research Institute, Karnal
SHRI SITA RAM SWAMI (*Alternate*)	
SHRI RATAN GUPTA	Roller Flour Millers' Federation of India, New Delhi
SHRI V. K. VENKATARAMAN (*Alternate*)	
DR R. C. GUPTA	Directorate of Plant Protection, 'Quarantine and Storage (Ministry of Agriculture), Faridabad
SHRI R. S. SHARMA (*Alternate*)	

(Continued on page 2)

(*Continued from page* 1)

Members	*Representing*
DR E. K. JAYANARAYANAN	Mohan Meakin Ltd, Mohan Nagar
SHRI JITENDRA MOHAN (*Alternate*)	
DR D. C. JOHRI	Central Avian Research Institute, Izatnagar
SHRI SIJSHIL KUMAR (*Alternate*)	
SHRI M.N. KHAN	Department of Dairy Development (Ministry of Agriculture), New Delhi
DEPUTY COMMISSIONER (DAIRY PRODUCTS) (*Alternate*)	
SHRI K. N. NAMBOODRI	Tea Board, Calcutta
SHRI R. N. MONDAL (*Alternate*)	
SHRI K. R. NARAYANA RAO	Food Corporation of India, New Delhi
SHRI T. N. RAMACHANDRA RAO (Alternate)	
PROF OF SUGAR CHEMISTRY	National Sugar Institute, Kanpur
KUMARI D. RAJALAKSHMI	Central Food Technological Research Institute (CSIR), Mysore
SHRI B. S. RAMFSH (*Alternate*)	
DR K. G. RAMAMURTHY	Indian Statistical Institute. Calcutta
SHRI A. N. NANKANA (*Alternate*)	
SHRI KISHORE KUMAR SHAMJI	India Pepper and Spice Trade Association, Cochin
SHRI MANIKANT V. KHONA (*Alternate*)	
SHRI G. D. SHARMA	Ministry of Food and Civil Supplies (Department of Food)
DR B. K. NANDI (*Alternate*)	
DR L. N. SINGH	Indian Veterinary Research Institute (ICAR), Izatnagar
DR N. SHARMA (*Alternate*)	
SHRI G. W. DATEY, Director (Statistics)	Director General, BIS (*Ex-officio Member*)

Secretary

SHRI A. K. TALWAR
Joint Director (Statistics), BIS

Panel on the Method of Sampling for Pesticide Residues Analysis, AFDC 57 : P-4

Convener

DR N. P. AGNIHOTRI	Indian Agricultural Research Institute (ICAR), New Delhi

Members

DR R. P. CHAWLA	Punjab Agricultural University, Ludhiana
DR (SMT) G. MUKHERJEE	Central Insecticides Laboratory, Faridabad
DR P. K. RAY	Industrial Toxicology Research Centre (CSIR), Lucknow

Indian Standard

METHOD OF SAMPLING FOR THE DETERMINATION OF PESTICIDE RESIDUES

PART I AGRICULTURAL AND FOOD COMMODITIES

0. FOREWORD

0.1 This Indian Standard (Part 1) was adopted by the Indian Standards Institution on 26 November 1985, after the draft finalized by the Sampling Methods for Food Products and Agricultural Inputs Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 For the examination of a lot in order to decide whether it complies with maximum 'limits for pesticide residues, it is necessary to provide a representative sample for analysis. The objective of this sampling procedure is to obtain a final sample representative of the lot in order to determine its average pesticide residue content in agricultural and food commodities.

0.3 The sites, techniques, frequency of sampling and the size and number of samples must allow the analytical results to be statistically evaluated.

0.4 Whereas this standard (Part 1) covers the method of drawing, preparing and storing the samples of agricultural and food commodities for the determination of pesticide residues, the method of sampling in respect of environment (air, water and soil) for the same purpose will be covered in Part 2.

0.5 In the preparation of this standard, assistance has been derived from Codex document 'Recommended Method of Sampling for the Determination of Pesticide Residues', CAC/Vol XIII-First Ed. 3 1 October 1983.

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

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard (Part 1) prescribes the procedures for drawing, preparing and storing the samples for the examination of a lot of agricultural and food commodities with respect to pesticide residues analysis.

2. TERMINOLOGY

2.1 Lot-An identifiable quantity of goods delivered at one time, having or presumed to have common properties or uniform characteristics such as the same origin, the same variety, the same consignor and the same type of packing. Several lots may make up a consignment.

2.2 **Consignment** — A quantity of material covered by a particular consignment note or shipping document. Lots in the same consignment may be delivered at different times and may have different amounts of pesticide residues.

2.3 **Primary Sample-A** quantity of material taken from a single place in the lot.

2.4 Bulk Sample — Combined total of all the primary samples taken from the same lot.

2.5 **Final Sample** — Bulk sample or representative part of the bulk sample to be used for control purposes.

2.6 **Laboratory Sample** — Sample submitted to the laboratory for testing. The final sample may be used as a whole or subdivided into representative portions (laboratory sample), if required.

2.7 **Test Sample -The** sample as prepared by the laboratory for testing after suitable reduction of the laboratory sample, if required.

3. GENERAL REQUIREMENTS OF SAMPLING

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

3.1 In the course of taking the primary samples and in all subsequent procedures, precautions must be taken to avoid contamination of the samples or any other changes which would adversely affect the amount of residues or the analytical determinations or make a laboratory sample not representative of bulk sample.

3.1.1 Diseased or under-sized commodities may also be avoided.

3.2 As many pesticides are photo-degradable, it is advisable to protect samples and any solution or extracts from needless exposure to light.

3.3 In the case of solid/dry products, plastic/polyethylene/kraft paper bags shall be used and for liquid/wet products, glass jars or bottles with alumiuium foil or metal teflon-lined containers shall be used.

NOTE — Polyethylene bags must be put in additional heavy paper bags, as polyethylene bags alone may become brittle in contact with dry ice if kept frozen.

3.4 It must be ensured that glass containers used are entirely free from contamination. The operation for cleaning of glass containers generally includes:

a) Soaking and washing in a high temperature (50°C) bath or synthetic detergent in water;

b) Rinsing with tap water;

c) Rinsing with distilled water; and

d) Rinsing with distilled acetone.

3.4.1 Cleaning of glass containers used to concentrate samples (evaporative concentrated tubes) should include a soak for at least 15 min in hot (40 to 50" C) chromic acid cleaning solution (observe rigid safety precautions) after the tap water rinse to remove all traces of organic material. This soak is followed by thorough rinsing with tap and distilled water and then with acetone and hexane. Pipettes are washed in the same way.

3.4.2 As an extra precaution, each piece of glass containers should be rinsed with the solvent to be employed in the analysis immediately before use. As soon as possible after a piece of glass containers has come in contact with a sample containing pesticides, it should be mixed with acetone to remove surface residues.

3.5 Aluminium foil can be cleaned by agitating it in analytical reagent grade acetone followed by safe rinsing with pesticide grade ethyl acetate and hexane.

3.6 Samples should be analysed as soon as possible after their collection. If storage of sample before analysis is necessary, it should be under prescribed conditions which preserve the integrity of the original sample.

3.7 Agricultural or environmental samples that are to be analysed for organophosphates should be placed in tight containers and stored in deep freezing as soon as possible after sampling, unless sample preparation is to be conducted within a few hours.

3.8 If lengthy storage is required prior to analysis, a good alternative to the storage of sample is to extract the sample at once, remove most or all the solvent, and store the extract at a low temperature. Decomposition in samples that must be stored can be evaluated by storing spiked controls along with the samples.

3.9 It may be ensured that a sample after drawing should reach the laboratory for analysis within 24 h and should be stored in a freeze below 0°C.

4. SAMPLING PROCEDURE

4.1 Each lot (see 2.1) which is to be examined must be sampled separately.

4.2 **Primary Samples**

4.2.1 The primary samples should be taken throughout the lot. Departures from this requirement must be separately recorded.

4.2.2 The primary samples should be of similar size and the combined total of all the primary samples (bulk sample) must not be less than that required for the final sample, bearing in mind the possible requirement of further reduction and the provision of adequate laboratory samples.

4.2.3 The minimum number of primary samples to be taken shall be according to Table 1.

TABLE 1 SCALE OF SAMPLING

WEIGH.1 OF LOT (kg)	MINIMUM NUMBER OF PRIMARY SAMPLES TO BE TAKEN
(1)	(2)
up to 50	3
51 to 500	5
501 to 2000	10
2001 and above	15

NOTE — When the material is supplied in bulk, for bigger size food grains, smaller size food grains and milled cereals and pulses products, the alternative sampling procedures given in the relevant Indian standard may be used provided this departure is recorded and the minimum requirements given in **4.5.1** are met.

4.2.4 For processed products in cans, bottles, packages or other small containers, especially when the mass of the lot is not known, the minimum number of primary samples to be taken from the lot shall be according to Table 2.

TABLE 2 MINIMUM NUMBER OF PRIMARY SAMPLES FROM A LOT CONTAINING PACKAGES

Number of Cans, Packages or Containers in the Lot	Minimum Number of Primary Samples to be Taken
(1)	(2)
up to 25	1
26 to 100	5
101 to 250	10
251 and above	15

4.2.5 For homogeneous lots. a sample fully representative of the whole is obtained by withdrawing any single sample.

4.3 Preparation of Bulk Sample

4.3.1 The bulk sample shall be made by combining and mixing the primary samples.

4.4 Preparation of Final Sample

4.4.1 The bulk sample should, if possible, constitute the final sample.

4.4.2 If the bulk sample is too large, the final sample may be prepared from it by a suitable method of reduction. In the process of reduction, however, individual fruits and vegetables must not be cut or divided.

4.5 Preparation of the Laboratory Sample

4.5.1 The minimum amount of material to be submitted to the laboratory that is the size of the laboratory sample shall be as follows:

Commodity	*Exarnple*	*Minimum Quantity Required*
(1)	(2)	(3)
Small or light products, unit mass up to about 25 g	barries peas	1 kg
Medium sized products, unit mass usually between 25 and 250 g	apples oranges carrots potatoes	1 kg (at least 10 units)
Large sized products, unit mass over 250 g	cabbages melons cucumbers	2 kg (at least 5 units)
Dairy products	milk cheese butter cream	0·5 kg
Poultry products	eggs	0·5 kg (10 units if whole)
Animal tissue	meat, chicken, fish and their products	0·5 kg
Oils and fats	cottonseed oil	0·5 kg
Spices	cumin, coriander	0·25 kg
Cereals and cereal products	wheat, rice	1 kg
Forage	grass, straw	1 kg
Animal feeds	husk, bran, oil cakes	1 kg
Beverage products	coffee, tea, cocoa	0·25 kg

4.5.1.1 A test sample may be obtained by reducing a laboratory sample by a suitable method of reduction. A typical example is given in Fig. 1. Out of the three test samples each of 80 g finally obtained, two may be used for analysis and one may be kept as a referee sample.

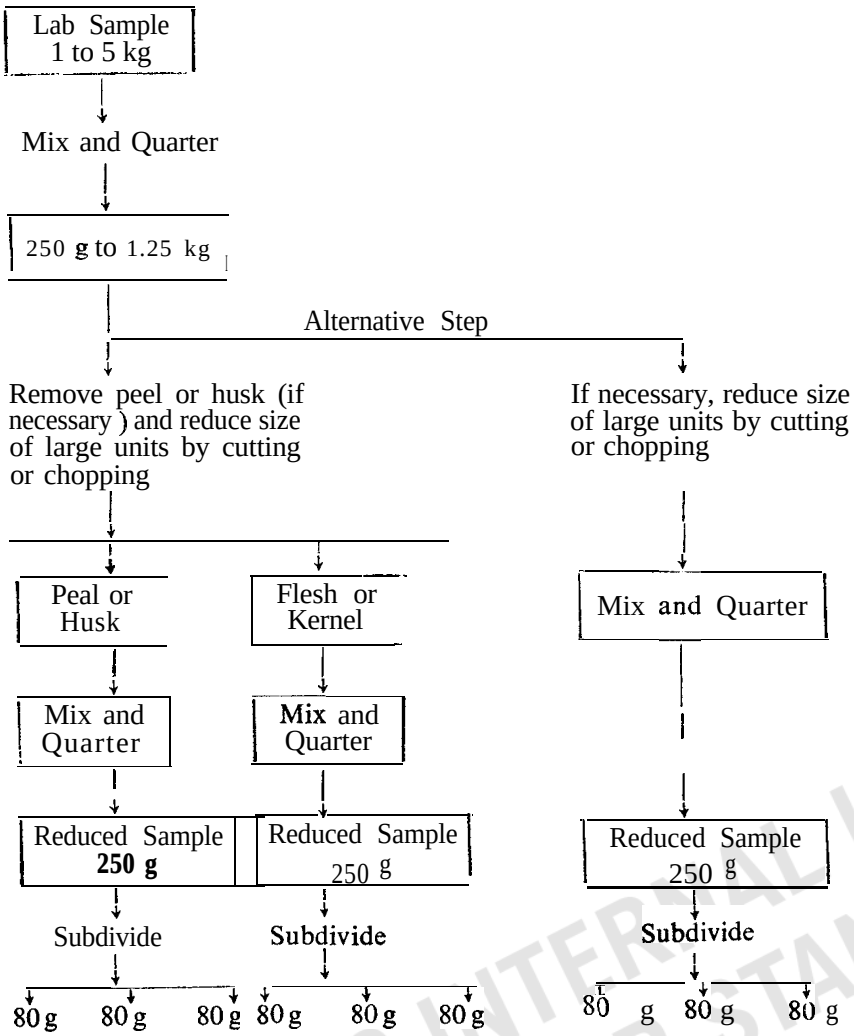

FIG. 1 TYPICAL PROCEDURE FOR REDUCTION OF A LABORATORY SAMPLE

5. PACKAGING AND TRANSPORTATION OF LABORATORY SAMPLES

5.1 The laboratory sample must be placed in a clean inert container offering adequate protection from external contamination and protection against damage or deteriorations of the sample in transit. The container must then be sealed in such a manner that unauthorised opening is detectable, and sent to the laboratory as soon as possible, taking necessary precautions against leakage or spoilage, for example frozen foods should be kept frozen and perishable samples should be kept cooled or frozen.

5.2 Each laboratory sample must be correctly identified and should be accompanied by a note giving the nature and origin of the sample and the date and place of sampling together with any additional information likely to be of assistance to the analyst.

BUREAU OF INDIAN STANDARDS

Headquarters
Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002
Telephones: 323 0131,323 3375,323 9402
Fax : 91 11 3234062, 91 11 3239399, 91 11 3239362

Telegrams : Manaksanstha
(Common to all Offices)

	Telephone
Central Laboratory :	
Plot No. 20/9, Site IV, Sahibabad Industrial Area, Sahibabad 201010	6-77 00 32
Regional Offices:	
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	32376 17
*Eastern : l/l 4 CIT Scheme VII M, V.I.P. Road, Maniktola, CALCUTTA 700054	337 86 62
Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022	60 38 43
Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113	235 23 15
†Western : Manakalaya, E9, Behind Marol Telephone Exchange, Andheri (East), MUMBAI 400093	832 92 95
Branch Offices::	
'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001	5501348
‡Peenya Industrial Area, 1 st Stage, Bangalore-Tumkur Road, BANGALORE 560058	839 49 55
Gangotri Complex, 5th Floor, Bhadbhada Road, T.T. Nagar, BHOPAL 462003	55 40 21
Plot No. 6263, Unit VI, Ganga Nagar, BHUBANESHWAR 751001	40 36 27
Kalaikathir Buildings, 670 Avinashi Road, COIMBATORE 641037	21 01 41
Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001	8-28 88 01
Savitri Complex, 116 G.T. Road, GHAZIABAD 201001	8-71 1996
53/5 Ward No.29, R.G. Barua Road, 5th By-lane, GUWAHATI 781003	541137
5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001	201083
E-52, Chitaranjan Marg, C-Scheme, JAIPUR 302001	37 29 25
117/418B, Sarvodaya Nagar, KANPUR 208005	21 68 76
Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road, LUCKNOW 226001	23 89 23
NIT Building, Second Floor, Gokulpat Market, NAGPUR 440010	52 51 71
Patliputra Industrial Estate, PATNA 800013	26 23 05
institution of Engineers (India) Building 1332 Shivaji Nagar. PUNE 411005	32 36 35
T.C. No. 14/1 421, University P. 0. Palayam, THIRUVANANTHAPURAM 695034	621 17
*Sales Office is at 5 Chowringhee Approach, P.O. Princep Street, CALCUTTA 700072	271085
†Sales Office is at Novelty Chambers, Grant Road, MUMBAI 400007	309 65 28
‡Sales Office is at 'F' Block, Unity Building, Narashimaraja Square, BANGALORE 560002	222 39 71

Printed at Printograph, New Delhi (INDIA).

AMENDMENT NO. 1 OCTOBER 1987

TO

IS:11380(Part 1)-1985 METHOD OF SAMPLING FOR THE DETERMINATION OF PESTICIDE RESIDUES

PART 1 AGRICULTURAL AND FOOD COMMODITIES

(Cover page, pages 1 and 3, title)
Substitute the following for the existing title:

'Indian Standard

METHOD OF SAMPLING FOR DETERMINATION OF PESTICIDE RESIDUES IN AGRICULTURAL AND FOOD COMMODITIES '

(Page 1 and all other pages) - Delete 'Part 1' wherever it occurs.

(AFDC 57)

Printed at Printograph, New Delhi. Ph : 5726847