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PAKISTAN STANDARD

SPECIFICATION FOR BANASPATI (3RD REV.)

PAKISTAN STANDARDS AND QUALITY CONTROL AUTHORITY
STANDARDS DEVELOPMENT CENTRE,
39 – Garden Road, Saddar,
Karachi-74400.

BANASPATI
(3RD REVISION).

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FOR

BANASPATI (3RD REV.)

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PAKISTAN STANDARD SPECIFICATION

FOR

BANASPATI (3RD REVISION)

0. FOREWORD

0.1 This Pakistan Standard was adopted by the Pakistan Standards & Quality Control Authority, Standards Development Centre on 28-01-2003 on the endorsement by the Agricultural & Food Products Divisional Council of the draft finalized by the Oilseeds & their Allied Products Sectional Committee.

0.2 This Pakistan Standard Specification on Banaspati (PS:221) was first revised in 1981 and secondly 1997. The committee felt it necessary to revise it again in the light of latest development in the Industries.

0.3 In the preparation of this standard the views of the manufacturers, technologists and testing authorities have been taken into consideration.

0.4 PS:56-1996 "Methods of Sampling and Test for Vegetable Oils and Fats" (1st Rev.) is a necessary adjunct to this standard.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the results of a test or analysis, shall be rounded off in accordance with PS:103-1991 (1st Rev.) "Methods of Rounding Off Numerical values" the number of significant places retained in the rounded off value shall be the same as that of the specified value in the standard.

0.6 This standard is intended chiefly to cover the technical provisions relating to the supply of the material and it does not purport to include all the necessary provisions of a contract.

1. SCOPE

1.1 This Pakistan Standard prescribes the general requirements and Methods of Sampling & Test for Banaspati.

2. TERMINOLOGY

2.1 Banaspati means the product obtained by hydrogenation of edible oil of vegetable origion or blend of vegetable oils. It shall contain no flavouring, colouring or any other matter deleterious to health. The product shall also conform to the following requirements :

2.2 Edible Vegetable oils to be used for the preperation of Banaspati include Refined Cotton Seed Oil, Refined Low Erucic Acid Rapeseed Canola Oil, Refined Sesame Seed Oil, Refined Ground Nut Oil, Refined Soyabean Oil (Edible Grade), Refined Palm Oil, Refined Palmolein, Refined Sunflower Oil, Refined Maize (Corn) Oil and Refined Safflower Oil.

3. REQUIREMENTS

3.1 The product shall be prepared from properly refined, bleached and deodorized hydrogenated vegetable oils in premises maintained under hygienic condition according to PS:1825-1987 for Good Manufacturing Practice in Manufacturing, Processing, Packing or Holding Human Food.

3.2 The product shall be produced from any of the following vegetable oil or a blend thereof:

i. Refined Cotton Seed Oil.
ii. Refined Low Erucic Acid Rapeseed (Canola Oil).
iii. Refined Edible Sesame Seed Oil.
iv. Refined Ground Nut Oil.
v. Refined Soyabean Oil
vi. Refined Palm Oil (Edible Grade).
vii. Refined Maize (Corn) Oil.
viii. Refined Safflower Oil.
ix. Refined Sunflower Oil.
x. Refined Palmolein.

3.3 The product shall be clean and wholesome (conducive to sound health).

3.4 When melted the product shall be clear, bright and free from sediment, unpleasant taste and smell.

3.5 The product shall also conform to the characteristic given in Table – 1.

TABLE – 1.

SL: #	CHARACTERISTIC	LIMITS	REFER TO PS:56-1996 (1ST REV.) * FOR RELEVANT TEST METHODS	REFERENCE TO APPENDICES OF THIS STANDARD
i.	Moisture & Volatile matter percent by weight, max.	0.15	4	-
ii.	Free fatty Acid (calculated as oleic acid) % by weight, max.	0.2	6	-
iii.	Melting point, as estimated by the capillary tube open at both ends.	36 ± 2 ^{o}C	8	-
iv.	Butyro Refractometer reading at 40 ^{o}C	Not less than 48.0	9	-
v.	Unsaponifiable matter percent by weight, max.	1.5	7.	-
vi.	Nickel, mg/kg, max.	0.2	-	B
vii.	Peroxide value, amx.	5.0	20	-
viii.	Anisidine Value max/***Rancidity (Kries Test)	3.0 R	-	C

ix.	Vitamin – A	33000 I.U. per kg ± 10 % (Assay variation) Per kg of the finished product.	23	-
x.	Soap content., ppm, max.	50	-	D

NOTE :- The use of indigenous seed oil should be encouraged in the blend for use in the manufacturing of Banaspati.

*Method of Sampling Test for Vegetable Oil & Fats.

***Colour produced in Kries Test shall be interpreted alongwith Peroxide Value and shall be sensory test as negative. If the colour is not deper than 3.0 R 1 inch cell lovibond scale.

4. SAMPLING

4.1 Representative samples of the product shall be drawn in the manner prescribed in Appendix – A.

5. TEST

5.1 Test shall be carried out as prescribed in 3.5 and in the appropriate section of PS:56-1996 "Methods of Sampling and Test for Vegetable Oil and Fats" (1st Rev.).

6. PACKING AND MARKING

6.1 Banaspati shall be packed in well-closed tin containers made from food grade material and it shall conform to PS:4773-2002 for Tinplate containers for Ghee, Banaspati, Cooking Oil/Edible Oils or the material shall be packed in suitable sealed flexible packs (PS:4797-2002)* or Plastic containers (made from food grade plastic).

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*Flexible Packs for the packing of Banaspati, Ghee, Cooking Oil & Edible Oil.

6.2 The weight of Tin Container for Packing of Banaspati should be as follows :-

WEIGHT OF FINISHED PRODUCT	**WEIGHT OF TIN CONTAINERS**
16 Kg	852 g to 1000 g
5 Kg	300 g to 350 g
2.5 Kg	225 g to 250 g

6.3 Marking – The following particulars shall be clearly given on each containers :-

a. Name of the material in block letters e.g. BANASPATI.
b. Batch Number.
c. Name and address of the manufacturer.
d. Net weight of the content and Gross weight in Kg.
e. Date of manufacture and Date of expirty***.
f. Nutritional values / Chemical parameters and their limits by weight of finished products shall be displayed on the label.
g. The words contain "33,000 I.U. per kg ± 10 % (Assay variation) of Vitamin – A when packed.
h. Pakistan Standard Number and PS Mark.
i. License Number.
j. Storage conditions.

EXAMPLE :

BANASPATI	
MESSRS.	**XYZ**
BATCH NO.	**ABC**
NET WEIGHT	**'M' kg.**
PAKISTAN STANDARD NUMBER:	**221-**

6.2.1 No label, declaration, methods of preparation and publicity concerning the pruduct, shall be made in a manner likely to mislead the purchaser and/or consumer as to the true nature/or composition of the product as a whole.

***(PS:4449-1999 Expiration periods for food product shall be strictly followed).

APPENDIX – A.
(CLAUSE 4.1)

A-0 SAMPLING

A-1 GENERAL REQUIREEMENTS OF SAMPLING

A-1.0 In drawing, preparing, storing and handling samples the following precautions and directions shall be observed.

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

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

A-1.3 Precaution 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.

A-1.4 Each container shall be sealed air-tight 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.

A-1.5 All the samples shall be stored in such a manner that there is no deterioration of the material.

A-1.6 Unless otherwise specified, 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).

A-2 SCALE OF SAMPLING

A-2.1 Lot – All the containers 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 manufacturer, the batches shall be grouped separately and the containers in each group shall constitute a separate lot.

A-2.1.1 Samples should be tested for each lot for as pertaining conformity of the material to the requirements of the specification.

A-2.2 Gross Sample – A number of containers not less than the cube root of the total number in the lot rounded up to the next higher whole number, shall be selected at random for drawing sample. Minimum number of containers to be selected for sampling from various sizes of lots.

TABLE – II.

Lot Size	Sample Size
2 to 8	2
9 to 27	3
28 to 64	4
65 to 125	5
125 to 216	6
217 to 343	7
344 to 512	8
513 to 729	9
730 to 1000	10
1001 to 1331	11

A-3 TEST SAMPLES & REFEREE SAMPLE

A-3.1 Preparation of Test Sample – Take in a suitable glass or tin container with the help of appropriate sampling device a quantity of the material, as prescribed in Table – III from each of the top, middle and bottom of each container selected for sampling melt by warming in a water batch, mix thoroughly and then draw with a second sampling device at lest 2.5 kg of the material to form the composite sample. Take out about 1.5 kg of the material and divide into three equal parts, each having at least approx. 500 gm (½ kg) of the material. Each portion thus obtained shall be transferred immediately to a clean and dry sample container and sealed airtight. The container shall be labeled with the particulars given under A-1.5 one of these samples shall be for the purchaser and one for the vendor.

TABLE – III.

Amount in the Container	Amount to be sampled from each part of the Container
Up to 1.5 kg	25 g
From 1.6 kg to 2.5 kg	50 g
From 2.6 kg to 4 kg	75 g
From 4.1 kg to 10 kg.	100 g
From 10.1 kg to 20 kg or above	250 g
Approximately	500 g

A-3.2 Referee Sample – The third sample (see A-3.1) bearing the seals of purchaser and the vendor shall constitute the referee sample to be used in case of dispute.

APPENDIX – B.

(Table – 1, Item (vi).

B-0 DETERMINATION OF NICKEL

B-1 REAGENTS

B-1.1 Standard nickel solution – Dissolve 0.673 g of nickel ammonium sulphate in water and dilute to 1 litre. One portion of this solution is diluted ten-folds to obtain solution containing 10.0/Ug nickel per ml.

B-1.2 Dimethyl glyoxime solution 1 g dissolved in 100 ml of ethyl alcohol.

B-1.3 Sodium citrate solution 10 g dissolved in 100 ml of water.

B-1.4 Bromine water – Saturated solution prepared.

B-2 PROCEDURE

B-2.1 Take the sample, slightly acidic, containing from 1 to 15 Ug of nickel per ml. Transfer a 10 ml aliquot to a separating funnel followed by 5 ml of sodium citrate solution which is slightly ammonical. Add 2 ml (excess of cobalt if present) or diemthylglyoxime solution. Extract with three 2 to 3 ml portions of chloroform, washing the combined extracts at the same time with 5 ml or 1 : 50 ammonia and collecting the chloroform layer. Take the extraction of chloroform and wash vigorously with two 5 ml portions of 0.5 hydrochloric acid. Transfer all this solution to 25 ml volumetric flask : and 10 drops of bromine water and then ammonia till the colour of bromine vanishes. Again add 1 ml of concentrated ammonia solution, cooling the solution below 30 ^{o}C and adding 1 ml of dimethyl glyoxime solution. Dilute to the mark and obtain the optical density at 540 within 5 minutes using a reagent blank as reference liquid.

B-3 APPARATUS

B-3.1 Spectrophotometer.

B-3.2 Separatory funnel, 100 ml.

B-3.3 Platinum or other suitable dish, 50 ml.

B-4 REAGENTS

B-4.1 Hydrochloric acid, 1 : 25

B-4.2 Ammonium citrate, 20 percent.

B-4.3 Sodium diethyl-dithio-carbomate reagent, 0.2 percent.

B-4.4 Ammonium hydroxide, 1 : 1

B-4.5 Isoamyl alcohol, A.R. Grade.

B-5 PROCEDURE

B-5.1 Weigh into a platinum or other suitable dish about 10 g of the material and ignite cautiously over a burner. Remove burner while the Banaspati is burning. When flame dies out complete ignition in a muffle furnace at a temperature not exceeding 500 ^{o}C. Take out the dish with the ash from the muffle furnace and cool to room temperature. Dissolve the ash in about 25 ml 1 : 25 hydrochloric acid. Add 5 ml of 20 percent ammonium citrate solution, purified by extraction with diethyl dithio carbamate reagent. Make the solution alkaline (pH 10) to litmus by 1 : 1 ammonium hydroxide. Add 0.2 percent aqueous sodium diethyl dithio carbamate. Shake vigorously and read the solvent layer at 285 in a spectrophotometer against water.

B-6 CALCULATION

B-6.1 Read result against a plotted curve obtained by taking spectrophotometric reading of known quantities of nickel sulphate by the method prescribed in B-5.

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APPENDIX – C

p-ANISIDINE VALUE

DEFINITION

The p-anisidine value is defined by convention as 100 times the optical density measured at 350 nm in a 1-cm cuvette of a solution containing 1.00 g of the oil in 100 ml of a mixture of solvent and reagent according to the method described.

SCOPE

This method determines the amount of aldehydes (principally 2-alkenals and 2,4-dienals) in animal and vegetable fats and oils, by reaction in an acetic acid solution of the aldehydic compounds in an oil and the p-anisidine (see Notes 1), and then measuring the absorbance at 350 nm.

APPARATUS

1. Test tubes – 10 mL min. With either ground-glass stoppers or Teflon^{TM}-lines screw caps.

2. Volumetric flasks – 25 ml.

3. Automatic pipette or automatic burette.

Note – Any pipette and/or burette capable of delivering exactly 1 m and 5 ml is satisfactory.

4. Spectrophotometer suitable for observation at 350 nm.

5. Glass cuvettes – 1.00 ± 0.01 cm, the two cuvettes of each pair must be identical.

REAGENTS

1. Isooctane (2,2,4-trimethylpentane) – optically clear (see Notes, Caution and 2).

2. Glacial acetic acid – analytical reagent quality (see Notes 3).

3. p-Anisidine – analytical reagent quality (see Notes, Caution and 4) 0.25 g/100 mL solution in glacial acetic acid (Reagents, 2) (see Notes 5)

PROCEDURE

Note – The sample should be perfectly clear and dry (see Notes 3).

1. Weigh 0.5 – 4.0 ± 0.001 g of the sample into a 25-ml volumetric flask. Dissolve and dilute to volume with isooctane.

2. Measure the absorbance (Ab) of the solution at 350 nm in a cuvette with the spectrophotometer, using the reference cuvette filled with solvent as a blank.

3. Pipette exactly 5 ml of the fat solution into one test tube (Apparatus, 1) and exactly 5 ml of the solvent into a second test tube. By means of an automatic pipette (Apparatus, 3) and exactly 1 ml of the solvent into a second test tube. By means of an automatic pipet (Apparatus, 3) and exactly ml of the p-anisidine reagent (Reagents, 3) to each tube, and shake (see Notes, 6).

4. After exactly 10 min measure the absorbance (As) of the solvent in the first test tube in a cuvette (Apparatus, 5) at 350 nm, using the solution from the second test tube as a blank in the reference cuvette.

CALCULATIONS

The p-anisidine value (p-A.V.) is given by the formula

$$\text{p-A.V.} = \frac{25 \times (1.2\ As - Ab)}{m}$$

where,

As = absorbance of the fat solution after reaction with the p-anisidine reagent (Reagents, 3).

Ab = absorbance of the fat solution

m = mass of the test portion, g.

PRECISION (See References, 2)

	Crude Rapeseed Oil		Refined Palm Oil	
	Sample 1	**Sample 2**	**Sample 1**	**Sample 2**
No. of labs.	20	20	20	20
Mean value	2.0	2.0	2.3	2.3
Repeatability, CV, %	4.0	5.8	4.8	4.6
Reproducibility, CV, %	35	37	30	31

NOTES

CAUTION

Isooctane is flammable and a fire risk. Explosive limits in air are 1,1-6.0 %. It is toxic by ingestion and inhalation. A property operating fume hood should be used when working with this solvent.

Acetic acid in the pure state is moderately toxic by ingestion and inhalation. It is a strong irritant to skin and tissue. The TLV in air is 10 ppm.

p-Anisidine is an irritant and should be handled with care, preferably in a fume hood. p-anisidine is an aromatic amine, a class of toxic and possibly carcinogenic chemicals. p-Anisidine is a carcinogen in rats and mice, causing urinary carcinomas or papillomas. [Fourth Annual Report on Carcinogens, NTP 85-002, 1985, p. 2: Chem. Res. Toxicol. 4:474 (1991)]. The TLVis 0.1 ppm.

NUMBERED NOTES

1. In the presence of acetic acid, p-anisidine reacts with aldehydic compounds in oil or fats. The intensity of color of the yellowish reaction products formed depends not only on the amounts of aldehydic compounds present but also on their structure. It has been found that a double bond in the carbon chain conjugated with the carbonyl double bond increases the molar absorbance four to five times. This means that 2-alkenals and dienals, especially, will contribute substantially to the value found.

2. In most cases n-hexane can be substituted for isooctane as a solvent. However, oils containing high amounts of oxidized fatty acids will not dissolve completely in n-hexane. For such oils isooctane should be used as the solvent. The absorbance of the solvent used (isooctane or n-hexane), measured in a 1.00-cm cuvette between 300 and 380 nm, must be nil or nearly nil. The commercial product can be freed from absorptive material by percolating it through a glass column (3.5 cm i.d., and 100 long) filled with silica gel.

3. The reaction between p-anisidine and aldehydes involves the formation of water. Hence, the presence of moisture in any of the reagents or in the sample leads to incomplete reaction and, consequently, low values. Since glacial acetic acid is highly hygroscopic, it is essential to check its moisture content by a Karl Fischer determination. If the content exceeds 0.1 percent, the acetic acid must be discarded.

4. In storage, p-anisidine tends to darken as a result of oxidation. The p-anisidine crystals, which should be cream colored, should be stored at 0 – 4 ^{o}C in a dark bottle. The crystals should not be exposed to strong light and should be used before any colour change is observed. A discolored reagent can be reduced and decolorized in the following way. Dissolve 4.0 g of p-anisidine in 100 mL of water at 75 ^{o}C. Add 0.2 of sodium sulphite and 2.0 g of active carbon and stir for 5 min. The filter through a double filter paper. If carbon passes through, repeat filtration. Cool the filtered solution to about 0 ^{o}C, allow to stand at this temperature for at least 4 hr. or, preferably, overnight. Filter off the crystallized p-anisidine and wash with a small amount of water at a temperature of about 0 ^{o}C. After drying in a vacuum desiccator, transfer the crystals into a brown glass bottle. If stored in the dark and at low temperature, the crystals obtained should not darken appreciably for 1 year.

5. Reagent solutions having an absorbance greater than 0.200 when measured in a 1.00-cm cuvette at 350 nm against isooctane or n-hexane as a blank should be discarded.

6. The mixture should be completely homogenized with minimum shaking and then allowed to react for 10 min before proceeding with the absorbance measurement (Reference, 4).

REFERENCES

1. IUPAC, standard Methods for the Analysis of Oils, Fats and Derivatives. 7th ed., Method Number 2.504 – Determination of the p-anisidine value (p-A.V.), Blackwell Scientific Publications, Bostan, MA and Oxfored, UK (1987).

2. FOSFA International Collaborative Study #P 15, May 1986, Document No.384, ISO/TC 34/SC 11, February 12, 1987.

3. JAOCS 51 : 17 (1974).

4. Hamilton, R.J., and S. Hamilton, Lipid Analysis, Oxford University Press, New York, 1992, pp. 45 – 47.

APPENDIX – D

DETERMINATION OF SOAP CONTENT TITRIMETRIC METHOD

DEFINITION

The titrimetric method determines the alkalinity of the sample as sodium oleate.

SCOPE

Applicable only to refined vegetable oils.

APPARATUS

1. Test tubes – approximately 150 x 40 mm of borosilicate glass fitted with ground glass stoppers and flattened at the base.

2. Microburet – 5 ml.

3. Steam bath – hot water bath may also be used.

REAGENTS

1. Acetone – containing 2 % water, prepared by adding 20 ml distilled water to 980 ml of reagent-grade acetone (see Notes, Caution).

2. Hydrochloric acid (HC1) – approximately 0.01 N, accurately standardized, See AOCS Specification H 14 – 52.

3. Bromophenol blue indicator solution – 1.0 % in water.

4. Sodium hydroxide (NaOH) – approximately 0.01 N.

PROCEDURE

1. Just prior to the analysis, prepare the test solution by adding 0.5 ml of the bromophenol blue indicator solution (Reagents, 3) to each 100 ml of the aqueous acetone solution (Reagents, 1) and titrating with 0.01 N HC1 (Reagents, 2) or 0.01 N NaOH (Reagents, 4) until the test solution is just yellow in colour.

2. Weigh 40 g (Notes, 1) of the oil or fat to be tested into a test tube (Apparatus, 1) which has been well rinsed with the test solution (Procedure,1)

3. Add 1 ml of water to the test sample, warm on a steam bath (or in water bath) and shake vigorously. Add 50 ml of the test solution (Procedure, 1), and after warming, shake the tube well and allow the contents to separate until two distinct layers are formed. Note – If soap is present in the oil or fat, the upper layer will be colored green to blue.

4. Slowly add 0.01 N HC1 (Reagents, 2) from the microburet until the color just changes from green/blue to yellow. Repeat the warming, shaking and addition of 0.01 N HC1 until the yellow color of the upper layer remains permanent. Record the total volume of acid required as ml.

5. A blank correction should be determined on soap-free oil, using Procedure, steps 1 – 4. Record the volume of acid required for the blank as ml.

CALCULATIONS

1. ppm soap as sodium oleate =

$$\frac{(mL_s - mL_b) \times N \times 304{,}400}{\text{sample mass, g}}$$

where.

mL_S = volume, ml HCI obtained in Procedure, 4

mL_b = volume, ml HC1 obtained in Procedure, 5

N = normality of HC1.

NOTES

Caution

Acetone is highly flammable. Forms explosive peroxides with oxidizing agents. Use effective fume-removal device. Do not mix with chloroform.

Hydrochloric acid is a strong acid and will cause severe burns. Protective clothing should be worn when working with this acid. It is toxic by ingestion and inhalation and strong irritant to eyes and skin. The use of a properly operating fume hood is recommended. When diluting the acid, always add the acid to the water, never the reverse.

NUMBERED NOTES

1. The method as written is suitable for the determination of soap of concentrations of up to 0.05 % in oils and fats. At higher concentrations it is better to analyze 4 g of sample and use 0.01 N HCI.

REFERENCES

1. A study among seven industrial organizations indicated that this method is suitable only for refined oils. Yukagaku (Japan) 39 : 1056 (1990).

2. This method is identical with Codex Alimentarius method CAC/RM 13 – 1969 and similar to British Standard 648 : 1958.

3. Wolf, J.P. Oleagineus, p, 197 (April, 1948).